"Electromagnetic Wave Interaction with Water and Moist Substances"
6th International Conference on Electromagnetic Wave Interaction with Water and Moist Substances

Papers, Posters and Exhibits

Conference Proceedings

CD-ROM

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Welcome to the 6. International Conference on Electromagnetic Wave Interaction with Water and Moist Substances

It is my great pleasure to welcome you to Weimar the town of Goethe and Schiller.

The Material Research and Testing Institute (MFPA) Weimar is delighted to host the  6. International Conference on Electromagnetic Wave Interaction with Water and Moist Substances 2005, and we thank you for participation.

This conference was created as a forum of theory and practice, where experts meet each other, and exchange results of research, development, and application. The interested public is invited to participate. We invited scientists, manufacturers and users in order to exchange information, latest findings, and to offer solutions and devices.

Material investigations and moisture measurements using electromagnetic waves in a wide spectrum serve for quality control and improvement in many branches like industry, civil engineering, agriculture, commerce, but also for foodstuffs, for instance: for quality detection of meat, fruits, coffee, and so on.

The research of moist materials and development of measuring devices are absolutely necessary. Experts in this complex area, being too complicated and time consuming for large industry, are relatively rare. Their cooperation around the world helps to solve quality problems in the framework of ISO 9000, to save water and energy, and to reduce environmental damage.

The conference, but also the foundation of International Society of Electromagnetic Aquametry will help to support the cooperation of scientists and users of material and moisture measurements by using microwave and dielectric measuring methods in a wide frequency spectrum.

The predecessors of the 6. Conference were workshops held in connection with the IEEE MTT-S Symposium in Atlanta 1993, and in San Francisco 1996, organised by Dr. Kraszewski. The third workshop was held as an independent conference and was organised by Dr. Kraszewski and Dr. Lawrence in Athens, GA, in April 1999. The 4. Conference were held in Weimar 2001, organized by Dr. Kupfer, Dr. Nitsche, assisted by Prof. Knöchel (Technical Faculty of University Kiel) and Dr. Hübner (Research Center Karlsruhe). The conference was a big success; 130 attendees came from 26 countries all over the world. The conference was greatly supported by the Deutsche Forschungsgemeinschaft and the Thuringian Ministry of Science, Research and Art. In 2003 the 5. Conference was organized with high effort at a wonderful place of volcanic and geothermal activities in Rotorua / New Zealand by Dr. Kailash Thakur. The conference offered an outstanding scientific programme with 50 contributions, which also gave some insight into the special needs of moisture measurement technology in the south pacific area and New Zealand. Although the meeting was well attended, the participation suffered from strong influence by the war in Iraq and the SARS disease.

The results of the workshops and conferences were published in three books "Microwave Aquametry" edited by Kraszewski, "RF & Microwave Sensing of Moist Materials, Food and other Dielectrics" edited by Kupfer, Kraszewski, Knöchel, and “Electromagnetic Aquametry” edited by Kupfer.

The Call for Papers of the 6. International Conference on Electromagnetic Wave Interaction with Water and Moist Substances, was very successful. The papers came from all over the world. The results of selection of abstracts were summarised in the "Programme and Invitation" which has been sent around the world. We were also happy that manufacturers of moisture...
measurement systems and devices presented their products in the framework of such a conference. For cooperation concerning the programme arrangement and its review we thank Prof. Knöchel, Prof. Kummer, Prof. Lasri, Prof. F. Volgyi, Prof. Okamura, and Dr. Trabelsi.

The scope of the conference covered nearly all aspects of measurement and investigation of materials in connection with water, but also other dielectrics. "Microwave Aquametry" is the wide area of metrology that includes science and technology applied in Microwave Sensing of moist materials, foods, and other dielectrics.

The proceedings are arranged corresponding to the sessions of the conference:

1. Dielectric Properties of Aqueous Materials
2. Dielectric Properties of Biological Substances
3. Application of Model Systems, Mixing Rules, Calibration, and Reconstruction Algorithm
4. Electromagnetic Wave Propagation in Polarizable Wet Media
5. Measurement Methods
7. Application of Methods and Sensors for Civil Engineering, Pharmaceutical Industry, Oil Industry, and Quality Inspections
8. Application of Methods and Sensors to Foodstuffs and Agricultural Products and
9. Moisture Content Determination in Soils, Snow, and Waste Disposals
10. Multi-parameter Measurement for Determination of Properties such as Conductivity, Moisture, Density, etc.
11. Poster Session

We are grateful to all authors who submitted their work to the conference. The newest results of research and development of Microwave Aquametry were presented in 70 contributions. The 167 contributors came from 22 countries: Belarus, Canada, Croatia, Czech Republic, France, Germany, Greece, Hungary, India, Israel, Japan, Korea, Malaysia, New Zealand, The Netherlands, Portugal, Russia, Spain, Sweden, Switzerland, UK, and USA.

An exhibition, which accompanies the scientific conference, demonstrates the basis for practical applications. We appreciate all exhibitors who came from Belarus, USA, and Germany.

A special conference web-site designed by Dr. Kuhne and Dr. Kupfer showed the conference programme, information on accommodation, and pictures from Weimar and Thuringia.

This event and this publication could not get successful without help from certain quarters. We would like to acknowledge the support we received from the Deutsche Forschungsgemeinschaft (German Research Foundation) and from the Federal Ministry for Education and Research for this conference. By keeping the fees to a minimum we have enabled also scientists from India and East Europe to participate in the conference.

We would like now to thank Prof. Hübner, Fachhochschule Mannheim and Mrs. Rieger, for all the work they have done during the preparation and the realisation of the conference. Within the MFPA we thank especially Mrs. Bergmann, Mrs. Spangenberg, and Ms. Georgi for mailing calls and programmes, but also Ms. Kupfer and the students, M. Trinks and T. Trinks for their busy work during the formatting of the conference papers and preparation of the exhibition. We thank the Bauhaus-University for the support of the conference.

At this place I want to thank Prof. Bergmann, Dr. Nitsche, Dr. Girlich, Mr. Trinks, Dr. Wagner, and all colleagues of MFPA who helped during preparation and realisation of the conference.
We would like to acknowledge the professional design of the Calls, Programmes, and proceedings worked out by Klapproth & Koch and the good work of the Gutenberg Druckerei Weimar.

We hope that we, together with the authors and reviewers of the papers, have produced a publication of lasting value.

The meeting was intended to be a well accepted event for all attendees to meet each other, to exchange information, and to establish contacts and acquaintances for the future.

I feel confident that this conference will continue the series of conferences that began in Atlanta into the future. On behalf of the Organisation Committee thank you for your participation in this conference once more.

Weimar, May 2005

Dr. Klaus Kupfer
Conference Chairman
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Polymer Chains Linked by Water Molecules. A Dielectric Spectrometry Study

Elke S. Hanke, Kerstin von Roden, Udo Kaatze
Drittes Physikalisches Institut, Georg-August-Universität Göttingen

ABSTRACT. Complex permittivity measurements as a function of frequency between 1 MHz and 3 GHz are described for mixtures of water with poly(ethylene glycol) oligomers of different chain length and also with two poly(ethylene glycol) dimethylethers. The dielectric spectra are analytically represented by a Cole-Cole relaxation spectral function. The principal relaxation time of this function is discussed to show two opposite effects in the hydrogen network fluctuations upon the water content. At low water content the water molecules obviously form hydrogen bonded links between polymers and thus lead to a slowing down of the reorientational motions. At higher water content the large concentration of additional hydrogen bond partners leads to faster reorientational motions of the dipolar groups or molecules, as characteristic to water, alcohols, alcohol-water, and alcohol-alcohol mixtures.

Keywords: permittivity, relaxation, hydrogen bonds, polymers

1 Introduction

Electromagnetic wave interactions with water-containing materials are largely controlled by the complex dielectric spectrum \(\varepsilon(\nu) = \varepsilon'(\nu) - i\varepsilon''(\nu)\) of the sample. At microwave frequencies (100 MHz \(\leq \nu \leq 100\) GHz) the real part \(\varepsilon'(\nu)\) and negative imaginary part \(\varepsilon''(\nu)\) of the permittivity spectrum of water at 25°C is displayed in Fig. 1.

![Fig. 1 Real part \(\varepsilon'\) and negative imaginary part \(\varepsilon''\) of the complex dielectric spectrum of water at 25 °C [1,2].](image_url)
A dispersion \((\text{d}\varepsilon'(\nu)/\text{d}\nu < 0)\) / dielectric loss \((\varepsilon''(\nu) > 0)\) region emerges which, within the frequency range under consideration, can be analytically represented by a Debye type relaxation function \(([3], \omega = 2\pi\nu)\)

\[
\varepsilon(\nu) = \varepsilon(\infty) + \frac{\varepsilon(0) - \varepsilon(\infty)}{1 + i\omega\tau_w}
\]

(2)

with the discrete relaxation time \(\tau_w\). Hence \(\varepsilon(0)\) denotes the permittivity extrapolated to low frequencies (“static permittivity”), \(\varepsilon(\infty)\) is the permittivity extrapolated from the microwave data to high frequencies, and subscript “\(w\)” refers to water.

The nonvanishing relaxation time \(\tau_w\), on the order of 10 ps at room temperature \([1,2]\) is due to the hindered reorientational motions of the water molecules. As the prevailing hindrance in these motions results from hydrogen bonding, dielectric relaxation of water is a reflection of the three-dimensional hydrogen network fluctuations.

The dielectric relaxation time of liquids depends upon the degree of hydrogen bonding in an intricate manner. When, for example, substituting a terminal methyl group of \(n\)-heptane by a hydroxyl group, thus when going from the nonassociating nonpolar alkane to dipolar \(n\)-hexanol, capable of forming hydrogen bonds, the dielectric relaxation time increases by a factor of roughly \(10^3\) from about 2 ps to about 2 ns \((20\,^\circ\text{C}\, [4])\). This result clearly reveals the impediment of molecular motions by association. However, if, within the series of monohydradic normal alcohols \(\text{CH}_3(\text{CH}_2)_{n-1}\text{OH}, n = 1, 2, \ldots\) the concentration of hydrogen bonding hydroxyl groups is increased, the dielectric relaxation time does not increase. Rather it decreases significantly to reach 50 ps for methanol \([5]\). A further increase in the concentration of hydrogen bonding groups or molecules finally leads to the even noticeably smaller relaxation time of water \((\tau_w = 8.27\, \text{ps}, 25\, ^\circ\text{C}\, [1])\). Hence the dielectric relaxation of associating liquids obviously is governed by opposing trends. This situation has prompted us to perform a dielectric relaxation study of poly(ethylene glycol)/water and poly(ethylene glycol) dimethylether/water mixtures and to investigate the dependence of the dielectric relaxation time upon the overall content of hydrogen bonding groups or molecules.

2 Experimental

Poly(ethylene glycol) samples with molar weights 200 (PEG 200), 300 (PEG 300), 400 (PEG 400), and 600 (PEG 600), as well as poly(ethylene glycol) dimethylethers with weights 250 (PEGDME 250) and 500 (PEGDME 500) have been purchased from Aldrich and used as delivered. The water content of the polymers was determined by Karl-Fischer-titration and was found smaller than 0.3 % by weight throughout. In order to avoid undesired water uptake the polymers were always stored in vacuum and handled under dry nitrogen. The mixtures with water were prepared by weighing appropriate amounts into suitable flasks. Millipore water was used. The density \(\rho\) of the samples has been determined to within \(\pm 5 \cdot 10^{-4}\%\) with the aid of a vibrating tube densitometer (Paar Physica DMA 5000). The shear viscosity \(\eta_s\) was obtained with an error of less than 1 % from measurement with a falling ball viscosimeter (Haake B/BH) and also with Ubbelohde capillary viscosimeters (Schott).

Between 1 MHz and 3 GHz the complex permittivity \(\varepsilon(\nu)\) has been measured by input impedance measurements on two coaxial cells from the cut-off variety \([6]\). One cell was
matched to the low frequency part ($\nu < 100$ MHz typically), the other one to the high frequency part ($\nu > 20$ MHz) of the measuring range. The complex input impedance of the cells has been determined utilising a computer-controlled network analyser (HP 8753A), combined with a suitable reflection test set (HP 85044A). The specimen cells were calibrated using air, water, and acetone as references. At $\nu \leq 1$ GHz the experimental errors were smaller than 1.5% in both, $\varepsilon'$ and $\varepsilon''$. Above 1 GHz the error was 5% in $\varepsilon'$ and 7% in $\varepsilon''$. The error in the frequency of measurement was negligibly small. The temperature of the samples was controlled to within 0.05 K and was measured with an error of less than 0.02 K.

3 Results

In Fig. 2 the complex dielectric spectrum of a PEG 400 / water mixture is presented. Obviously, the binary liquid displays only one relaxation region. This feature has already been shown previously by complex permittivity measurements up to 40 GHz [7], covering also the frequency range of the dielectric relaxation of water (Fig. 1). Hence there do not exist separated phenomena due to the relaxation of the dipolar polymer molecules and to the water molecules, respectively.

![Complex dielectric spectrum of a mixture of PEG 400 with water (8 % w/w) at 10 °C.](image)

Rather the mixtures seem to display dielectrically homogeneous liquids. The dispersion / dielectric loss region, however, extends over a somewhat broader frequency range than with water, thus indicating a distribution of relaxation times. It is found that all spectra can be well represented by the empirical Cole-Cole relaxation function [8]

$$\varepsilon(\nu) = \varepsilon(\infty) + \frac{\varepsilon(0) - \varepsilon(\infty)}{1 + (i\omega \tau_s)^{\Gamma-h}}$$

in which $\tau_s$ denotes the principal relaxation time and parameter $h$ controls the width of the underlying relaxation time distribution function which, when plotted versus $\ln(\tau/\tau_s)$ is symmetrically bell shaped. Parameters of the relaxation function (Eq. (3)), as found by
nonlinear least-squares regression analysis of the experimental spectra, are discussed in the following section.

4 Discussion

For two series of polymer/water mixtures the static permittivity $\varepsilon(0)$ is displayed versus mass fraction $Y$ of water in Fig. 3.

Fig. 3 Static permittivity $\varepsilon(0)$ versus mass fraction of water for PEG 400 / H$_2$O (triangles) and PEGDME 500 / H$_2$O (circles) mixtures at 25 °C.

The data reveal a linear dependence of $\varepsilon(0)$ upon $Y$. The static permittivity of a mixture of different dipolar species is not just given by the amounts of molecular dipole moments, the concentrations of dipolar species, and the high frequency permittivity, but also by dipole orientation correlation factors [9]. Strictly, water-water, water-polymer, and polymer-polymer dipole orientation correlations have to be considered. Since we cannot derive different correlation factors from one permittivity value, we extend Fröhlich’s theory for the static permittivity of a one-component dipolar liquid [10] by introducing an effective dipole orientation correlation factor $g_{\text{eff}}$, thus using the relation

$$\varepsilon(0) - \varepsilon(\infty) = \frac{N_A}{3\varepsilon_0 k_B T} \frac{3\varepsilon(0)}{2\varepsilon(0) + \varepsilon(\infty)} g_{\text{eff}} \left( c_p \mu_{\text{p,eff}}^2 + c_w \mu_{\text{w,eff}}^2 \right).$$

Here $N_A$ is Avogadro’s number, $\varepsilon_0$ is the electric field constant, $k_B$ is Boltzmann’s constant, $c_p$ and $c_w$ denote the polymer and the water concentrations, respectively, and $\mu_{\text{p,eff}}$ as well as $\mu_{\text{w,eff}}$ are effective polymer and water dipole moments. These effective dipole moments follow from the static permittivities of the pure liquids assuming $g_{\text{eff}} = 1$ in eq.(4). In doing so $\mu_{\text{p,eff}}$ results at $c_w = 0$ and vice versa. According to this treatment of static permittivity data $g_{\text{eff}}$ shows...
variations in the molecular structure of one constituent on addition of the other one. Evaluation of the experimental $\varepsilon(0)$ values yielded effective correlation factors close to 1. Hence there do not seem to exist noticeable effects in the structure of the polymer/water mixtures, as reflected by the static permittivities of the liquids.

The principal dielectric relaxation times $\tau_s$ of the PEG/water mixtures reveal quite different characteristics. In Fig. 4 the $\tau_s$ values for PEG 400 mixtures at 10 °C and 25 °C are displayed as a function of mass fraction $Y$ of water. Also shown for comparison are literature data for PEG 600/H$_2$O mixtures at 25 °C [7].

![Graph showing dielectric relaxation time $\tau_s$ versus mass fraction of water $Y$ for PEG 400 / H$_2$O mixtures at 10 °C (points) and 25 °C (closed triangles). Also shown are literature data for PEG 600 / H$_2$O mixtures at 25 °C ([7], open triangles).](image)

The latter data had been obtained from different methods of measurement. With all series of PEG/H$_2$O mixtures the relaxation time values first increase on addition of water. This is an unusual result because, normally, the relaxation time of mixtures from dipolar constituents displays a monotonous transition from the $\tau_s$ value of one constituent to that of the other one. Mixtures of monohydric alcohols with water [11] and alcohol/alcohol mixtures [5] are examples.

The positive initial slope $S = \lim_{Y \to 0} (d\tau_s/dY)$ in the dependence of the dielectric relaxation time of PEG/H$_2$O mixtures upon $Y$ is a strong indication of the formation of water links between poly(ethylene glycol) molecules. Likely the hydrogens of a water molecules form hydrogen bonds to the lone pair electrons of PEG oxygens. The relative slope $S/\tau_s(0)$ is larger at lower temperatures (17.1, 10 °C) than at higher temperatures (3, 25 °C). A similar behaviour emerges with the viscosity data of the mixtures (Figure 5).
The initial slope $\lim_{Y \to 0} (d\eta_s/dY)$ is positive at 10 °C, zero at 25 °C, and negative at 40 °C, indicating an interaction energy of the water links on the order of the thermal energy at room temperature. The initial relative slope in the principal relaxation time of mixtures of the aprotic PEGDME 500 with water is noticeably larger ($S/\tau_s(0) = 24$, 25 °C) than that of the corresponding PEG 400/water system ($S/\tau_s(0) = 3$, 25 °C). We assume this difference to be due to the fact that part of the oxygen lone pair electrons of PEG form hydrogen bonds with the terminal hydroxy groups of the polymers. This assumption is in conformity with the significantly larger principal relaxation time of PEG 400 ($\tau_s = 165$ ps, Y = 0, 25 °C) as compared to PEGDME 500 ($\tau_s = 31$ ps, Y = 0, 25 °C).

![fig5](image)

**Fig. 5** Shear viscosity $\eta_s$ of PEG 400 / H$_2$O mixtures displayed as a function of mass fraction of water at 10 °C (points), 25 °C (triangles) and 40 °C (squares).

The $\tau_s$-versus-$Y$ relations of the PEG/water mixtures show some additional special characteristics, like the relative minimum at $Y = 0.09$ (10 °C) or $Y = 0.075$ (25 °C) and the shoulder at around $Y = 0.22$ at 10 °C. Though it has been verified by multiple measurements that these features are no artefacts, no clear conclusion can be drawn presently on the underlying molecular mechanisms.
5 Conclusions

The overall trends in the principal dielectric relaxation time of poly(ethylene glycol)/water mixtures reflect (at least) two opposite effects. At low water content, water molecules form hydrogen bonded links between two oxygens of polymer molecules. Because of the hydrogen network established thereby the molecular dynamics of the liquids is slowed down, corresponding with an initial increase in the relaxation time dependence upon the water content. Towards high water concentrations additional water leads to a reduction of the relaxation time. The hydrogen network fluctuations are faster because of the availability of additional hydrogen bond partners. This tendency corresponds with the wait-and-switch model of electrical dipole reorientation [4] as based on computer simulation studies of water [12]. Within the framework of this model the dielectric relaxation time of associating liquids is dominated by the time for which the dipole has to wait until, due to thermal fluctuations, a suitable neighbour molecule exists in a position that promotes reorientation. As the probability for such a constellation increases with increasing concentration of dipolar species, capable of forming hydrogen bonds, the relaxation time decreases with increasing content of polar molecules or groups and so do the $\tau_s$ values of the PEG/water mixtures at mass fraction $Y$ of water larger than 0.1.

References


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Accurate Determination of Debye Relaxation Data of Polar Liquids by a Multistep Retro-modelling Technique

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ABSTRACT. The expanding use of microwave modelling for design of microwave heating systems requires improved dielectric data of the materials to be processed. There is a need for a larger number of reference substances than just water, also for tests and calibrations of microwave sensors of various kinds. Various criteria limit the choice to only a handful pure liquid compounds. Pyridine, dimethylformamide (DMF) and a methanol-water mixture were chosen and measurements were made at about 950 and 2300 MHz over the temperature interval −5 to +40 °C, using an accurate multi-step retro-modelling technique and a FDTD modelling. Data fits are presented in Debye relaxation format.

Keywords: polar liquids, reference, permittivity, microwave, retro-modelling

1 The Choice of Liquids

Desirable properties of a reference liquid are: representativity for expected practical load substances; reasonably non-hazardous; stable; a low vapour pressure at room temperature; reasonably inexpensive and common; to be obtainable with high purity and the impurities not influencing the permittivity much.

Water is of course a primary choice; it possesses all these properties and is the liquid having by far the best available literature data on microwave permittivity. It can therefore be used for certain calibrations of dielectric measurement equipment which is subsequently used with other liquids. Water at 20 °C has been used for calibrations towards establishing permittivity data over an extended temperature range, as described in a companion on paper [1].

Other reference liquids should not absorb ambient water vapour. As an example, the moisture absorption criterion excludes pure methanol and ethanol. In spite of this, there are reports of these liquids being used for calibrations of equipment of the open coaxial probe type [2].

The criteria should also include a spread in permittivity between the chosen liquids. Water has an ε’ of almost 80 in the ISM frequency range of major interest (about 900…2500 MHz). The choices of ε’ must consider that the effective wavelength contraction is proportional to √ε’ and the power penetration depth dp is roughly proportional to √ε’/ε”, but also the practical need for a substance with data close to those which are to be routinely measured or used. When all criteria above are applied, the choice becomes limited to only a handful pure liquid compounds.

1-octanol (CH3(CH2)7OH) is a proper choice as low-ε’ reference liquid. Rather good but old literature data are available in [5]. It is obtainable with >99,5 % purity and <0,1 % H2O, and the major residual constituent is 2-octanol (CH3(CH2)5CH–OH–CH3) which has quite similar dielectric properties. Approximate literature data at +20 °C are ε= 3,17 –j0,71 at 900 MHz and ε= 3,10–j0,26 at 2450 MHz.

Pyridine (C5H5N) is a proper choice as medium-εϖ reference liquid. Old literature data are available in [5]. It is obtainable with >99,5 % purity and <0,1 % H2O, with the major residual constituent being α-picoline (H in the benzene ring position nearest to the N being replaced by CH3), which has rather similar εs (static, f=0) to pyridine. Approximate literature data at
+20 °C are $\varepsilon = 13,5 - j0,46$ at 900 MHz and $\varepsilon = 13,4 - j1,25$ at 2450 MHz.

**DMF (dimethylformamide) (HCON(CH$_3$)$_2$)**
is a proper choice for a high-$\varepsilon'$ and low-$\varepsilon''$ reference liquid. Some scattered literature data exist, as well as unpublished data. Approximate data at +20 °C are $\varepsilon = 38 - j2$ at 900 MHz and $\varepsilon = 37 - j4$ at 2450 MHz.

It is extensively used in pharmaceutical microwave chemistry and is probably the overall least toxic choice of the special pharmaceutical MW chemistry compounds obtainable with very high purity. DMF is readily obtainable with $\geq 99.9\%$ purity and $<0.02\%$ H$_2$O. **Methanol-water mixtures** can also be used, primarily for obtaining high $\varepsilon'$ values under conditions of a high tan$\delta$ at 2450 MHz – similar to the dielectric properties of typical compact food substances. Literature data are available (e.g. $\varepsilon \approx 46 - j20$ at 2450 MHz, for 60 % MeOH and 40 % H$_2$O by volume), but are expected not to be very accurate. Even if these high losses may be favourable for some comparisons, the high dispersion may be a problem but results in a more stable $\varepsilon''$ value under temperature variation, at 2450 MHz. The mixing inaccuracy is an additional source of error, but should be small. The risk of stronger evaporation of methanol than water during handling must, however, be observed.

In view of the choice of about 3.3 inner diameter glass vials for the water measurements described in [1], the three liquids with higher $\varepsilon'$ was chosen. 1-octanol would require a larger diameter for high accuracy measurements.

### 2 The Measurement Applicator

A resonant applicator which allows the use of two different frequencies with the same sample has been developed, since this property facilitates practical use and in essence eliminates the only disadvantage with single frequency resonant measurement systems. A rotationally symmetrical cavity was chosen, to simplify precise manufacturing. – The system is shown in Fig. 1 and described in the companion paper [1].

### 3 The Retro-modelling Principle and Practise

The principle and procedure for water measurements is described in the companion paper [1].

The inner diameter of the vial is determined with the use of pure water at a carefully controlled temperature at about +20 °C. The best available literature data at the actually measured resonance frequencies with the water load are then used for changing the vial dimension in the modelling, until a good agreement is obtained.

In the final steps, the sensitivity factors of resonant frequency variation $\Delta f/\Delta\varepsilon'$ and (mainly) resonant transmission attenuation $\Delta T/\Delta\varepsilon''$ are modelled, around the specific values obtained by the measurements. All retro-modelling [3, 4] for obtaining the unknown permittivities of
the liquid samples is done analogously, with empty cavity and empty vial measurements at each temperature as references.

4 Major Sources of Error

In high accuracy work, one has to consider and control many sources of error. However, the difference principle and the use of accurate and proven retro-modelling eliminates most of the first-order errors. What remains are the kinds of errors addressed in the companion paper [1] – and the inaccuracy of the Debye relaxation data of the reference liquid (water) used to determine the sample tube inner diameter.

Another kind of error is related to what dielectric properties the major residual constituent of the sample liquid has. The liquids used here have been chosen to also minimise any such effects, but some uncertainty about the typical 0.5% of "other" liquid remains. Dissolved water may pose the most significant uncertainty for low-ε″ liquids.

4.1 Temperature

This is addressed in the companion paper [1].

4.2 Influences by the Axial Insertion Holes

Reference is again made to the companion paper [1]. Since εϖ of the liquids used here is lower than for water, the influence by the various aspects of the insertion hole and the sample placement become less. There is, however, a much lower εϖϖ of DMF than of water, which means that only hole microwave mode evanescence and not additional wave absorption determines the overall influence. Therefore, possible problems with DMF at the high frequency must be observed.

4.3 Reference Choices

Reference is again made to the companion paper [1]. Water with the following data was used.

\[ t = 20 \, ^\circ C \quad \varepsilon_S = (80,30 \pm 0,10) \quad \varepsilon_\infty = (5,0 \pm 0,2) \quad f_D = (16,8 \pm 0,6) \, \text{GHz} \]  

The measurements of ε’ at the low frequency was more accurate than at the high, due to higher Q values, less disturbances by the insertion holes and sample centring, and a more linear frequency change function. Therefore, the low frequency values (including Debye calculations) were used for determination of the ε’ value of DMF at the high frequency and room temperature (+19,75 °C), instead of a calibration using water.

5 Procedure and Measurements

The sample diameter is an important parameter: a larger diameter provides a better resolution of both ε’ and ε″, but the transmission attenuation may become too large and disturbed by spurious modes and instrument noise for high ε″ values. A smaller diameter gives smaller
changes of resonance frequency and transmission damping. A nominal diameter of about 3 mm was chosen as a compromise, since only one diameter was to be used so that measurements at both frequencies could be made simultaneously – and since water was also to be measured in the same program. The instrumentation was the same as described in the companion paper [1]. Measurement temperatures were about +40, +20, +5, -5 °C, but since some of the liquids have dielectric properties which are not representative for frozen substances with a high water content, measurements at -5 °C were not made.

6 Results, Calculations and Discussion

The Debye equation for the complex \( \varepsilon \) becomes, with \( f, \varepsilon_s, \varepsilon_\infty \) (for \( f = 0 \) and \( \rightarrow \infty \), respectively) and the distribution parameter \( \alpha \) as usual:

\[
\varepsilon = \varepsilon_\infty + \frac{\varepsilon_s - \varepsilon_\infty}{1 + j(f / f_D)^\alpha}
\]

(1)

It is employed in both forward and reverse directions, analytically.

The permittivity values in the following tables are not rounded. The repeatability was much better than the accuracy, and the latter is given in all individual cases. The temperature inaccuracy was ± 0.2 K, as addressed in the companion paper [1].

6.1 Pyridine

The liquid was manufactured by Merck, with a stated purity of 99.5 %, <0.2 % alpha-picoline and <0.1 % water. The measurement and retro-modelling results are summarised in Table 1. Using these values, the Debye parameters \( \varepsilon_s \) and \( f_D \) are given in Table 2. \( \varepsilon_\infty \) is set to 2.30 and temperature independent – that value is taken from [5].

It is seen that the \( \varepsilon' \) values at the low frequency are lower by about 0.2 units for all temperatures except +19.75 °C. Since the low frequency values are more reliable, they are used for the following fit:

\[
\varepsilon_\infty = 2.30 \quad \varepsilon_s = 1/(0.68078 + r \cdot 0.0003277)
\]

All residuals are < 0.007.

As to \( f_D \), the agreement between the low and high frequencies is quite good and the averages at each temperature are used to construct the fit. One gets

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Frequencies (MHz)</th>
<th>( \varepsilon_s ) : ( f_p ) low freq. (GHz)</th>
<th>( \varepsilon_s ) : ( f_p ) high freq. (GHz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>+40,65</td>
<td>950/2271</td>
<td>12,28;39,18</td>
<td>12,56;38,40</td>
</tr>
<tr>
<td>+19,75</td>
<td>949/2271</td>
<td>13,42;29,62</td>
<td>13,41;28,81</td>
</tr>
<tr>
<td>+5,0</td>
<td>949/2271</td>
<td>14,35;21,66</td>
<td>14,43;23,37</td>
</tr>
<tr>
<td>-5,2</td>
<td>949/2270</td>
<td>15,06;17,341</td>
<td>15,33;19,07</td>
</tr>
</tbody>
</table>

Table 1 Measurement and retro-modelling results for pyridine

Table 2 Calculated \( \varepsilon_s \) and \( f_D \) values for pyridine
\[ f_D = 20,385 + t \cdot 0,4508 \quad \text{(GHz)} \]

All residuals are <0,2 GHz. The approximation indicates that \( f_D \) becomes 0 at about – 45 °C.

Using the literature results in [5] one obtains at 19,75 °C and 949 MHz: \( \varepsilon = 13,53 - j0,491 \); at 2271 MHz the result becomes \( \varepsilon = 13,42 - j1,165 \). These are difficult to compare with the present results, since the single reference is from 1955 and the liquid purity unknown. The probable residual (picoline) has similar \( \varepsilon' \) but significantly lower \( f_D \), which will result in a higher \( \varepsilon'' \).

The inaccuracy of \( \varepsilon' \) at the low frequency is less than ±0,1; it is less than ±3 % for \( \varepsilon'' \) over the frequency band.

### 6.2 Dimethylformamide

The liquid was manufactured by Merck, with a stated purity of >99,9 % and <0,02 % water. The measurement and retro-modelling results are summarised in Table 3. Using these values, the Debye parameters \( \varepsilon_s \) and \( f_D \) are given in Table 4. Note that \( \varepsilon_{\infty} \) is set to 4 and temperature independent; the influences by a different \( \varepsilon_{\infty} \) are insignificant, since \( f_D > f \) here.

The \( \varepsilon_s \) agreements between the values for the two frequencies in Table 4 is good. Therefore, a simple average is taken for the following fit:

\[ \varepsilon_{\infty} = 4 \quad \varepsilon_s = 14,754 + 27,246 \cdot \exp(-t/133,62) \quad (4) \]

As to \( f_D \), curvefitting clearly shows that the low frequency data are more reliable. They are therefore used for the following fit:

\[ f_D = 1,303 + 9,747 \cdot \exp(t/56,012) \quad \text{(GHz)} \quad (5) \]

The inaccuracy of \( \varepsilon' \) at the low frequency is less than ±0,3; it is estimated to be less than ±4 % for \( \varepsilon'' \) over the frequency band.

### 6.3 Methanol-water Mixture

The methanol (MeOH) was manufactured by Merck, with a stated purity of >99,8 % and with <0,05 % water and <0,1 % ethanol. Since a mixture of 60 % MeOH and 40 % H2O by volume has been used "historically" [4] as a calibration substance for dielectric measurements on foods at 2450 MHz – and has suitable dielectric properties at that frequency – the same mixture was used in this work. However, weighing the liquids is more reliable, so preparation was now by weighing in 65,0 g...
MeOH and 45.0 g deionised water. This is equivalent with less than 0.5 % difference. The measurement and retro-modelling results are summarised in Table 5.

Different $\varepsilon_{\infty}$ values were tested, for obtaining the best possible agreement with a single Debye relaxation for the mixture, with $\alpha = 0$. It was found that $\varepsilon_{\infty} = 5$ gave the best results. $\varepsilon_s$ and $f_D$ values are given in Table 6. – The agreement between the values obtained with low and high frequency is quite good, but it is seen that using $\alpha \neq 0$ and another $\varepsilon_{\infty}$ is likely to provide an even better agreement. Measurements at additional temperatures would then be needed.

In view of the significantly smaller influence by $f/f_D$ at the low frequency, the $\varepsilon_s$ values 48.6; 53.8 and 59.6 at the three temperatures and low frequency are for now used to obtain the following fit:

$$\varepsilon_{\infty} = 5.0 \quad \varepsilon_s = 66.08 - 2.271^t$$

As to $f_D$, the agreement between the low and high frequencies is quite good, and the averages at each temperature are used to construct the fit. One gets

$$f_D = \exp(1.0055 + 0.02656^t)(\text{GHz})$$

The inaccuracy of $\varepsilon_s$ is less than ±0.4; it is less than ±4 % for $\varepsilon_{\omega\omega}$ over the frequency band.

## References

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ABSTRACT. A method is proposed to utilize facilities of modern admittance analysers and network analysers to measure not only the complex admittance or reflection coefficient of a dielectric sensor, but also their derivative with respect to frequency. A dielectric sum function is obtained by such mode of operation which can significantly facilitate the evaluation of measured spectra. Especially investigations into the dielectric properties of conducting samples may greatly benefit from the derivative spectrometry, since the sum function does not contain the conductivity term which normally interferes with the dielectric terms and thus obstructs the analysis of data.

Keywords: permittivity, conductivity, relaxation, electrolytes

1 Introduction

The dielectric properties of liquid systems are of great significance for our fundamental understanding of the structure and microdynamics of complex fluids [1,2] as well as for many applications. The widespread use of electromagnetic wave interactions with materials for moisture sensing and non-destructive water content determinations [3-5] is an example of current applications. Other examples are microwave heating and microwave-induced reaction enhancement in chemistry [6,7]. Further current interest in electromagnetic wave interactions with liquids, particularly with aqueous systems, springs from the global distribution of modern communication facilities, calling for detailed insights into the dielectric properties of the biosphere [8,9].

In the frequency domain the dielectric properties of matter are normally expressed in terms of the (relative) electric permittivity

$$\varepsilon = \frac{P}{(\varepsilon_0 E)} + 1$$  \hspace{1cm} (1)

that relates the polarisation $P$ to the electrical field strength $E$. In Eq.(1) $\varepsilon_0$ denotes the electrical field constant and underlined quantities are vectors. In order to account for relaxation phenomena, as resulting from intermolecular forces, a complex permittivity ($i^2 = -1$)

$$\varepsilon(\nu) = \varepsilon'(\nu) - i\varepsilon''(\nu),$$  \hspace{1cm} (2)

dependent upon frequency $\nu$, is used in which the real part $\varepsilon'(\nu)$ represents the polarisation component in phase with $E(\nu)$ and the negative imaginary part $\varepsilon''(\nu)$ represents the contribution with a $\pi/2$ phase shift. With conducting samples the electrical conductivity $\sigma$, defined by the current density

$$j = \sigma E,$$  \hspace{1cm} (3)

also contributes to the polarisation current with $\pi/2$ phase shift. The contribution to the complex permittivity is usually described by the relation ($\omega = 2\pi\nu$)
\[ \varepsilon'_\sigma (v) = \frac{\sigma_o}{(\varepsilon_o \omega)}, \]  \hspace{1cm} (4)

assuming a frequency independent conductivity \( \sigma_o \). Hence for conducting samples the total negative imaginary part \( \varepsilon''_{tot} = \varepsilon'' + \varepsilon''_{\sigma} \) is given by the sum of the dielectric contribution and the conductivity contribution, with serious consequences for the study of the relaxation phenomena at which dielectric spectrometry particularly aims.

Fig. 1 Real part \( \varepsilon' \) and negative imaginary part (\( \bullet \), total; \( \circ \), dielectric contribution) of the complex permittivity spectrum of a 0.1 mol/l \( \text{Sc}_2(\text{SO}_4)_3 \) solution in water at 25 °C [10]. The inset shows a complex plane representation of the data.

In Fig. 1 the real part and negative imaginary part of the permittivity of an aqueous electrolyte solution, with \( dc \) electric conductivity \( \sigma_o = 1.2 \) S/m, are displayed as a function of frequency \( \nu \) and also in a complex plane representation. Two dispersion (\( \frac{d\varepsilon'(\nu)}{d\nu} < 0 \)) / dielectric loss (\( \varepsilon''(\nu) > 0 \)) regions emerge, indicating a low frequency relaxation at around 0.7 GHz, as resulting from reorientational motions of ion complex structures, and a high frequency relaxation at around 20 GHz which is due to the dipolar solvent. In the total negative imaginary part \( \varepsilon''_{tot} \) the dielectric contribution \( \varepsilon''(\nu) \) is largely masked by the conductivity contribution \( \varepsilon''_{\sigma}(\nu) \), which, in this example, exceeds \( \varepsilon''(\nu) \) at 10 MHz by a factor of \( 10^4 \). Hence the extraction of \( \varepsilon'' \) as the difference \( \varepsilon''_{tot} - \varepsilon''_{\sigma} \) from the measured \( \varepsilon''_{tot} \) data requires an
extremely high accuracy in the measurements. Especially the temperature $T$ has to be precisely known and kept constant during the measurements. Due to the strong dependence of $\sigma$ upon $T$, the small temperature variation $\Delta T = 0.005$ K results already in a change $\Delta \varepsilon''_{\text{tot}}$ of $10^{-4}$ in the total imaginary part, corresponding with a variation by 100 % in the dielectric contribution $\varepsilon''$ at 10 MHz.

In this article a method is proposed to substantially reduce errors by temperature fluctuations and to thus increase the sensitivity in the dielectric loss determinations.

### 2 Conceptions of Dielectric Loss Determination

Early attempts to reduce small differences in the temperatures of the sample permittivity and conductivity determinations proceeded from the use of a single cell to measure $\varepsilon(\nu)$ and $\sigma_0$ simultaneously [11]. At shielded open-ended coaxial line probe, as sketched in Fig. 2, is suitable for this purpose.

![Fig. 2 Sketch of a shielded coaxial line cell of the cut-off variety, provided with electrodes (4) for low frequency conductivity measurements [11]. 1, feeding coaxial line; 2, matched dielectric window; 3, sample volume, essentially consisting of a coaxial line with length $l$ and a short piece of circular waveguide.](image)

Using this probe the total complex permittivity $\varepsilon_{\text{tot}}(\nu) = \varepsilon'(\nu) - i\varepsilon''_{\text{tot}}(\nu)$ is obtained from the input impedance or reflection coefficient of the cell which essentially consists of a coaxial line/circular waveguide transition, the liquid-filled waveguide being excited below the cut-off frequency of its $TM_{01}$ mode. For the conductivity measurements two additional electrodes are inversed in the liquid. The distance of these electrodes from the coaxial line/waveguide transition is sufficiently large to avoid interference of the conductivity measurements with the high-frequency evanescent field that fringes into the waveguide-below-cut-off section. If
temperature gradients within the cell are small $\sigma_o$ and thus $\varepsilon''_o$ can be determined at nearly the same temperature as $\varepsilon''_{tot}$. Since different electrodes and different electronic set-ups are used for the measurement of $\varepsilon''_{tot}$ and $\varepsilon''_o$, however, experimental errors, due to electrode polarisation layers and imperfections of the electronic devices, may not compensate. Another limitation of this approach is its restriction to dielectric measurement cells that allow for an introduction of conductivity electrodes.

For the above reasons the electric $dc$ conductivity $\sigma_o$ is usually treated as an unknown parameter in a nonlinear least-squares regression analysis of the experimental spectra in terms of relaxation function. The drawback of such evaluation procedure is the use of the large total loss $\varepsilon''_o(\nu)$ instead of the relevant dielectric loss $\varepsilon(\nu)$, leading to a reduced effect of the relaxation parameters, at which the analysis aims, in the reduced variance

$$\chi^2 = \frac{1}{N - P - 1} \sum_{n=1}^{N} \left[ \frac{\left( R'(\nu_n) - \varepsilon'(\nu_n) \right)}{\Delta \varepsilon'(\nu_n)} + \frac{\left( R''_{tot}(\nu_n) - \varepsilon''_{tot}(\nu_n) \right)}{\Delta \varepsilon''_{tot}(\nu_n)} \right]^2$$

that is minimized in the fitting procedure. In Eq.(5) $\nu_n$, $n = 1, \ldots, N$, denotes the frequencies of measurement, $P$ is the number of the adjustable parameters of the model relaxation spectral function $R(\nu) = R'(\nu) - iR''(\nu)$, and $\Delta \varepsilon'$, $\Delta \varepsilon''_{tot}$ are the experimental errors used as inverse weighing factors.

The alternative approach, that is proposed here, proceeds again from an experimental determination of the conductivity contribution. It is not restricted to a special cell type and thus includes the widely used easy-to-handle flanged open ended coaxial sensor [12] sketched in Fig. 3.

Fig. 3 Sketch of a flanged open-ended coaxial line sensor with “infinite half space geometry” [12]. 1, feeding coaxial line; 2, matched dielectric window; 3, sample.

In the low frequency regime, in which we are predominantly interested, both the shielded (Fig. 2) and the flanged (Fig. 3) open ended sensor can be represented by the simple equivalent network given in Fig. 4, with the conductivity contribution shown by the resistance

$$R_o = 2\pi\nu\varepsilon\sigma C = \sigma_o C / \varepsilon_o.$$  

(6)
Fig. 4 Lumped element circuit representations of the coaxial line sensors shown in Figures 2 and 3.

Here $C$ is the frequency independent capacity of the cell. In Fig. 4 $C_0$ denotes the capacitance of the short piece of feeding coaxial line. The admittance of the cell, filled with the conducting liquid, is thus given by the relation

$$Y(\nu) = i 2\pi \nu \left[ \epsilon'(\nu) C - i \epsilon''(\nu) C + C_0 \right] + \sigma_o C / \epsilon_o$$

(7)

Since the last term of this equation does not depend upon frequency, the conductivity contribution can be eliminated by a determination of the admittance not just at the measuring frequency $\nu_n$ but also at a neighbouring frequency $\nu_n + \Delta \nu_n$ with $\Delta \nu_n << \nu_n$. At appropriately chosen $\Delta \nu_n$ the quotient $(Y(\nu_n + \Delta \nu_n) - Y(\nu_n)) / \Delta \nu_n$ can be used to calculate the derivative

$$\frac{dY(\nu)}{d\nu} = 2\pi i C \left[ \nu d\epsilon(\nu) / d\nu + \epsilon(\nu) \right] + 2\pi i C_0$$

(8)

which, obviously, is independent of $\sigma_o$. Again elimination of the conductivity contribution results from two measurements. Since these measurements, however, are performed in a split second, using the same cell and the same electronic equipment, almost perfect elimination of the conductivity contribution can be expected, if modern admittance analysers or network analysers are utilised. The latter usually determine the reflection coefficient

$$r = (Y_o - Y) / (Y_o + Y)$$

(9)

of the cell rather than the cell admittance $Y$ itself. Here $Y_o$ is the frequency independent characteristic admittance of the feeding line. It has been shown by simple arguments [13] that the derivative $dr/d\nu$ of the complex reflection factor likewise yields the “conduction-free” sum function

$$S(\nu) = \nu d\epsilon(\nu) / d\nu + \epsilon(\nu)$$

(10)

as contained in the brackets of Eq.(8).
3 Discussion

The first term on the right hand side of eq.(10) is the logarithmic derivative

\[ D(\nu) = d\epsilon(\nu)/d\ln\nu = \nu d\epsilon(\nu)/d\nu \]  

(11)
of the complex permittivity. Evaluation of the logarithmic derivative function, calculated from experimental permittivity spectra, has been proposed [14,15], because the real part \( D'(\nu) \) features characteristics of a conduction-free loss function. This aspect can be easily realised for a Debye-type relaxation function with conductivity terms

\[ R_{\text{tot}}(\nu) = \epsilon(\infty) + \frac{\epsilon(0) - \epsilon(\infty)}{1 + i\omega\tau} - i\frac{\sigma_o}{\epsilon_o\omega}, \]

(12)

for which

\[ D'(\nu) = -\frac{2(\epsilon(0) - \epsilon(\infty))\omega^2\tau^2}{(1 + \omega^2\tau^2)^2} = -\frac{2}{\epsilon(0) - \epsilon(\infty)} \epsilon'^2 \]

(13)
follows. Here \( \epsilon(0) \) and \( \epsilon(\infty) \) are the permittivities extrapolated to low and high frequencies, respectively. \( D'(\nu) \) extends over a somewhat smaller frequency range than \( \epsilon''(\nu) \). It thus allows for an easier separation of relaxation terms from complicated experimental spectra. This is a noticeable advantage beyond the elimination of the conductivity term.

For the relaxation spectral function \( R_{\text{tot}}(\nu) \) defined by eq.(12) the sum function \( S(\nu) = S'(\nu) - iS''(\nu) \) reads

\[ S'(\nu) = \epsilon(\infty) + \left(\epsilon(0) - \epsilon(\infty)\right)\frac{1 - \omega^2\tau^2}{(1 + \omega^2\tau^2)^2} \]

(14)

and

\[ S'(\nu) = \left(\epsilon(0) - \epsilon(\infty)\right)\frac{2\omega\tau}{(1 + \omega^2\tau^2)^2} \]

(15)

In Figures 5 and 6 graphs of the real part and negative imaginary part \( S' \), and negative imaginary part \( S'' \), respectively, are shown and compared to the real and negative imaginary parts of the original relaxation function \( R_{\text{tot}} \). In addition to the elimination of the conductivity contribution, the sum function features some favourable characteristics. Significant parts of the \( S \) spectrum are shifted to lower frequencies as compared to \( R_{\text{tot}} \). This shift may be utilized in the study of VHF and microwave relaxations with relaxation frequency close to the upper limit of the measuring range. Also the special shape of the \( S'(\nu) \) function, displaying a relative minimum (Fig. 5), may be helpful in the analysis of experimental spectra.
Fig. 5 Real parts $R'(\bullet)$ and $S'(\circ)$ of the model relaxation function defined by Eq.(12) and of the sum function $S$ (Eq.(14)), respectively, displayed versus normalised frequency $2\pi\tau\nu$.

Fig. 6 A log/log plot of the negative imaginary parts of the relaxation function $R_{tot}$ (Eq.(12)) and of the sum function $S$ (eq.(15)) versus normalised frequency $2\pi\tau\nu$. In this example $\sigma_0 = \varepsilon_0(\varepsilon(0) - \varepsilon(\infty))/(4\pi)$ has been assumed, corresponding with $\varepsilon''_\sigma(\omega\tau = 1) = \varepsilon''(\omega\tau = 1)$. Also shown by the dashed line is the graph of the dielectric contribution $\varepsilon''$ to $R''_{tot}$.

4 Conclusions

The determination of the admittance $Y$ or the reflection coefficient $r$ of a sample cell not just at the frequencies $\nu_n$ of measurement but also at neighbouring frequencies $\nu_{n+\Delta\nu_n}$ enables the determination of the derivatives $dY/d\nu$ or $dr/d\nu$, respectively, and thus of the dielectric sum
function $S = \varepsilon + (d\varepsilon / d\ln \nu)$. This sum function features some favourable properties which facilitate the evaluation of the measured data in terms of relaxation functions. A particularly appealing characteristic in the sum function of conducting samples in the missing of any conductivity contributions.

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Accurate Dielectric Properties of Liquid Water from –15 to +40 °C as Determined by Retromodelling of a Dual Resonant Applicator

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ABSTRACT. A critical analysis is made of literature reports on the dielectric properties of pure water from +40 °C down to supercooled at -20 °C, in principle in the frequency range 0 to 3 GHz. Differences among literature data are comparatively larger at 0°C and below. New measurements using complete multi-step numerical modelling of a dual resonant cavity at about 920 MHz and 2230 MHz are presented. The measured data at about + 20°C are used as reference for the calculation of data at other temperatures. Due to the high resolution and considerations of various error sources, the resulting accuracy becomes high and allows the construction of improved empirical formulæ for the Debye relaxation behaviour.

Keywords: water, supercooled, permittivity, microwave.

1 Introduction

Non-contaminated water in sealed glass capillary tubes can be supercooled down to about ^20 °C and then measured by for example the common TM010 transmission resonant cavity technique. However, accurate measurements of the absolute kind (where the dielectric data are obtained by only geometric and microwave resonance data) require very well-controlled sample and tube dimensions and elaborate corrections for sample insertion holes, coupling port properties, and losses in these and the cavity [1].

Cavity resonance methods can be designed for very good resolution. The overall accuracy depends on the applicability of the theoretical assumptions used for obtaining permittivity data, and how other factors such as sample tube data, conductivity of the cavity and its coupling devices, and insertion holes, are handled. In addition, data are until recently obtained only at one frequency per measurement applicator. Different samples then have then to be used for different frequencies, which may introduce an error factor.

Accurate measurements where the water is in metallic cells (coaxial line or waveguide) were among the first reported [2]. They offer possibilities of using frequency bands of up to a factor of 2. Drawbacks are the complicated machining/manufacturing of parts including windows, probes and seals. Also the requirements on the microwave instrumentation become quite complicated, since quite small amplitude and phase changes must be measured. However, with careful consideration of the several possible sources of error, accurate results have been obtained [3]. A drawback remains: due to the water directly contacting metal, significant supercooling of the water becomes virtually impossible.

To the knowledge of the author, there is only one comprehensive literature report on the microwave permittivity of supercooled water [4]. Measurements were made at 27,5 MHz and 9,4 GHz. Another report was made in the Soviet Union in the late 1970's [5], but details of the procedures are not available. – Data from both these reports are discussed here, together with those in report [3].

2 The Measurement Applicator

A resonant applicator which allows the use of two different frequencies with the same sample has been developed [6] (Fig.1). This property facilitates practical use and in essence
eliminates the major disadvantage with single frequency resonant measurement systems. A rotationally symmetrical cavity was chosen, to simplify precise manufacturing.

Since elongated (i.e. having a smaller diameter than height) non-magnetic samples couple best to an axial electric field, the basic cylindrical TM$_{010}$ mode should be used for the lowest frequency. However, since the lower useable frequency is to be around 900 MHz, such a circularly cylindrical applicator will have a diameter of about 200 mm. Therefore, a re-entrant-like design was developed, having an inwards-pointing conical section from the top of the circular wall, with an axially centred hole for sample insertion. Both the inductance and the capacitance of this distributed system then become larger than for the simple TM$_{010}$ resonator. This allows a resonator diameter of only 80 mm for a resonant frequency of about 940 MHz in empty condition. The active load height is then 15 mm and the basic insertion hole diameter is 10,2 mm for low-$\varepsilon'$ loads. It should be noted that this mode has zero magnetic field at the cavity axis, so the mode itself is quite insensitive to any magnetic properties of the sample.

The next item was to modify the cavity so that a higher frequency mode can also be used. This was by introducing a metal tube starting from the cavity bottom some radial distance away from the sample, and extending about halfway towards the cavity ceiling; the cavity inner height is about 70 mm. The function of this system cannot be deduced by analytical functions

- so design, analysis and optimisation were made by microwave modelling, using the commercial QW3D software [7].

- What happens is that a TM$_{011}$ mode is excited outside the inner metal tube, and then transforms into the TM$_{010}$ mode type inside and at the load. The resulting empty cavity resonance frequency becomes about 2300 MHz.

- Again, the mode is not sensitive to magnetic properties of the sample. Since the cavity modes are rotationally symmetrical, the tendency for coupled hybrid HEM modes to occur is also minimised.

Another major advantage with the system is that the resonance frequency changes with sample permittivity ($\Delta f/\Delta \varepsilon'$) become similar in MHz for both resonances; about 0.5 MHz/unit for a sample of 3.3 mm diameter. This difference would become much larger in a regular cylindrical cavity using the TM$_{010}$ and TM$_{020}$ resonances (the TM$_{110}$ mode is unsuitable since it has zero $E$ field at the axis).

The system has a further advantage: there is a third resonance at about 2415 MHz, which is not sensitive to a dielectric sample but gives a change of resonant frequency and transmission for magnetic samples. This property is not dealt with further here, but its use necessitates that the two cavity coupling loops are diametrically located.
3 The Retro-modelling Principle and Practise

A measurement applicator of the kind at hand cannot be calibrated by the use of analytical functions. Any perturbation theory would require very small samples for achieving a reasonable linearity allowing at least comparative measurements. Using very small (thin) samples has the drawback of small shifts in resonant frequency, attenuation or quality factor ($Q$ value) of the system; the instrumentation must thus be very stable and have a high resolution. – A cavity resonance method for using larger samples of a size practical of e.g. food substances at 2450 MHz has been used for many years [8]. It relies entirely on the accuracy of the dielectric data of several reference liquids. Certain assumptions on an "analytical" cavity behaviour must also be made.

By the use of numerical modelling techniques which are available since the end of the 1990's for use on modern PC's, and nowadays implemented as very accurate software, there is no longer a need for analytical function elaborations and small samples. Instead, basic limitations apply on the dynamic range of the measurement applicator in terms of mainly the transmission damping for lossy samples, and mode purity.

A chosen bandwidth waveform of a chosen duration was used as input, for extracting the transmission data (only amplitude as a function of frequency is used here). Circuits with sharp features in the frequency domain, or partially overlapping resonances, such as may occur with the measurement cavity, require considerably longer simulation times than broadband structures devoid of internal resonances. Semi-automated algorithms according to Prony's method [7], which is based on the generalised Pencil of Function Method [9] were selected here. The computations may then be speeded up by a factor of 5 to 10 under conditions with $Q > 100$.

The scenario (= the complete input data, including actual dimensions, material properties, frequency input type and bandwidth) also contains test data for the metal(s) conductivity. The first set of simulations is with completely empty cavity in a narrow band around the resonant frequency; one run for each resonance. Consecutive fine adjustments of the conductivity and coupling loops are then made, until the coupling factor and $Q$ values coincide with those measured. – Actually, this procedure entails the only principle difficulty: that of obtaining correctly balanced surface conductivities of the various parts of the cavity (walls and coupling loop regions, in this case). Adapting a single conductivity for agreement in transmission loss between the model and the real empty cavity is easy, but the surface current distribution changes with complex permittivity of the sample, modifying the overall surface current losses and by that changing the equivalent no-load transmission loss that is used here for determination of the sample loss factor. The quotient between these true empty and current distribution modified empty cavity transmission losses becomes less than 1 dB. In the present case, this quotient is due to the wire coupling loops being forced by the software to be superconducting. Test modelling using conductive metal loops indicates that the quotient becomes quite constant when the overall transmission quotient with/without lossy sample exceeds about 6 dB.

The next steps are measurements and modelling with empty glass vial. Its outer dimensions are known, but the inner dimensions need to be finally known with extreme accuracy, and are still only approximately known. Using these data and test values of the complex permittivity of the glass, new simulations are made until there is again agreement with the measured values. – The resonant frequency of the applicator is temperature dependent, which is of course taken into account by all resulting measurement data being differences between empty and filled vials. There is typically no need to consider the empty cavity data variation due to
thermal expansion in the modelling; unchanged cavity dimensions are used.

The following step is calibration (determination) of the inner diameter of the vial, using pure water at a carefully controlled temperature at about +20 °C. The best available literature data at the actually measured resonance frequencies with the water load are then used for changing the vial dimension in the scenario, until a good agreement is obtained.

FDTD modelling must be done with frequency-independent dielectric data of the sample. This is a more of a problem for \( \varepsilon' \) than for \( \varepsilon'' \). An equivalent conductivity \( \sigma \) rather than the loss factor \( \varepsilon'' \) must be used in the computations. Since the actual and modelled resonant frequencies of the measurement applicator deviate by less than 4 MHz and water at 20 °C is used for calibration, \( \varepsilon'' \) is linearly corrected in the computations.

In the final steps, the sensitivity factors of resonant frequency variation \( \Delta f/\Delta \varepsilon' \) and resonant transmission attenuation \( \Delta T/\Delta \sigma \) are modelled, around the specific values obtained by the measurements. All retromodelling [10,11] for obtaining the unknown permittivities of the following water samples with changed temperatures is done in analogy, with empty cavity measurements at each temperature as reference. It is of course too tedious to require an exact data match between measurement and modelling runs. The \( \Delta f/\Delta \varepsilon' \) function is linear over small intervals (but becomes \( \varepsilon' \) -dependent for large \( \tan \delta \) of the sample) – and the \( \Delta T/\Delta \sigma \) function can be simplified [8]. A modelled factor (typically between 9 and 19 dB, rather than the 20 dB "standard" value for an \( \varepsilon'' \) quotient of 2) now allows more precise approximations of \( \sigma \).

4 Major Sources of Error

In high accuracy work, one has to consider and control many sources of error. However, the difference principle and the use of accurate and proven retro-modelling eliminates most of the first-order errors – but the cavity conductivity distribution uncertainty problem mentioned in the previous section remains, unless goldplating or similar is made. What remains are errors in temperature, influences by the axial insertion holes – and the inaccuracies of literature data between about +20 °C. A fourth kind of uncertainty is due to possible inherent (diffraction) resonance phenomena at the sample itself. With the measurement frequencies, sample geometry and permittivities in this work, the only such possibility is the circular TM_{00} resonance. It is dealt with in section 6.

4.1 Temperature

This kind of error can easily be quantified, since the temperature variations of the primary Debye parameters \( \varepsilon_S \) and \( f_D \) are quite well known. The Debye relaxation frequency \( f_D \) is used here instead of the relaxation time constant \( \tau_D \), since \( f_D \) is more practical for approximating the complex \( \varepsilon \) variations with frequency. The Debye equation for the complex \( \varepsilon \) becomes, with \( f, \varepsilon_S, \varepsilon_\infty \) ( for \( f = 0 \) and \( \rightarrow \infty \), respectively) and the distribution parameter \( \alpha \) as usual:

\[
\varepsilon = \varepsilon_\infty + \frac{\varepsilon_S - \varepsilon_\infty}{1 + j(f/f_D)^{1-\alpha}}
\]

The \( \Delta \varepsilon_S/\Delta T \) factor is about 0.4 units (0.46 %) per K, at 0 °C. The \( \Delta f_D/\Delta T \) factor is about 3.9 %/K, at 0 °C. At the frequencies of 2.25 and 0.85 GHz and 0 °C, a temperature rise of 1K results in differences in \( \varepsilon \) of 0.023-j0.76 and 0.34-j0.333, respectively. Interestingly,
providing a temperature ramping of the sample can be used for obtaining an increased accuracy of $f_D$ at the temperature where the variation of \( \varepsilon' \) with temperature is zero. The reason for this is a compensation of the rising \( \varepsilon_g \) with decreasing temperature, by the lowering of \( \varepsilon_\infty \) as the factor \( f/f_D \) increases with decreasing temperature. As seen in Fig.2, this happens at about 0°C for 2.6 GHz, and thus at lower temperatures for lower frequencies.

4.2 Influences by the Axial Insertion Holes

The use of insertion holes through which the sample extends is a means of avoiding errors due to cavity top/bottom surface contacting, and sample expansion with temperature. These issues are indeed problematic for high-permittivity loads. Analytical theories for the influence of holes have been published [12], but the analysis does astonishingly not cover the practical case of a small high-\( \varepsilon' \) sample in a low-\( \varepsilon' \) vial, typically surrounded by air in the hole – i.e. a radially inhomogeneous filling. What happens in that case is that there is no longer a simple waveguide mode cut-off condition; a coaxial TEM-like mode is also created. This fact seems to have been overlooked in the otherwise very good work by Ho and Hall [1]. Their result is what should then be expected: a fictitiously slightly higher \( \varepsilon_\infty \) was obtained (the higher curve at the right in Fig.1). Since \( \varepsilon' \) increases much for the lower temperatures at the frequency 2653 MHz they used, the errors should then be reduced; this seems also to be the case (see Fig. 2).

There are two possibilities to control the influence: by using an axially short hole and allowing radiation by the sample and then making it so long and with the ends in free space that the length does not influence the radiation – or to have an axially long hole to cause an essentially complete absorption of the resulting attenuated modes, and the sample extending some distance beyond the opening so that the mismatching created at the hole exit plane becomes effective and essentially only a non-radiating nearfield remains. In the present system, the second alternative is preferred, since there is an inherent complication with closely located absorbing boundaries in FDTD algorithms; a larger scenario would be needed. The scenario can then be enclosed in metal. – A particular advantage with modelling for system evaluation and investigation in this context is that all fields at resonance can be studied and used to quantify the necessary accuracy of geometric conditions of sample length and position. With an insertion hole section of 6.2 mm diameter, the field decay rate in the axial direction was strongly reduced. After an investigation by modelling, metal tubes with this inner diameter and extending the insertion hole lengths to 31 mm was mounted on the measurement applicator (see Fig.1).

Another factor which was analysed is that the TEM-like mode in the region of the dielectric in the insertion holes does not have the compensating effect of a coaxial metal TEM transmission line (which results in no first order effects with a radially displaced centre conductor). Therefore, geometrical centring of the sample is crucial. Modelling using very fine cells was made to investigate this phenomenon – actually after it had been found that some initial measurement data for high-\( \varepsilon' \) samples at the higher frequency seemed questionable. The hybrid HEM_{11} mode is then excited and propagates into the hole region; it becomes necessary to keep a diameter 3 mm sample centred within 0.1 mm, and in addition, the dielectric line TM_{00} mode becomes partially resonant and causes a deterioration of the system resonant frequency discrimination. However, it can be shown that the sample \( \varepsilon' \) evaluation is not influenced by these effects (again by modelling, using fine cells in scenarios approaching today's practical usability of the FDTD and Prony methods, with about 4 million cells and 200 000 time steps, requiring about 8 h runtime on a 3 GHz computer for each scenario).
4.3 Inaccuracies of Literature Data Between about +20 °C. Reference Choice

There are numerous literature reports presenting accurate microwave permittivity data of water, but in most cases these do – like this report – firstly analyse some of the available data and then present some few own experimental data and (hopefully favourably) compare these with literature data. A tendency not to clearly separate own data from those of others often diminishes the degree of independence. On the other hand, a goal is also to achieve some consensus on what are the true data, so new reports are typically intended to strengthen rather than change the consensus.

In this context, it is of interest to note that the literature deviations are larger at +10 °C and below. A major set of reasons for this seems to be associated with the fact that $f_D$ gets closer to the commonly used measurement frequencies for low temperatures, resulting in a high tan δ; this in turn may cause some waveguide modes which are highly evanescent for low-loss media to combine into hybrid HEM modes, in particular in circular waveguide measurement cells. The simplest example is the circular $TE_{11}$ and $TM_{11}$ modes combining into the $HEM_{11}$ mode. The analytical calculations for these phenomena become quite involved [13], even more so with a highly lossy waveguide filling. What actually happens is also sensitive to the mode excitation system. It is believed that such phenomena in filled waveguide measurement cells, in combination with unaccounted differences in resonant cavity hole influences due to the coaxial mode propagation/absorption addressed above, are major sources of the deviations among literature data.

Illustrations from Ho and Hall [1] are used and extended in Figures 2 and 3, which show data from that and earlier works, as well as the resulting values from the algorithms by Kaatze [3], Bertolini et al [4] and the present work. Data which are better related to actual possible measurements at a single frequency (2653 MHz) are used. It can be concluded that the Kaatze data in this temperature interval are very likely to be accurate, corresponding to less than ±0,4 K error, probably less than ±0,25 K. For low MHz frequencies, $\varepsilon'$ data are still more accurate and likely to correspond to half these temperature errors. As a result of a critical analysis of literature data for pure water at +20,0 °C for microwave frequencies up to about 5 GHz, the following reference values are based on primary literature data on $\varepsilon_S$ and $\varepsilon_\infty$, and $f_D$ as calculated from the value $\varepsilon''_{\text{ref.1}} = 11.6\pm 0.4$ at 2653 MHz from Figure 2 and [14].

\[
\begin{align*}
\varepsilon_S &= (80.30 \pm 0.10)  \\
\varepsilon_\infty &= (5.0 \pm 0.2)  \\
f_D &= (16.8 \pm 0.6) \text{ GHz}  \\
t &= 20.00 \text{ °C}
\end{align*}
\]

In addition, no temperature dependence of $\varepsilon_\infty$ is assumed in the following. – An error of 0,1 units
in ε\textsubscript{s} corresponds to a temperature deviation of 0,3 K. An error of 0,2 units in ε\textsubscript{∞} influences mainly f\textsubscript{D} for f < f\textsubscript{D} and the major uncertainty is in f\textsubscript{D}. The changes of the international temperature scales since the end of the 1940's do not have any significant influence.

5 Equipment, Procedure and Measurements

The sample diameter is an important parameter: a larger diameter provides a better resolution of both ε’ and ε’’, but the transmission attenuation may become too large and disturbed by spurious modes and instrument noise for high ε’’ values. A nominal diameter of about 3 mm was chosen as a compromise, realising that f\textsubscript{D} might be too close to the higher resonant frequency of about 2250 MHz for temperatures below 0 °C.

A Hewlett-Packard network analyser 8720D (50 MHz - 20 GHz) was used and calibrated in bands of 15 to 30 MHz for improved accuracy. Controls of the attenuation measurement accuracy was made with a precision attenuator.

A temperature-controlled cabinet with inner fan and several heating and cooling settings was used, allowing efficient manual control within ±0,3 K as read by a fast thermocouple sensor in the cabinet interior airflow. Another, accurately calibrated, thermocouple was fixed in good contact with the measurement cavity, which is of thick brass and weighs about 2 kg. This, the waiting times as well as repeated measurements (other liquids were also measured in conjunction) ensured that the temperature fluctuations of the sample were within ±0,1 K. The cavity thermocouple was calibrated in a diphenylether cell (+26,86 °C; ITS90 value), an icebath and against a reference thermometer at about -20°C. The resulting total uncertainty of the cavity temperature was ±0,2 K.

The empty sample tubes were chosen to have very similar properties and dimensions, by microwave measurements when empty. However, different sets of inner diameters were used in the numerical evaluation. The samples were prepared using freshly deionised and ultrafiltered water which was quickly boiled and then filled into the borosilicate glass sample tubes, which were subsequently sealed with the seal never contacting the liquid column.

Data at +20° C were used as for determination of the actual sample diameter by retro-modelling of ε’. This in principle allowed an independent determination of ε’’ by retro-modelling, but this value was used to compensate for the cavity surface conductivity variations, as mentioned earlier.

Measurement temperatures were about +40, +20, +5, -5 °C, and some successively lower temperatures down to about -15 °C, at which at least one sample did not freeze.
6 Results and Calculations

The permittivity values in the following tables are not rounded. The repeatability was much better than the accuracy, and the latter is given in all individual cases. As stated above, the temperature inaccuracy was ±0,2 K.

The reference values in section 5 were used, together with corrections using the smoothed equations in [3], for sample tube calibration at the actual sample temperature of +19,75 °C and the measurement frequency 923 MHz. The (reference) result is given in Table 1. As an example, the resulting inner diameter of the single vial that could be used down to −15,3 °C turned out to be about 3,34 mm.

It was found that both measurements and modelling gave somewhat unstable resonances for water below 10 °C, at the higher frequency. This is explained by the underlying phenomenon of inherent circular TM00 resonance of the sample, which will have an impact on the response variable for the combination of actual frequency, sample permittivity and diameter. A first-order condition for this resonance can be pseudo-analytically calculated to be

\[ a_R \approx \left( \frac{2450}{f} \right) \cos^2 \delta \exp\left(3,02 - 0,589 \ln \varepsilon' \right) \]

(δ is the loss angle; f in MHz; object radius \( a_R \) in mm)

As an example, the resonant diameter for water at 20 °C at 2225 MHz becomes 3,39 mm. The result is that the TM00 resonance interferes to some extent with the system resonance and reduces the possibility of accurately calculating \( \varepsilon' \), but not so much \( \varepsilon'' \). Therefore, no \( \varepsilon' \) values are given here at the high frequency, but will be obtained later, using a smaller sample diameter.

One may then calculate \( \varepsilon_s \) and \( f_D \) from the low frequency values in Table 1 by the inverse of equation (1). However, the \( \varepsilon' \) values for ± 5 °C are first smoothed, by curvefitting to a suitable function such as \( \varepsilon' = a + b \exp (t/c) \), where \( t \) is the temperature. One then finds that the residuals are maximally 0,25, except for at \( t = -5,2 \) °C, where it is −0,75. \( \varepsilon' = 88,8 \) is then changed to 89,55. \( \varepsilon_s \) and \( f_D \) are then computed using also \( \varepsilon'' \); results are given in Table 2.

Using these, the following \( f_D(t) \) and \( \varepsilon_s(t) \) fits are obtained:

\[ \varepsilon_s(t) = \left( 0,0112844 + r \cdot 5,81145 \cdot 10^{-5} + r^2 \cdot 9,8178 \cdot 10^{-10} \right)^{-1} \]

\[ f_D(t) = \exp\left[\left( 2,18787 + r \cdot 0,05247 \right) / \left( 1 + r \cdot 0,0073768 \right) \right] \]
The residuals of $\varepsilon_S$ at all values in Table 2 are within 0.4 units, and those of $f_D$ are within 2.5%, for all temperatures except –15.1°C where it is 5%.

7 Discussion

The accuracy of results are dependent on the accuracy of the calibration data from the literature at +20°C, as specified in equation (2).

The single high frequency $\varepsilon''$ value at the high frequency is to be compared with that obtained by equations 1, 4 and 5: $\varepsilon''=16.33$. The deviation is about 2.5%, which indicates an influence by the TM00 sample resonance also with regard to the transmission damping.

The choice of the particular fitting equation for $f_D$ has an interesting property: prediction of a temperature where $f_D$ becomes 0. Equation (4) gives –136°C.

It is of interest to compare the resulting data from equations (3) and (4) with the literature data given in particular in [1], [4] and [5]. This is done as the variations of $\varepsilon'$ and $\varepsilon''$ with temperature at 2653 MHz, and is shown in Figures 2 ($\varepsilon'$) and 3 ($\varepsilon''$). It is seen that the values in this work are close to those by Kaatze around 0°C, and that the data from Bertolini et al give a lower $\varepsilon'$.

However, Bertolini et al have stated too optimistic accuracy values and other work referenced in their paper actually give higher $\varepsilon_S$ at lower temperatures, as in this work.

It is believed that the method and results presented here will provide a significant improvement of the reliability of supercooled water permittivity data at microwave frequencies.

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Dielectric Study of Temperature-Dependent Behavior of Bound Water in Grain

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ABSTRACT. Dielectric behavior of bound water in grain was investigated through measurement of the dielectric properties at microwave frequencies over a wide temperature range between –80 °C and +10 °C. Both \( \varepsilon' \) and \( \varepsilon'' \) were found to increase linearly with temperature with a characteristic slope change at around –20 °C.

Keywords: Bound water, dielectric properties, microwaves, temperature

1 Introduction

Water is an important component of many biological and nonbiological materials. It is sometimes divided into two categories: free water and bound water. From an electrical standpoint, behavior of free liquid water is well described by the Debye dielectric model [1] with a single relaxation at microwave frequencies. In contrast, little is known about the dielectric properties of bound water, which are often described as lying somewhere between those of liquid water and those of ice depending on the degree of binding of the water molecules. In granular materials such as cereal grain and oilseed, bound water is expected to have several modes of binding depending on the inner structure of the kernels, the amount of water available, and the nature of surrounding molecules. It is well established that water dominates the dielectric properties of materials containing water at microwave frequencies [1]. Recently, it has been shown that moisture content and temperature have interchangeable effects on the microwave dielectric properties of grain [2]. The dielectric properties measured for a given moisture content at high temperatures are those of a virtual higher moisture content at lower temperatures. This takes place because the mobility of the water-molecules dipoles, in response to the electric field, increases as the temperature increases, making their contribution to the overall polarization of the medium higher and increasing the losses at the same time. In contrast, the lower the temperature, the slower the action of the water molecules, and the losses decrease. Therefore, for a given moisture level, tracking the variations of the dielectric properties resulting from temperature change could lead to a better understanding of the dielectric behavior of bound water. There are different degrees of binding, and each water molecule may have up to three bonds (ice), depending on the structure and composition of the material and the amount of water available.

In this study, bound water behavior is examined through measurement of changes in dielectric properties resulting from temperature change. Two samples of wheat of given bulk density and moisture content were cooled to –80 °C and their dielectric properties were measured in free space between 2 and 18 GHz as their temperature gradually increased to room temperature. The variations of the two components of the relative complex permittivity, \( \varepsilon' \) and \( \varepsilon'' \), with both temperature and frequency were then examined.
2 Complex Permittivity Measurement

2.1 Sample Preparation

Two samples of hard red winter wheat, each weighing about 7 Kg, were prepared by spraying the desired amount of distilled water on them. They were placed in plastic bags and held in a chamber at 5 °C to equilibrate. After three days, the samples were taken out and tested for moisture content with an oven-drying technique [3]. One sample had a moisture content of 15.7%, wet basis, and the other had a moisture content of 23.6%. Two measurement series were conducted. In the first, the 15.7% sample was poured into a Styrofoam box of rectangular cross section that was then sealed with tape and placed in a freezer at about –80 °C. The sample was taken out three days later, and the two components of the relative complex permittivity, \( \varepsilon' \) and \( \varepsilon'' \), were measured as the temperature increased gradually to room temperature. For temperature measurement, a small hole was made in one side of the Styrofoam box to insert the probe for a digital thermocouple thermometer (± 0.5 °C accuracy) to the center of the sample. After the temperature was recorded, the probe was removed and the hole was sealed with tape. The measurement sequence was repeated every hour in the beginning and then every two hours for a total duration of about 40 hours. In the second measurement series, the same measurement procedure was applied to the 23.6% sample.

2.2 Free-Space Permittivity Measurement

Grains are random dense media, which are mixtures of at least three different dielectrics, namely air, dry matter and water. Therefore, the measured relative complex permittivity of a given sample of grain is the effective relative complex permittivity of the mixture [4]. For simplification, the effective relative complex permittivity will be referred to as the complex permittivity in the rest of the paper. At microwave frequencies, several techniques can be used to determine the complex permittivity [5]. In this study, a free-space transmission technique was used to determine the two components of the complex permittivity of wheat samples. The complex permittivity of each sample was determined from measurement of the scattering transmission coefficient \( S_{21} \) with a Hewlett-Packard 8510C vector network analyzer (VNA). For each sample, the sealed Styrofoam box was placed between two linearly polarized horn/lens antennas (BAE SYSTEMS model AHO-2077-N) providing a focused beam. The antennas, facing each other, were placed 37 cm apart on a wooden structure, which kept them well aligned. Special precautions were taken to ensure accurate measurements [6]. Fig. 1 shows the measurement setup. At each temperature and for each sample, microwave measurements were carried out between 2 and 18 GHz.

Assuming the samples are low-loss materials (\( \varepsilon'' \ll \varepsilon' \)), the two components of the complex permittivity are calculated as follows [7]:

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1 Mention of company or trade names is for purpose of description only and does not imply endorsement by the U. S. Department of Agriculture.
\[ \varepsilon' = \left[ 1 + \frac{\Delta \Phi}{360 \frac{c}{f}} \right]^2 \]  
\[ \varepsilon^* = \frac{\Delta A}{8.686 \pi d} \frac{c}{f} \sqrt{\varepsilon'} \]  

where \( c \) is the speed of light, \( f \) is the frequency, \( \Delta A \) is the attenuation, and \( \Delta \Phi \) is the phase shift attributable to the presence of the sample. The attenuation in dB and phase shift in degrees are calculated from the measured modulus, \( |S_{21}| \), and argument, \( \varphi \), of \( S_{21} \) as follows:

\[ \Delta A = 20 \cdot \log_{10}|S_{21}| \]  
\[ \Delta \Phi = \varphi - 360 \cdot n \]

where \( n \) is an integer to be determined [8]. Both \( \Delta A \) and \( \Delta \Phi \) are taken as positive numbers in equations (1) and (2).

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**3 Results and Discussion**

Results obtained at 8 GHz are shown for purpose of illustration in Fig. 2. Both the dielectric constant and loss factor increase with temperature. For both wheat samples a slope change can be observed around \(-20 \, ^\circ\text{C}\) with the change more pronounced for the sample with higher moisture content. This change is similar to, but less abrupt than, that of free water which takes place at \(0 \, ^\circ\text{C}\) because of phase change from solid (ice) to liquid. It could be interpreted as a change in the nature of bound water behavior from tightly bound to more loosely bound. Figure 3 shows the Argand diagram where the dielectric loss factor divided by density is plotted versus the dielectric constant divided by density. Data corresponding to both wheat samples are superimposed and form a straight line in the complex plane confirming previous observations [2]. The x-axis intercept corresponds to the dielectric constant divided by density of a sample with zero moisture content or a sample of any moisture at very low temperature. At this temperature, the mobility of the water-molecule dipoles is zero, and thus the

---

Fig. 1 Measurement setup
electromagnetic wave does not undergo appreciable attenuation because the dielectric loss factor is nearly zero.

Figures 4a and 4b show variations of the dielectric properties of the wheat sample of 23.6% moisture content at different frequencies. The dielectric response seems to be consistent at all three frequencies.

Fig. 2 Variation of the dielectric constant and loss factor of hard red winter wheat of indicated moisture content and bulk density with temperature at 8 GHz.
Fig. 3 Argand diagram for wheat samples at 8.0 GHz and temperatures from –80 °C to 11 °C.

Fig. 4a Variation of the dielectric constant of hard red winter wheat with temperature at indicated frequencies, $M = 23.6\%$, $\rho = 0.81 \text{ g/cm}^3$. 
Fig. 4b Variation of the dielectric loss factor of hard red winter wheat with temperature at indicated frequencies, $M = 23.6\%$, $\rho = 0.81 \, \text{g/cm}^3$.

4 Conclusions

The study of the temperature dependence of the dielectric properties of wheat indicates the existence of two types of bound water with a pronounced behavioral change around $-20 \, \text{°C}$. Examination of the thermal dielectric response of the sample with higher moisture content at different frequencies did not reveal any difference in the nature of the response of bound water observed at a single frequency. The data collected constitutes the basis for better understanding and modeling of the electrical behavior of bound water.

References


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Session 2

**Dielectric Properties of Biological Substances and Tissues**
Chairmen: P. Pissis, S. Yagihara

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2. **Water Effects in Hydrogels Studied by Dielectric Techniques**  
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4. **Dielectric Properties of Water Solutions with Small Content of Sugar and Glucose in the Millimeter Wave Band and the Determination of Glucose in Blood**  
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6. **Dielectric Properties of Human Semen at Microwave Frequencies**  
   A. Lonappan, G. Bindu, A. V. Praveen Kumar, V. Thomas, J. Yohannan, K. T. Mathew;  
   Kochi, India
ABSTRACT. A Stratified structure representing a simplified model of the human head irradiated by plane wave in the frequency range of 100 MHz – 300 GHz, is investigated in this paper. General electromagnetic formulation on the absorption properties of the layered head model is given. Exact analytical expressions are given for the plane wave complex reflection coefficient as a function of frequency at the interfaces of the stratified structure. Numerous curves are presented, exhibiting the nature of wave reflection from and power absorption by the stratified structure. Results highlight the position of maximum power absorption values and their dependence on frequency and dielectric parameters. The maximum absorption can occur in the frequency range of the most common used commercial available communication systems.

Keywords: Absorbed power density, human head, reflection coefficients, stratified medium.

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Water Effects in Hydrogels Studied by Dielectric Techniques

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ABSTRACT. Poly(hydroxyethyl acrylate), PHEA / silica nanocomposites were prepared by sol-gel techniques and investigated, as potential scaffold materials for tissue engineering, by scanning electron microscopy, dynamic mechanical analysis, water sorption/desorption from the vapor phase, dielectric relaxation spectroscopy and thermally stimulated depolarization currents techniques at various levels of relative humidity / water content. The silica nanoparticles are homogeneously distributed, forming a continuous phase, and reinforce significantly the polymer matrix, without affecting its hydrophilicity and, thus, its biocompatibility. Water is more homogeneously distributed, as compared to pure PHEA matrix, forming smaller clusters. The glass transition temperature of the nanocomposites is controlled by water content and is less affected by the concentration of silica nanoparticles.

Keywords: hydrogels, poly(hydroxyethyl acrylate), nanocomposites, water clustering

1 Introduction

Hydrogels based on hydrophilic crosslinked polymers may absorb large amounts of water preserving their integrity. Of special interest are hydrogels based on biocompatible polymers for applications as biomaterials in drug delivery systems, implantation, preparation of scaffolds for tissue engineering etc [1, 2]. For these and similar applications issues related with the organization (state) of water and the influence of water on the final properties of the hydrogel are of fundamental interest. Two different approaches have been followed in the past to investigate such issues. The first is based on the classification of water in hydrogels into different classes, depending on the experimental technique employed, such as freezable and non-freezable water, mobile, immobile and clustered water, and free and bound water [3, 4]. The properties of water, determined to a large extent by specific polymer-water interactions [3] and by geometrical confinement of water in the pores of the hydrogel [4], and the influence of water on the properties of the polymer matrix are different in the different classes of water. Within the second approach, results obtained with hydrogels are discussed and explained in terms of phase diagrams [5] and chemical exchange process between water protons and hydroxyl protons of polymer chains [6] and no resource is made to the concept of different classes of water.

In previous work we employed broadband dielectric relaxation spectroscopy (DRS) and thermally stimulated depolarization currents (TSDC) techniques, in combination with several other partly complementary techniques, to study water effects in biocompatible hydrogels based on poly(hydroxyethyl acrylate), PHEA [7-11]. In several cases PHEA was combined with a second, hydrophobic polymer in the form of blends, copolymers and, more successfully, interpenetrating polymer networks (IPNs) in order to improve mechanical stability [10, 11]. This work has been recently extended to include porous hydrogels, either homopolymers or IPNS, prepared by solution polymerization using either methanol or ethanol as diluents, aiming to improve water capacity [12]. In order to further improve mechanical stability, the concept of nanocomposites may be used. Polymeric nanocomposites have been shown by several investigators to exhibit, for the same filler fraction, by far better mechanical
properties than conventional composites (micro- or macrocomposites) [13]. Here we present preliminary results of our studies on PHEA hydrogels reinforced with silica nanoparticles. These nanohydrogels were prepared and investigated as potential materials for three-dimensional porous networks to be used as basis for the development of scaffolds for tissue engineering. Sol-gel techniques in the presence of the polymeric matrix were employed for preparing the nanohydrogels. Owing to the combination of the concepts of nanocomposites and of hydrogel in one material, several interesting questions arise with respect to the organization of water in relation with the hydrophilic/hydrophobic nature of the filler, the changes in free volume due to loosened molecular packing of the polymeric chains, interface and interphase effects etc. In addition to DRS and TSDC several other experimental techniques were employed to investigate the organization of water and the effects of water on the final properties of the nanocomposite hydrogels in relation to structure and morphology. These include electron microscopy, water sorption/diffusion techniques, differential scanning calorimetry and dynamic mechanical analysis. The results are discussed in terms of confinement of water in small volume, specific polymer-water interactions, interfacial effects and filler-water interactions.

2 Experimental

2.1 Materials

The nanocomposites were prepared in two steps. In the first step PHEA hydrogels were prepared following procedures described in previous work [7-9]. In the second step standard sol-gel techniques were employed to prepare silica nanoparticles in the presence of the PHEA hydrogel. Details of the preparation will be given elsewhere. The silica concentration in the dry nanocomposites was varied between 0 and 30 wt.%

2.2 Experimental Techniques

Standard equipment was used for scanning electron microscopy (SEM), thermogravimetric analysis (TGA) and dynamic mechanical analysis (DMA). For water content determination samples were allowed to equilibrate over saturated salt solutions in sealed jars at controlled relative humidities. The water content h, defined as the ratio of the weight of water in the hydrogel to the weight of the dry sample (dry basis), was determined by weighing. Dry weights were determined by drying in vacuum (5x10⁻² Torr) for 24 h at 333 K. DRS (10⁻²-10⁶ Hz) and TSDC techniques (-180 to 30 °C) have been described elsewhere [7, 8]. The less familiar TSDC technique corresponds to measuring dielectric losses against temperature at low frequencies of 10⁻²-10⁻⁴ Hz, i.e. in a range not easily available for DRS measurements. Moreover, the technique is characterized by high sensitivity and high peak resolving power [7].

3 Results and Discussion

3.1 Morphological and Mechanical Characterization

SEM micrographs showed an excellent dispersion of the nanoparticles in the matrix. Pyrolysis revealed that the nanoparticles form a continuous phase. Thus, the nanocomposites under
investigation may be considered as IPNs consisting of two networks, an organic and an inorganic one.

DMA measurements, to be reported in detail elsewhere, gave for the glass transition temperature \( T_g \) of the dry nanocomposites values in the range 10-20 °C (at 1 Hz), slightly shifting to higher temperatures and becoming broader on addition of silica nanoparticles. The main result, however, was the significant and systematic increase of the storage modulus of the nanocomposites in the rubbery phase up to about three orders of magnitude, as compared to pure PHEA.

### 3.2 Water Sorption

Figure 1 shows preliminary results of water sorption measurements of several nanocomposites at a few values of relative humidity at room temperature. The water content has been normalized to PHEA content. The main result in Fig. 1 is that the normalized water content does not depend on composition, i.e. hydrophilicity and thus biocompatibility of the nanocomposite hydrogels are not affected by the presence of the nanoparticles. It is interesting to note that similar results were obtained in previous work with IPNs consisting of PHEA and a second, hydrophobic polymer, which was used for improving the mechanical stability of the hydrogel [10, 11]. In addition, no hysteresis effects were observed in sorption/desorption measurements, indicating good performance of the hydrogels under investigation.

![Fig. 1 Water content vs. relative humidity](image)

Fig. 1 Water content \( h \), normalised to PHEA content, against relative humidity \( RH \) of the samples indicated on the plot at 25 °C. The line is a fit of Eq. (1) to the experimental data.

The sorption isotherms in Fig. 1 can be classified as type III according to Brunauer [14], indicating that the results should not be described in terms of free and bound water and of hydration layers. Instead, the water molecules are distributed randomly at the hydrophilic sites up to a critical water content \( h_c \), above which clusters begin to form. The data were quantitatively analyzed by following the analysis proposed by Brown [15]. This analysis combines conventional solution theory (Flory-Huggins) and cluster theory (Zimm and Lundberg) and provides an interpretation for the sorption of water molecules in polar materials [9]. The data in Fig. 1 were fitted to the equation [15]

\[
h = \frac{P_t a_w}{P_z + a_w}
\]  

(1)
where $\alpha_w$ is the water activity (equal here to relative humidity RH) and $P_1, P_2$ fit parameters. $P_1$ is equal to the critical hydration layer $h_c$ above which water clusters are formed, $h_c = P_1$. The inset to Figure 2 shows results for $h_c$ of the nanocomposites. The number of water molecules in a cluster $N_c$ at water content $h$ can be calculated from [15]

$$N_c = 1 + \frac{h}{h_c}$$

Figure 2 shows results for $N_c$ at $h = 0.9$. For comparison $h_c$ was estimated to 0.05 in pure PHEA hydrogels and $N_c$ to about 4.5 at RH = 0.9 [9]. We observe that at higher silica contents $h_c$ increases and $N_c$ decreases. Thus, water is more homogeneously distributed in the presence of the silica nanoparticles and water clusters are formed at higher water contents and are smaller, as compared to pure PHEA, i.e. clustering is suppressed in the nanocomposites. Although the quantitative analysis of the results suggests that water is sorbed only in the hydrophilic PHEA phase, it can not be excluded that the silica nanoparticles interact with and contribute to the good dispersion of water in the nanocomposites. This point, which is of interest also for silica nanocomposites prepared for other applications, will be further followed in future work by tailoring the hydrophilic/hydrophobic nature of silica used and the type of chemical treatment [13].

![Fig. 2 Number of water molecules in a cluster $N_c$ at RH =0.9 against silica content. The inset shows the critical water content $h_c$ against silica content.](image)

**3.3 Molecular Mobility**

Figure 3 shows results of DRS measurements, real, $\varepsilon'(a)$, and imaginary part of dielectric permittivity (dielectric loss), $\varepsilon''(b)$, at room temperature for samples equilibrated in air (RH around 0.5, i.e. with water contents between 0.05 and 0.10 (Fig. 1). The high values of $\varepsilon'$ and $\varepsilon''$ at low frequencies do not reflect bulk properties, being related with dc conductivity and space charge polarization, respectively [8]. The investigation of conductivity effects, in particular the temperature and water content dependence of dc conductivity, may reveal important information on polymer-water interactions and water-induced flexibility of the polymeric chains [8, 16]. A shoulder observed in $\varepsilon''(f)$ and, less clear, a drop in $\varepsilon'(f)$ in the kHz frequency region are related with the $\alpha$ relaxation, associated with the glass transition (dynamic glass transition [17]) of the PHEA matrix. The $\alpha$ relaxation, in particular at higher
water contents, is masked by conductivity, similar to many other polymers [17]. TSDC, which is less influenced by dc conductivity, offers then an attractive alternative to investigate the \( \alpha \) relaxation (see below). The increase of \( \varepsilon'' \) at higher frequencies in Fig. 3 arises from the secondary \( \gamma \) and \( \beta_{sw} \) relaxations, which are intimately related with polymer-water interactions [8]. The secondary relaxations will be studied in detail by both DRS, at lower temperatures than in Fig. 3 to shift the relaxations into the frequency range available for measurements, and TSDC in future work. \( \varepsilon' \) and \( \varepsilon'' \) in Fig. 3 do not change systematically with composition. This is due, at least partly, to the different water content of the samples at ambient conditions and the strong dependence of dielectric properties on water content [8]. Measurements at various temperatures and water contents, now in progress, are expected to shed more light on that point.

Fig. 3 Real \( \varepsilon'(\alpha) \) and imaginary part \( \varepsilon''(b) \) of complex dielectric permittivity against frequency \( f \) of the nanocomposites indicated on the plot at ambient temperature and relative humidity conditions

Fig. 4 TSDC thermograms (normalized depolarization current \( I \) against temperature \( T \)) in the glass transition region of the samples and the relative humidity conditions indicated on the plot

Figure 4 shows results of TSDC measurements on the nanocomposites in the region of the \( \alpha \) relaxation at three values of relative humidity. The depolarization current \( I \), normalized to the same polarizing field, sample surface area and heating rate, is a measure of \( \varepsilon''(T) \) at a frequency in the range \( 10^{-2} \) - \( 10^{-4} \) Hz, depending on heating rate and the activation parameters of the mechanism under investigation [7]. The TSDC peak temperature is a good measure of
the calorimetric glass transition temperature \( T_g \). Detailed TSDC studies in PHEA hydrogels have indicated that dc conductivity makes a significant contribution to the TSDC \( \alpha \) peak without affecting significantly the temperature position of the dipolar contribution \([7]\). We observe in Fig. 4 that the \( \alpha \) peak (and, thus, \( T_g \)) shifts significantly to lower temperatures on addition of water, this shift reflecting the strong plasticizing action of water on the glass transition and the corresponding \( \alpha \) relaxation studied in detail in previous work \([7-12]\).

![Fig. 5 Glass transition temperature \( T_g \) against silica content at three levels of relative humidity indicated on the plot](image)

The composition and the relative humidity dependence of \( T_g \) determined by TSDC is shown in Figure 5. The silica content dependence is rather weak, in particular at higher levels of relative humidity / water content. A possible explanation for this behavior is that any differences between the dry nanocomposites, associated with the presence of various amounts of silica nanoparticles and their effects on chain dynamics, are masked by the strong plasticization induced by water at higher water contents. Also, it can not be excluded that drying of the samples was incomplete at the drying conditions adopted in this work, resulting in different amounts of residual water in the samples considered as completely dry in Fig. 5. Detailed studies of the water content dependence of \( T_g \) in pure PHEA by DSC and TSDC have revealed that this dependence is particularly pronounced at the initial levels of hydration, in agreement also with phenomenological descriptions by Fox and by Couchman-Karasz \([9]\).

In order to further follow the dependence of \( T_g \) on silica content in the nanocomposites and evaluate that as a measure of the effects of the nanoparticles on molecular mobility and chain dynamics, we show in Figure 6 the dependence of dc conductivity \( \sigma_{dc} \) on \( T_g \) for the various compositions. \( T_g \) refers to the dry samples (data in Fig. 5), whereas \( \sigma_{dc} \) to ambient conditions (measurements in Fig. 3). It is interesting that a correlation is observed between \( \sigma_{dc} \) and \( T_g \) in Fig. 6 for the various compositions. Detailed studies in PHEA hydrogels at various levels of relative humidity / water content have shown that dc conductivity is governed by the motion of the polymeric chains and that the \( \sigma_{dc} \) values are determined, to a large extent, by the distance of the temperature of measurements from \( T_g \) \([8, 16]\). Bearing that in mind, the results in Fig. 6 suggest that real differences between the dry nanocomposites do exist and that the non-systematic change of their properties with composition observed throughout this work may be related with details of preparation, which should be better controlled. This point will be further followed in future work.
Fig. 6 Dependence of dc conductivity $\sigma_{dc}$ at ambient conditions on glass transition temperature $T_g$ of the dry nanocomposites indicated on the plot by their silica content.

TSDC measurements in pure PHEA hydrogels have shown that, under the conditions adopted for drying in this work, $T_g$ is around $–10$ °C [7], i.e. in the same range as in the nanocomposites in Fig. 5. Compared to DMA, the values of $T_g$ by TSDC are shifted to lower temperatures, the higher frequency of DMA measurements being the explanation for that shift. Much work has been devoted in recent years to the investigation of the composition dependence of chain dynamics and glass transition in organic-inorganic nanocomposites [18-20]. The shift to higher temperatures due to constraints imposed to the motion of the polymeric chains by the rigid nanoparticles, in particular in the case of covalent bonds between the two components [18], is often cancelled out, or even overcompensated, by increase of molecular mobility, i.e. shift of $T_g$ to lower temperatures, due to loosened molecular packing of the polymeric chains, resulting in increase of free volume. This point will be further followed in future work by systematic DSC, DMA and TSDC studies on the nanocomposites under investigation here at defined levels of relative humidity / water content.

4 Conclusions

The results obtained by the various techniques indicate that the PHEA/silica nanocomposites prepared by sol-gel techniques and investigated in this paper exhibit enhanced properties, as compared to pure PHEA, regarding their application as scaffold materials for tissue engineering. DMA measurements show that the polymer matrix is significantly reinforced by the nanoparticles, which, following SEM and pyrolysis studies, are homogeneously distributed, forming a continuous network of threads. Water sorption/desorption measurements show that the hydrophilicity and with that the biocompatibility of PHEA is preserved in the nanocomposites and that no hysteresis effects exist. Water molecules are more homogeneously distributed in the nanocomposites, forming smaller water clusters at the same relative humidity. It should be noted, however, that these results refer to water sorption from the vapor phase, where water activity remains always smaller than 1, and that, in future work, the materials should be tested also in water immersion experiments. The dynamic properties, investigated by dielectric techniques, are governed by water content, similar to the pure PHEA matrix, and to a lesser extent by the concentration of the nanoparticles.
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References


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Dielectric Relaxation Study on Water Structure Restricted in Rice Kernel

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ABSTRACT. Dielectric relaxation measurements were performed for rice kernels by time domain reflectometry with flat-end coaxial electrodes. Though there existed some difficulties to realize a good contact between surfaces of the electrodes and the kernels, the water content could be estimated from the relaxation curves. Dielectric measurements were also performed for rice kernels with various water contents, and not only the water amount but also dynamical behaviors of water molecules were obtained. The dynamical behaviors were explained from restricted dynamics of water molecules and also from a diagram of the relaxation time vs. the shape parameter of the relaxation curve.

Keywords: rice, microwave dielectric spectroscopy, water structure, heterogeneity

1 Introduction

Dielectric spectroscopy generally offers information of the molecular mechanism of dynamical structures and functions of various molecular complex systems. Some moist materials are easily studied by dielectric measurements because of the large value of dielectric constant of water, though there exist moist materials for which the electric conductivity masks the lower frequency side of relaxation processes. In former meetings of this international conferences, we have reported that appropriate equipments, electrodes, and analytical methods chosen for enough high and wide frequency measurements provided dielectric behaviors and the characteristic feature of moist materials [1-5].

However it is still difficult to make dielectric relaxation measurements with the flat-end coaxial electrodes especially for smaller and harder materials like kernels. The dielectric relaxation measurement with flat-end coaxial electrodes is one of effective experimental techniques to offer information of dynamical structures of water in rice kernel [6]. The advantage of the flat-end coaxial electrodes is that the size can be adjusted for suitable frequency range for regardless whether hard or soft materials [7,8]. One of our two purposes in the present report is to examine how enough accuracy of the water amount obtained from dielectric measurements is realized for rice kernels.

Dielectric behaviors are quantitatively expressed by the relaxation parameters, such as the relaxation time, the relaxation strength, and so on. The water content is described by the relaxation strength and the dynamical behavior is expressed by the relaxation time. The distribution of the relaxation time and/or the heterogeneity are described by shape parameters of the relaxation curves. Water content dependences of these relaxation parameters express characteristic features of moist materials. However it is not so easy to compare these dependencies for moist materials and to find out some method of characterization, since moist materials in different categories indicate different dependences. This difficulty is brought from the different water structures. For example, the relaxation time obtained for water included in aqueous systems usually shows slow dynamics, since water molecules are restricted and/or confined in the mixtures. However the degree of restrictions at even the same
concentration depends on the category. Our second purpose in the present paper is then to examine how the dielectric behavior obtained from measurements should be analyzed.

In the present report, dielectric measurements were performed for rice kernels with various water amount by time domain reflectometry (TDR) [6-8] method at room temperature. The relaxation time and the distribution obtained from fitting procedures are compared with other aqueous systems to consider the water structure.

2 Experimental

2.1 Materials

Rice was obtained from a farm (Nanto-shi, Toyama, JAPAN) and stored at 10°C in a sealed container for 6-12 months. Bran was removed from the rice kernel before measurements and the kernels are ground and polished by a glassy file, as the surface would be flat for a good contact with the flat-end coaxial electrodes. Rice kernels were kept under atmosphere of our laboratory for 3-5 hours before the dielectric measurements, after getting out from the container. Kernels thus prepared were misted with MilliQ water to change the water amount, and were kept at room temperature around 25°C for 30-40 minutes before TDR measurements.

2.2 Dielectric Measurements

TDR measuring system equipped a sampling head (Hewlett-Packard 54121A) and a mainframe of digitizing oscilloscope (Hewlett-Packard 54121B), covered the frequency range from 10MHz to 14GHz. Details of TDR measuring system and the procedure were described in former literatures [6-9]. Flat-end coaxial electrodes (diameter of outer conductor: 2mm; electric length: 0.15mm) was employed. The schematic diagram of TDR measuring system and the electrodes is shown in Fig. 1. Benzene was used as the standard sample for the difference method of TDR measurements. TDR measurements were performed for five rice kernels.

Fig. 1 Schematic diagram of TDR measuring system set up for the rice kernel.
kernels at first, and also for other five wet kernels, examining the contact between the surfaces of sample and electrode with the reflected signals shown by the oscilloscope.

3 Results and Discussion

3.1 The Relaxation Process Observed for Rice Kernel

The difficulty in a good contact often causes the experimental error. Figure 2 shows experimental results for rice kernels under the same condition. Even under the same condition, the kernel shows each different relaxation curve. It is difficult to know exactly at the present if the difference reflects the actual moisture content of each kernel or experimental error brought from experimental procedures in TDR measurements such as a contact between the sample and the electrodes. In the latter case of error, the cause of the error necessarily brings smaller values of dielectric constant and loss because of a gap between the electrodes and the sample brought from the lack of the good contact. Then it is reasonable to consider that dielectric relaxation curves with the larger values must be more reliable with the better contact. Our former result reported already [6] as the first experience is shown as the dot dashed line in Fig. 2. The first result had been obtained from a lot of experiments of TDR measurements and careful analysis. The comparison of the first result with the present relaxation curves with larger values of dielectric constant and loss shows a difference within ±15%, and this rough analysis allows us to choose the relaxation curve with larger values of dielectric constant and loss from some trials of dielectric measurements.

Fig. 2. Frequency dependence of dielectric dispersion (a) and absorption (b) curves for rice kernels at 25°C. Solid lines were calculated from the Cole-Cole’s relaxation function. ( ○: A, □: B, ◇: C, △: D, ▽: E, dot dashed line: Ref.6 )

Fig. 3. Frequency dependence of dielectric dispersion (a) and absorption (b) curves for wet kernels at 25°C. Solid lines were calculated from the Cole-Cole’s function with the dc conductivity. ( ○: F, □: G, ◇: H, △: I, ▽: J, dot dashed line: Ref.6 )
Figure 3 shows dielectric relaxation curves for rice kernels with various moisture contents. Solid lines were calculated from the Cole-Cole’s relaxation function and also dc conductivity brought from the higher water content. The moisture contents of rice kernels change even during trials of TDR measurements, and some trials cannot be a help to determine the reliable measurement. Even in this case, some experimental error can be estimated from results of fitting procedure to determine the relaxation parameters.

The fitting procedure was set up for a relaxation curve in which the relaxation processes can be described by the Cole-Cole’s relaxation functions as written by

\[
\varepsilon^*(\omega) = \varepsilon_\infty + \frac{\sigma}{j\omega \varepsilon_0} + \frac{\Delta \varepsilon}{1 + (j\omega \tau)^\beta}
\]

where \(\varepsilon_\infty\), \(\sigma\), \(\omega\), \(\varepsilon_0\), \(\Delta \varepsilon\), \(\tau\), and \(\beta\) are the permittivity at limiting high frequency, the dc conductivity, the angular frequency, the permittivity of vacuum, the relaxation strength, the relaxation time, and the shape parameter indicating the broadness of the relaxation curve, respectively. It is reasonable to consider that the high frequency relaxation process observed in moist materials is due to the rotational diffusion of water molecules. Cole-Cole type of the relaxation function is used as the third term of Equation (1) to express the relaxation process. Smaller \(\beta\) values (\(0 < \beta < 1\)) represent broader symmetric relaxation curves and the broader distribution of the relaxation time. The lower-frequency side of the relaxation process was not analyzed exactly because of the existence of the dc conductivity, and fitting procedures are necessary to remove the affect from conductivity to the relaxation parameters. The detailed analysis of our procedure for water structures and its adequacy have been

### Table 3.1  Relaxation parameters obtained for rice kernels under the same conditions.

<table>
<thead>
<tr>
<th>Rice</th>
<th>(\tau/\sec)</th>
<th>(\log (\tau/\sec))</th>
<th>(\Delta \varepsilon)</th>
<th>(\beta)</th>
<th>(\sigma/\text{S} \cdot \text{m}^{-1})</th>
<th>(\varepsilon_\infty)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>3.00\times10^{-10}</td>
<td>-9.52</td>
<td>9.1</td>
<td>0.38</td>
<td>0.0</td>
<td>2.0</td>
</tr>
<tr>
<td>B</td>
<td>3.18\times10^{-10}</td>
<td>-9.50</td>
<td>11.7</td>
<td>0.39</td>
<td>0.0</td>
<td>2.2</td>
</tr>
<tr>
<td>C</td>
<td>3.29\times10^{-10}</td>
<td>-9.48</td>
<td>10.4</td>
<td>0.38</td>
<td>0.0</td>
<td>2.3</td>
</tr>
<tr>
<td>D</td>
<td>3.29\times10^{-10}</td>
<td>-9.48</td>
<td>7.7</td>
<td>0.41</td>
<td>0.0</td>
<td>2.6</td>
</tr>
<tr>
<td>E</td>
<td>4.98\times10^{-10}</td>
<td>-9.30</td>
<td>10.9</td>
<td>0.37</td>
<td>0.0</td>
<td>1.4</td>
</tr>
<tr>
<td>Average</td>
<td>(3.54\pm0.81)\times10^{-10}</td>
<td>-9.46 ± 0.09</td>
<td>10.0 ± 1.6</td>
<td>0.39 ± 0.02</td>
<td>0.0</td>
<td>2.2 ± 0.4</td>
</tr>
<tr>
<td>Ref.6</td>
<td>2.63\times10^{-10}</td>
<td>-9.58</td>
<td>9.8</td>
<td>0.40</td>
<td>0.0</td>
<td>2.1</td>
</tr>
</tbody>
</table>

### Table 3.2  Relaxation parameters obtained for rice kernels with various water contents.

<table>
<thead>
<tr>
<th>Rice</th>
<th>(\tau/\sec)</th>
<th>(\log (\tau/\sec))</th>
<th>(\Delta \varepsilon)</th>
<th>(\beta)</th>
<th>(\sigma/\text{S} \cdot \text{m}^{-1})</th>
<th>(\varepsilon_\infty)</th>
</tr>
</thead>
<tbody>
<tr>
<td>F</td>
<td>2.53\times10^{-11}</td>
<td>-10.6</td>
<td>20.7</td>
<td>0.58</td>
<td>9.6 \times10^{-3}</td>
<td>1.5</td>
</tr>
<tr>
<td>G</td>
<td>4.02\times10^{-11}</td>
<td>-10.4</td>
<td>15.3</td>
<td>0.58</td>
<td>3.7 \times10^{-3}</td>
<td>2.3</td>
</tr>
<tr>
<td>H</td>
<td>4.70\times10^{-11}</td>
<td>-10.3</td>
<td>11.4</td>
<td>0.57</td>
<td>2.6 \times10^{-3}</td>
<td>2.7</td>
</tr>
<tr>
<td>I</td>
<td>2.52\times10^{-11}</td>
<td>-10.6</td>
<td>12.4</td>
<td>0.58</td>
<td>2.4 \times10^{-3}</td>
<td>2.4</td>
</tr>
<tr>
<td>J</td>
<td>3.52\times10^{-11}</td>
<td>-10.5</td>
<td>9.9</td>
<td>0.59</td>
<td>2.3 \times10^{-3}</td>
<td>2.4</td>
</tr>
<tr>
<td>Ref.6</td>
<td>2.63\times10^{-10}</td>
<td>-9.58</td>
<td>9.8</td>
<td>0.40</td>
<td>2.5 \times10^{-3}</td>
<td>2.1</td>
</tr>
</tbody>
</table>
reported elsewhere [5,6,8,10]. The relaxation parameters obtained from fitting procedures to the relaxation curves shown in Figs. 2 and 3 are listed in Tables 3.1 and 3.2.

3.2 Water Structure in Rice Kernel

In our previous works [6-8], assuming that the contribution of dipole correlation to the relaxation strength is same with that for pure water, the water content, $C$, has been estimated from only the relaxation strength for free water as

$$C = C_w \frac{\Delta \varepsilon}{\Delta \varepsilon_w}, \quad (2)$$

here $C_w$ and $\Delta \varepsilon_w$ are the density and the relaxation strength of pure water, respectively. In the case of rice kernels, values of 73.2 and 0.997 g/cm$^3$ obtained for pure water at 25°C were employed for each value. The water content thus obtained for each sample was listed in Table 3.3.

Table 3.3. Water contents obtained from equation (2) for rice kernels.

<table>
<thead>
<tr>
<th>Rice</th>
<th>Water content, $C$ / g·cm$^{-3}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>F</td>
<td>0.282</td>
</tr>
<tr>
<td>G</td>
<td>0.208</td>
</tr>
<tr>
<td>H</td>
<td>0.155</td>
</tr>
<tr>
<td>I</td>
<td>0.169</td>
</tr>
<tr>
<td>J</td>
<td>0.134</td>
</tr>
<tr>
<td><strong>Average of kernels A-E</strong></td>
<td><strong>0.136 ± 0.022</strong></td>
</tr>
<tr>
<td><strong>Ref. 6</strong></td>
<td><strong>0.133</strong></td>
</tr>
</tbody>
</table>

The relaxation time was plotted against the relaxation strength in Figure 4. The relaxation strength reflects the water amount as expressed by Equation (2). Then Figure 4 also shows the moisture dependency of the relaxation time. The relaxation time becomes smaller with increasing water amount, since the degree of the restriction of water molecules in rice kernel is decreased. This behavior is generally shown in moist materials as reported so far [1-5].

The Cole-Cole’s shape parameter, $\beta$, of the relaxation curve was plotted against the relaxation strength in Figure 5. This decrease of the $\beta$ value in the drying process is also a typical behavior of slow dynamics of water in moist materials.

However these characteristic behaviors shown in Figs. 4 and 5 cannot be easily compared with other materials, since various behaviors are shown in different manner for respective materials, even if the moist materials are classified into the same category, such as aqueous systems of low molecular weight liquids, polymers, and/or dispersion of small particles.
Recently we have proposed a new diagram of the relaxation time - shape parameter, \( \tau - \beta \), to examine water structures in various aqueous systems [11-13]. Experimental results of dielectric spectroscopy for various aqueous systems indicate characteristic behaviors on the \( \tau - \beta \) diagram. For example, the \( \tau - \beta \) diagram in Fig. 6 indicates some trajectories of moisture dependence with the plots of \( \tau \) and \( \beta \) values obtained from dielectric studies for aqueous polymer systems [11, 12].

The slow dynamics of complex liquids are generally interpreted from increasing size of cooperative rearranging region (CRR) [14]. Dielectric spectroscopy indicates that the relaxation function is described by the KWW function with the coupling constant, which is considered to indicate the CRR (cooperative rearranging region) size. In the case of water
included in aqueous polymer systems, the relaxation function for water is usually described
by the Cole-Cole’s equation, and the relaxation time distribution is represented by the
Cole-Cole’s shape parameter. The shape parameter is treated to evaluate the heterogeneity of
structures of aqueous systems. Though usually the relaxation time and the distribution are
simply treated as independent quantities, our recent studies on water structures in polymer
aqueous solutions suggests a relationship,

\[ \beta = \frac{d_G}{2} \ln \frac{\tau \omega_s}{\tau_0}, \]

where \( \beta \) is the Cole-Cole shape parameter, \( d_G \) is a spatial fractal dimension for relaxing units
interacting with the surroundings, \( \tau_0 \) is the cutoff time of the scaling time, and \( \omega_s \) is written as

\[ \omega_s = \frac{2d_E G^{2/d_G} D_s}{R_0^2}, \]

with the Euclidean dimension, \( d_E \), a geometrical coefficient, \( G \), usually taking about unity,
self-diffusion coefficient, \( D_s \), and the cutoff size of the scaling in the space, \( R_0 \). Dynamics of
free water are thus characterized by the fractal structure of the aqueous system with the
scaling concept.

Water in the rice kernel held in storage exhibits the plot at \( (\tau = 354\text{ps}, \beta = 0.39) \). Increasing
moisture changed the plot into larger values of the relaxation time and smaller values of the
shape parameters on the \( \tau – \beta \) diagram. The trajectory of plots shown on the diagram with
changing water content suggests how water takes the fractal structure in the rice kernel.
Comparing with other aqueous systems, the dynamical structure of water in rice kernel is
different from usual polymer solutions except for PAA/water systems. The PAA/water system
could not be prepared for larger polymer concentration range. Furthermore our recent study
on polymer gels and dispersion systems indicate some trajectories around that for rice kernels.
Then the water structure on rice kernels characterized by the \( \tau – \beta \) diagram shows the
similarity of fractal structures with water included in dispersion and gel systems.

It is concluded from the present study that more systematic and precise measurements on rice
kernels make it possible to know more detailed dynamical structures of water in kernels, and
are applicable to some neighboring research fields, such as food science, denaturalization of
biological polymers and organs, and glass transition of complex fluids. More comparisons
with other aqueous systems are expected to offer important information in these research
fields.

### 4 Conclusion

Dielectric relaxation measurements were performed to obtain information of dynamical
structures of water in rice kernels. Even difficulties in dielectric measurements of small, solid,
and conductive materials brought reliable results, and the dielectric spectroscopy suggested
the dynamical structures of water. The relaxation time obtained for water included in rice
kernels shows slow dynamics as shown for conventional aqueous systems, since water
molecules are restricted and/or confined in the kernels. Experimental results indicate a
characteristic behavior on the \( \tau – \beta \) diagram. Water in the rice kernel held in storage exhibited
around the plot ($\tau = 354\text{ps}$, $\beta = 0.39$), and increasing moisture changed the plot into an average value ($\tau = 34.6\text{ps}$, $\beta = 0.58$). The trajectory of plots shown on the diagram with changing water content suggests how water takes the fractal structure in the rice kernel. Comparison with other aqueous systems, the dynamical structure of water in rice kernel is more similar with aqueous dispersion and gel systems than aqueous solutions.

References


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Dielectric Properties of Water Solutions with Small Content of Sugar and Glucose in the Millimeter Wave Band and the Determination of Glucose in Blood

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¹Institute of Radio Engineering and Electronics RAS, Fryazino Moscow Region, Russia
²Samsung Advanced Institute of Technology, Yongin, Gyeonggi-Do, Republic of Korea

ABSTRACT. Investigations of the dielectric properties of sugar solutions, as well as blood imitators and blood, in the millimeter-wave range allow one to obtain valuable information on the possibility of real-time control of glucose concentration in blood using electromagnetic waves in the millimeter-wave ranges. These investigations are also of interest for other applications such as, for example, wine industry, determination of water content in oil, oil products, and other liquids.

Keywords: Dielectrics, solutions, blood, skin, millimeter, waves

1 Introduction

To determine the complex permittivity, i.e., \( \epsilon' \) and \( \epsilon'' \) of a medium using noninvasive methods, one has to measure two parameters of the reflected electromagnetic wave. Usually (see, for example, [1]), one employs a sophisticated and expensive vector network analyzers and measures the modulus, \(|r|^2\), and phase, \(\phi\), of the reflection coefficient, \(R = |r|^2 e^{i\phi}\) (\(|r|^2\) is the power reflection coefficient and \(i = \sqrt{-1}\)). However the measurement of the phase of the reflection coefficient is a rather difficult problem, and the measurement error amounts to \(\pm 5\%\). For this reason, common measurement techniques cannot be applied to the noninvasive determination of small concentrations of glucose in water. Here, we use a sufficiently simple scheme for determining \(\epsilon'\) and \(\epsilon''\) of a medium, which consists in measuring the modulus, \(|r_{\text{min}}|^2\), and frequency, \(f_{\text{min}}\), of a millimeter (MM) wave (\(f_{\text{min}}\) corresponds to the minimum of the reflection coefficient \(R_{\text{min}}\)) from the following structure: a plane-parallel matching plate made of a low-loss dielectric – a medium under measurement with high losses. We developed computer programs to calculate the dielectric properties of the medium under test from the measured \(|r_{\text{min}}|^2\) and \(f_{\text{min}}\), and experimental setups.

2 Measurement Method

To determine the real \(\epsilon'\) and imaginary \(\epsilon''\) parts of the complex permittivity of a medium under test, we used a simple scheme consisting in measuring the modulus, \(|r_{\text{min}}|^2 = R_{\text{min}}\), and frequency, \(f_{\text{min}}\), of MM waves corresponding to the minimum of the reflection coefficient from the following structure: a plane-parallel matching plate made of a low-loss dielectric – a medium under test with high losses (water, solutions, blood, and human skin). Figure 1 shows the power reflection coefficients of two media (e.g., a reference medium (water) and a water with \(\chi\%\) of glucose) against the frequency of the incident MM wave. In Fig. 1, \(R_{\text{min},0}\) and \(f_{\text{min},0}\) represent the minimal power reflection coefficient and the corresponding frequency of the reference medium (pure water). When \(\chi\%\) of glucose is added to the reference medium, one can see the power reflection coefficient and the corresponding frequency shift from \(R_{\text{min},0}\) and \(f_{\text{min},0}\) to \(R_{\text{min},\chi}\) and \(f_{\text{min},\chi}\), respectively. We can calculate \(\epsilon'_{\text{m,\chi}}\) and \(\epsilon''_{\text{m,\chi}}\) from \(R_{\text{min},\chi}\) and \(f_{\text{min},\chi}\) using well-known expression (1) for the reflection coefficient \(r^*\) from such structure [2]:
Two types of measurement methods have been developed for the frequency range from 30 to 100 GHz. The first one is a waveguide method (WM) in which a plane-parallel matching plate is inserted into a single-mode rectangular waveguide with H_{10} mode. The second is a quasi-optical method (QM) in which the plane-parallel plate is placed between a horn and the media under test. For the plates available, \( r_{\text{min}}^* \) for structures with pure water was measured to be less than -20dB. The values of \( \varepsilon'_w \) and \( \varepsilon''_w \) of pure water needed for calculation were borrowed from [3]

\[
\begin{align*}
\beta^* = & \frac{2\pi}{\lambda_0} n_p^* = \frac{2\pi}{c} \varepsilon'_p = \frac{2\pi}{c} (n_p + i\kappa_p).
\end{align*}
\]

Here, \( n_m^* \) is the complex refractive index of the medium under test and \( n_p^* \) and \( l_p \) are the complex refractive index and the thickness of the plane-parallel matching plate. \( \varepsilon' \), \( \varepsilon'' \) and \( n, k \) are related by the formulas

\[
\varepsilon' = n^2 \quad \text{and} \quad \varepsilon'' = 2n \kappa.
\]

Here, \( \lambda_0 \) and \( c \) are the free-space wavelength and the speed of light in free-space, respectively. If the reflection coefficient in Eq. (1) is zero at given frequency \( f_{\text{min,0}} \), the thickness, \( l_p \), and the refractive index, \( n_p \), of the plane-parallel matching plate must be

\[
n_p = \left( n_m^* + \frac{\kappa_m^2}{n_m - 1} \right)^{1/2}
\]
Here, $s$ is an integer, $s = 0, 1, 2, \ldots$.

### 3 Experimental Setup and Measurements of Solutions

The measuring setup based on SWR and attenuation panoramic meters is shown in Fig. 2.

![Fig. 2 Schematic diagram of the experiment set up.](image_url)

Using the setup shown in Fig. 2, we carried out measurements of the properties of glucose solutions in water and blood imitators (physiological solution: 0.9% NaCl in water) in the millimeter wave band for small glucose concentrations $W$ from 5 to 0.25% wt. Some measurement results obtained in the frequency range from 9 to 93 GHz are shown in Tables 1 and 2.

Our measurements have shown that the values of $\varepsilon'_w$ and $\varepsilon''_w$ are in good agreement with the results of [3, 7].

The main conclusions of these measurements are as follows:

1. The dielectric properties of glucose solutions in water and in a solution of NaCl in water are measured for the first time in a wide range of frequencies from 10 to 93 GHz for glucose concentrations of $W \leq 5\%$ wt.
2. It is established that, for frequencies below 80 GHz, the values of $\varepsilon'$ and $\varepsilon''$ for 0.9% NaCl are less than those for water. In the frequency interval from 80 to 93 GHz, the difference between these values substantially decreases.
3. Depending on the glucose concentration $W$ in water up to $W = 5\%$, one observes a decrease in $\varepsilon'$ and $\varepsilon''$ in the entire range of frequencies except for frequencies of 92-93 GHz, where the values of $\varepsilon''$ slightly increase as $W$ increases.
Table 1 Dielectric properties of glucose solutions in water at frequencies 9.3 - 92.7 GHz.

<table>
<thead>
<tr>
<th>$\varepsilon'$, $\varepsilon''$</th>
<th>9.3 GHz</th>
<th>28.1 GHz</th>
<th>31.3 GHz</th>
<th>37.0 GHz</th>
<th>42.7 GHz</th>
<th>48.1 GHz</th>
<th>62.6 GHz</th>
<th>77.4 GHz</th>
<th>83.5 GHz</th>
<th>92.7 GHz</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\varepsilon'_w$</td>
<td>60.7</td>
<td>24.3</td>
<td>21.8</td>
<td>18.4</td>
<td>14.6</td>
<td>14.5</td>
<td>10.8</td>
<td>8.63</td>
<td>8.29</td>
<td>7.78</td>
</tr>
<tr>
<td>$\varepsilon'_{0.5}$</td>
<td>60.4</td>
<td>23.8</td>
<td>21.8</td>
<td>18.1</td>
<td>14.4</td>
<td>14.4</td>
<td>10.7</td>
<td>8.53</td>
<td>8.19</td>
<td>7.76</td>
</tr>
<tr>
<td>$\varepsilon'_3$</td>
<td>58.3</td>
<td>23.0</td>
<td>21.6</td>
<td>16.2</td>
<td>14.0</td>
<td>14.0</td>
<td>10.4</td>
<td>8.28</td>
<td>7.60</td>
<td>7.55</td>
</tr>
<tr>
<td>$\varepsilon''_{w}$</td>
<td>34.0</td>
<td>31.5</td>
<td>31.0</td>
<td>27.8</td>
<td>24.8</td>
<td>24.8</td>
<td>18.9</td>
<td>15.21</td>
<td>14.02</td>
<td>13.13</td>
</tr>
<tr>
<td>$\varepsilon''_{0.5}$</td>
<td>33.8</td>
<td>30.7</td>
<td>30.1</td>
<td>27.2</td>
<td>24.6</td>
<td>24.5</td>
<td>18.6</td>
<td>15.15</td>
<td>13.91</td>
<td>13.05</td>
</tr>
<tr>
<td>$\varepsilon''_3$</td>
<td>32.9</td>
<td>30.2</td>
<td>29.4</td>
<td>26.3</td>
<td>23.7</td>
<td>23.7</td>
<td>17.9</td>
<td>14.58</td>
<td>13.39</td>
<td>13.32</td>
</tr>
<tr>
<td>$T, ^\circ C$</td>
<td>18</td>
<td>18</td>
<td>19</td>
<td>19</td>
<td>17.5</td>
<td>17.5</td>
<td>20</td>
<td>18</td>
<td>19</td>
<td>19</td>
</tr>
</tbody>
</table>

Table 2 Dielectric properties of glucose solution in 0.9% NaCl at frequencies 9.3- 92.7 GHz

<table>
<thead>
<tr>
<th>$\varepsilon'$, $\varepsilon''$</th>
<th>28.1 GHz</th>
<th>31.3 GHz</th>
<th>37.0 GHz</th>
<th>42.7 GHz</th>
<th>48.1 GHz</th>
<th>62.6 GHz</th>
<th>83.5 GHz</th>
<th>92.7 GHz</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\varepsilon'_\phi$</td>
<td>19.9</td>
<td>23.0</td>
<td>15.1</td>
<td>14.5</td>
<td>14.5</td>
<td>9.35</td>
<td>8.73</td>
<td>7.83</td>
</tr>
<tr>
<td>$\varepsilon'_{0.5} \phi$</td>
<td>20.0</td>
<td>23.0</td>
<td>14.8</td>
<td>14.4</td>
<td>14.4</td>
<td>9.25</td>
<td>8.29</td>
<td>7.81</td>
</tr>
<tr>
<td>$\varepsilon'_3$</td>
<td>20.3</td>
<td>22.7</td>
<td>14.4</td>
<td>13.6</td>
<td>13.9</td>
<td>9.16</td>
<td>8.02</td>
<td>7.60</td>
</tr>
<tr>
<td>$\varepsilon''_{w}$</td>
<td>31.7</td>
<td>32.0</td>
<td>26.1</td>
<td>25.1</td>
<td>25.0</td>
<td>18.57</td>
<td>14.13</td>
<td>12.94</td>
</tr>
<tr>
<td>$\varepsilon''_{0.5}$</td>
<td>31.4</td>
<td>31.5</td>
<td>25.8</td>
<td>25.0</td>
<td>24.8</td>
<td>18.51</td>
<td>14.05</td>
<td>12.85</td>
</tr>
<tr>
<td>$\varepsilon''_3$</td>
<td>29.7</td>
<td>29.7</td>
<td>24.8</td>
<td>23.7</td>
<td>23.7</td>
<td>17.74</td>
<td>13.90</td>
<td>13.30</td>
</tr>
<tr>
<td>$T, ^\circ C$</td>
<td>18</td>
<td>17</td>
<td>19</td>
<td>17.5</td>
<td>17.5</td>
<td>20</td>
<td>19</td>
<td>19</td>
</tr>
</tbody>
</table>

4. Note that, in the investigations of Japanese scientists [4,5] of glucose concentrations in physiological solutions in the frequency range 30—40 GHz, the values of $\varepsilon'$ increased, in contrast to our results, with the glucose concentration, whereas the values of $\varepsilon''$ decreased, like in our experiments. The measurements in [4] and [5] showed that the frequency dependence of $\varepsilon'$ exhibits different behavior. These facts can be attributed to the significant error in measuring the phase by a Vector Network Analyzer, which amounts to at least 5%.

5. The maximum sensitivity of the method for measuring $\varepsilon'$ and $\varepsilon''$ of solutions to the glucose concentration was 2.2 dB / 0.5% wt. in water and 0.9 dB / 0.2% wt. in a physiological solution. The extrapolation of these results allows us to suggest that a sensitivity of about 0.1 dB / 0.04% wt., i.e., of about 2 mmoles /l, can be attained with the plates available.

4 Investigation of Blood

These experiments were carried out in a thermostatically controlled chamber when a drop of blood taken immediately from the fingertip of a test person was placed on a matching plate. The measurements were carried out with a waveguide of cross section 5.2 $\times$ 2.6 mm
(operating frequencies 41—42 GHz), which was completely covered by a drop of blood. Typical results of one of experiments for 4 persons in a thermostatically controlled chamber are shown in Table 3.

Table 3 Investigation of blood

<table>
<thead>
<tr>
<th>N</th>
<th>Hb, g/l</th>
<th>$T_1$; $t = 32-34^\circ C$</th>
<th>$T_1 + 40$ min; $t = 36^\circ C$</th>
<th>$T_1 + 120$ min; $t = 36-37^\circ C$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>150</td>
<td>3.9</td>
<td>253</td>
<td>37.4</td>
</tr>
<tr>
<td>2</td>
<td>142</td>
<td>3.6</td>
<td>332</td>
<td>37.9</td>
</tr>
<tr>
<td>3</td>
<td>136</td>
<td>3.7</td>
<td>268</td>
<td>39.1</td>
</tr>
<tr>
<td>4</td>
<td>140</td>
<td>3.7</td>
<td>367</td>
<td>39.0</td>
</tr>
</tbody>
</table>

Here, $W$ is the content of glucose in mmoles / l, $Ch$ is the content of cholesterol in mg, $Hb$ is the content of hemoglobin in g / l, $T_1$ is the moment of time when persons 1—4 took 50 g of glucose on an empty stomach. Thus, the data of Table 3 show the following:

1. The dependence of $R$ on $W$ has an individual character; however, all the persons show a decrease in $R$ after taking glucose.
2. The inverse process of decreasing $W$, which is recorded by the variation of $R$, is not so clearly pronounced. This fact can be connected with the physiological processes that take place in blood as the glucose concentration decreases.
3. There is no obvious effect of hemoglobin (which was constant during the measurements).
4. The content of cholesterol, may be responsible for the difference between the functions $R(W)$ measured as $W$ decreases and increases.

We determined $\varepsilon'$ and $\varepsilon''$ of blood at temperatures close to the temperature of a human body. At $f = 42.93$ GHz, $\varepsilon' = 18.1 \pm 0.2$ and $\varepsilon'' = 23.8 \pm 0.2$; i.e., the difference between $\varepsilon'$ and $\varepsilon''$ for different persons was small. Note that the data on $\varepsilon'$ and $\varepsilon''$ of blood (not in vivo) that are available in the only publication [8] (which were measured at 25°C: $\varepsilon' = 13 \pm 3$ and $\varepsilon'' = 20 \pm 3$) are in agreement with our data if we introduce temperature corrections by analogy with the temperature dependence of $\varepsilon'$ and $\varepsilon''$ of water.

### 5 Investigation of Skin

From the electrodynamical point of view, skin and adjoining blood-filled tissues represent a much more complicated object of study than blood. Many authors (see, for example, [9]) pointed out that the parameters of skin, such as thickness, blood richness, sweat, and moisture, depend on a test person, his age, and a place on his body. Moreover, the blood richness and moisture depend on external factors, such as temperature, humidity, and illumination, and internal factors, such as physical and intellectual stresses and a general state of health.
Therefore, at the first stage, we measured $R_{\text{min}}$ and $f_{\text{min}}$ for different parts of body at different frequencies. As was expected, fingertips, palms, wrists, forearms, and earlobes have substantially different values of the reflection coefficient. When we used the matching plates that guaranteed a deep minimum $R_{\text{min}}$ for water and blood, the maximum reflection $R_{\text{min}}$ (the minimal value of $|R_{\text{min}}|$) was attained with fingertips and palms. The best matching was achieved for earlobes and forearms. Therefore, further measurements of $R_{\text{min}}$ were carried out on forearms. Just as in [10], where the measurements were carried out in the infrared band, for different persons, we observed different values of $R_{\text{min}}$ and $f_{\text{min}}$ at equal values of glucose concentration. We also found that the values of $R_{\text{min}}$ and $f_{\text{min}}$ depend on the pressure of the matching plate to the forearm and its position on the forearm. To minimize these effects, we carried out systematic measurements in the long-wavelength region of the millimeter-wave band, where, as is well known (see, for example, [9] and our skin data presented in Table 4), the penetration depth of the wave $d \approx 1/\alpha$ (the skin depth) is maximal, and, as a result, the wave interacts with parts of body that are richer in blood.

Table 4 shows that $\varepsilon'$ and $\varepsilon''$ monotonically decrease as frequency increases. The penetration depth $d$ of the wave into the skin equals approximately $1/3\alpha$ and ranges from 0.7 mm for 30 GHz to 0.36 mm for 77 GHz at 36—37°C. These values are close to those of water at the same temperature. The results of Table 4 are obtained by averaging the results of more than 20 measurements at $W = 4—5$ mmoles/l. Note that these values of $\varepsilon'$ at frequencies 30—40 GHz are in satisfactory agreement with the results of [9], whereas $\varepsilon''$ in our experiments is substantially greater than that in [9].

As for the measurements of $R_{\text{min}}$ and $f_{\text{min}}$ as a function of $W$, just as in the case of measurements of blood at a frequency of 43 GHz, we observed a correlation between $R_{\text{min}}$ and $W$ as $W$ increased after taking glucose (sugar) on an empty stomach. At frequency of about 60 GHz, a variation in $R_{\text{min}}$ was much smaller that that at 42 GHz, which is likely to be attributed to the smaller penetration depth $d$. Note that, in the afternoon (after 3—4 p.m.), a variation in $R_{\text{min}}$ for close values of $W$ in normal situation (without additional taking glucose) was much greater than that before the noon. This fact indicates to certain physiological changes in skin at the depth $d$ that are associated with vital functions of the organism, such as movements, nourishment, and tiredness, which were also pointed out in [10]. To eliminate the effects of the position and the pressure of the matching plate and

Table 4 Dielectric properties of skin

<table>
<thead>
<tr>
<th>f , GHz, tanδ, α</th>
<th>29.8</th>
<th>tanδ</th>
<th>42.6</th>
<th>tanδ</th>
<th>66</th>
<th>tanδ</th>
<th>77.4</th>
<th>tanδ</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water: 37°C</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\varepsilon$</td>
<td>1.0</td>
<td>1.32</td>
<td>1.68</td>
<td>1.8</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\varepsilon' = 34.05+i34.3$ m = 6.42+i2.67</td>
<td>14.5</td>
<td>21.3</td>
<td>30.9</td>
<td>34.4</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\varepsilon'' = 23.1+i30.5$ m = 5.5+i2.8</td>
<td>21.3</td>
<td>30.9</td>
<td>4.93+i2.67</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\varepsilon$</td>
<td>2.2</td>
<td>2.2</td>
<td>1.62</td>
<td>1.4</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\varepsilon = 10.6+i23.5$ m = 4.3+i2.8</td>
<td>21.4</td>
<td>22.9</td>
<td>3.38+i1.88</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\varepsilon = 13.87+i23.29$ m = 4.93+i2.67</td>
<td>1.68</td>
<td>22.9</td>
<td>3.38+i1.88</td>
<td>1.4</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Skın</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\varepsilon = 13.0+i34.2$ m = 4.98+i3.44</td>
<td>18.7</td>
<td>22.9</td>
<td>7.6+i10.3</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\varepsilon'' = 10.6+i23.5$ m = 4.3+i2.8</td>
<td>21.4</td>
<td>22.9</td>
<td>3.38+i1.88</td>
<td>1.4</td>
<td></td>
<td></td>
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</tr>
<tr>
<td>$\varepsilon'' = 13.87+i23.29$ m = 4.93+i2.67</td>
<td>1.68</td>
<td>22.9</td>
<td>3.38+i1.88</td>
<td>1.4</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
the above-mentioned physiological phenomena of a person, we carried out repeated measurements of $R_{\text{min}}$ in a modified setup. In this setup, the diameter of the contact area between skin and the measurement plate was increased to 35 mm to reduce the effect of the position of the plate on the skin, the measurements were fulfilled at a fixed frequency near 35 GHz, and the test person took only glucose and was at rest during the whole period of measurements. The results of one experiment are shown in Table 5.

Table 5 Results of $W$ measurements with a non-invasive device

<table>
<thead>
<tr>
<th>$W$, mmol/l</th>
<th>4.5</th>
<th>11.0</th>
<th>11.7</th>
<th>10.8</th>
<th>8.5</th>
<th>6.0</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T$, min</td>
<td>0</td>
<td>3</td>
<td>60</td>
<td>90</td>
<td>120</td>
<td>150</td>
</tr>
<tr>
<td>$R$, - dB</td>
<td>7.2</td>
<td>4.8</td>
<td>4.2</td>
<td>4.2</td>
<td>4.8</td>
<td>7.0</td>
</tr>
</tbody>
</table>

Here, $W$ is the glucose content measured by a standard invasive glucometer, $R$ is the reflection coefficient measured by the MM setup. Table 5 shows a clear correlation between the reflection coefficient $R$ and $W$.

6 Conclusions

A new method has been applied to measure the dielectric properties of glucose solutions in water and in a blood imitator. The measurements have been carried out for the first time in the frequency range from 28 to 93 GHz for glucose concentrations $W$ ranging from 5 to 0.5%. A sensitivity of up to 2.2 dB per 0.5% wt. of glucose concentration was realized. Extrapolation of these results shows that the sensitivity may be increased to 0.04% wt. (2 mmoles/l). These results may serve a basis for the design of a laboratory or industrial equipment for controlling small concentrations of glucose (sugar) in water and in the physiological solution.

The dielectric properties of blood are measured in vivo (without preservatives) for the first time with a sufficiently high degree of accuracy (the measurement accuracy of $\varepsilon'$ and $\varepsilon''$ is ±0.2) at frequencies of 42 and 66 GHz. The method developed in the project allows a real-time determination of glucose content in blood using a single drop of blood.

The reflectivity of skin from various parts of human body has been measured at frequency intervals from 30 to 80 GHz. It has been established that, for close values of $W$, fingertips, on the one hand, and forearms and earlobes, on the other, have substantially different values of $R_{\text{min}}$ and $f_{\text{min}}$. As for the noninvasive determination of the glucose concentration $W$, we obtained a good correlation between $W$ and the output MM wave signal of a device that was in contact with skin in the case where $W$ increases after taking glucose on an empty stomach.
References


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Research of Complex Spectrum Interaction with Surface of Biological Tissue

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ABSTRACT. In this report the experimental results of the influence of non-heating complex spectrum electromagnetic wave radiation for increase of immunological state and results of the treatment of diseases are described. Practical results of our experiments: more than five time increasing of oocyties survival, increase of embryos putting into womb and immune resistantance. Investigate the model described from quantum point of view the penetration process of vibrations through surface of oocyties and embryos. Using this method at the fourth day we obtained milk of quality according to standard. The immune status of grown cows not change for its living time in during three-five years.

Keywords: biotechnology, treatment, immune status

1 Introduction

As early as the 18th century the German physiologist Virhoff asserted that the illnesses of all organs begin with illness of the cells. A mammal’s body consists of more than $10^{14}$ ... $10^{15}$ cells. The brain cannot operate efficiently on such a great number of cells. This fact suggested the idea that the cell heals itself. In spite of the fact that the way to treatment is in the cell itself, effective treatment has the sense when the cell is capable renewal of its own functions. Professor of Liverpool University Froehlich proposed that the sick cell radiates acoustic-electrical vibrations in a wide frequency spectrum including millimeter wave range. At first experiments were made on yeast culture. Yeast was radiated with electromagnetic waves (EMW) of 42 GHz frequency. It was found that the speed of the cells’ division increased. When the cell was ill, acoustic-electrical vibrations appeared on the surface of the membrane. Due to the interaction of the millimetre waves external vibration with the membrane surface a process of the albumen structures spatial “extraction” from the cell’s depth takes place and this process returns the cell to its natural state.

Later was noted other possibility to explain the phenomena of non-heating influence of complex spectrum EMW. Suggested vibration take place into membrans of mitachondria and outside wave interfere with it.

During the evaluation, development of the beings, nature used electromagnetic vibrations for the information exchange on the cell’s level. The electromagnetic vibrations of the millimeter range are maximum reduced in passing through the Earth’s troposphere. Experiments concerning the influence of millimeter wave were made on mice and rats. The mice and rats were processed of the non-heating doses of millimeter wave and they survived after they had been exposed to the X-rays. But the only non-heating doses of millimeter wave using is not always effective. This fact can be explained. If only millimeter wave influence on being, there is so named trigger effect and need a long time influence(approximately one hour and more).Complex spectrum include the low frequency (LF), millimeter wave(MW) and near infrared frequencies (IF) has not this.
Mentioned influence was found very effective. The cell’s energy balance, except energy transformation from one state to another, contains also small part of the loss energy and energy spent for the radiation. If the cell is ill, the part of the radiation energy increases. In the literature there is few information about complex spectrum EMW radiation influence, in particular complex use of the IF, MW and LF vibrations. To our mind the complex use of IF, MW and LF vibrations-named as T-therapy- the vibrations from the point of view of the biological influence is very effective method in the treatment of many illnesses and growing of the high-productive strains of cattle.

The penetration of electromagnetic wave was counted as quantum task. For this used Shroedinger equation and moving of quantum particle was presented as wave crest. The depth of penetration through potential barrier on surface of tissue depend on energy of quantum particle and height of potential barrier. Biophysical model of process inside of mithachenory can be explain from the model, which has presented the behaviour at the time of one period millimeter wave vibration. Every level inside of quantum hole is described an ellipse[1].Interaction between levels appeal the fall in standardized current density through this structure.

2 Transplantation of the Embryos which were Subjected to the Electromagnetic Influence

The oocyties have given from ovarys of a cows in slaughterhouse. Before this all animals were investigated for detection of chronic and noncontagious illnesses. A condition of ovarys diagnosed by palpation method.

The modes of oocyties cultivation in vitro are developed and tested in laboratory conditions. After complex spectrum influence impractical oocyties amount in 24 hours of cultivation were only 5,1% that 5 times less in comparison with control group(without influence).

All embryos were divided into two groups. First one — embryos of an excellent and good quality, second one — satisfactory quality. Each group was divided into 2 sub-groups. One subgroup was processed by the EMW for 2 minutes (1st Seri), than 5 minutes (2nd Seri) and 10 minutes. Second subgroup wasn’t processed by EMW. Criterion of the effectiveness of the EMW influence were embryos estimations which included morphological indices and getting accustomed to the receptor’s organism. The estimations were compared with the estimations of the analogical parallel groups without EMW influence (control).

We found out the tendency of rising embryos viability and getting accustomed after using EMW. The effectiveness depends on embryo’s quality. This fact insures us to continue research work in this direction. Indices efficiency of embryo transplantation after 10 minutes complex spectrum influence is the best. For excellent embryos quality the percental index of embryo reception into cow womb equal 72%(after three pregnancy months) that more in comparison with control group(less 60%).

Having analysed the growth and development of the experimental and the control groups calves , it was found out, that the calves got after the transplantation of the embryos which had been proceeded by the electromagnetic waves averaged 32.4+-1.3 kg at the moment of the birth. The difference between the experimental and the control groups calves’ bulk averaged 7.8 kg. In a month the experimental group calves’ bulk has increased to the point of
55.2 kg. It is by 11.7 kg more then the control group calves’ bulk. At the age of 2 months the experimental and the control groups calves’ bulk difference decreased to the point 6.9 kg. Obviously, it is connected with the fact that the non-contagious illnesses (dyspepsia) have been registered among the control group calves. At the age of 3 months the difference between the experimental and the control groups calves decreased to the point 5.4 kg.

By studying the transplanted calves’ physiological state the albuminous exchange was analysed. It was found out, that the full albuminous’ concentration of the experimental group calves averaged 74.83 g/l at the age of the 4 weeks and significantly increased to the point 75.4g/l at the age of the 2 months while the albuminus’ concentration of the control groups calves decreased to the point 64g/l to the end of the second months. This fact shows that there are breach processes of the bowels function in the control groups calves’ organism. These breach processes are accompanied by the hyperhydremious. The gamma-globulin’s concentration of the control group calves’ blood significantly exceeded the same concentration of the experimental group. This fact shows that the experimental group calves have the higher level of the natural resistibility. By the calves’ age increasing the appointed differences between the control and experimental groups calves are preserved.

The ways of the transplanted calves’ organism immunological tolerance were investigated. During the first 1-3 hours after the birth the calves according to their immunoglobulins’ content were distributed into 3 groups. The first group consists of the calves with high, the second - of calves with middle and the third - of calves with low immunoglobulins’ content. The followed observations have showed that among the calves of the first group the digestive disorder has been registered by 4.2 and 6.8 times rarely than of the second and the third groups accordingly. This fact has become a good reason for the working out and modification of the decreasing methods of the calves’ organism immunological tolerance during the first hours of the postnatal period.

The first series of the experiments were hold on the embryos. Before the transplantation to the receptors the 1 group embryos of the excellent and good qualities with the help of the special devices was subjected to the influence of EMW for 20 minutes. The intact embryos were transplanted to the receptors of the second group. Pairing age heifers of the black-motley strains with good developed yellow bodies in the ovaries were used as the receptors. During the first 2 hours after the birth 60% transplanted calves got from the first group receptors has immunoglobulins’ G content exceeding 10g/l and 40% has immunoglobulins’ content between 8.8...10.0g/l. The immunoglobulins’ G content of the calves’ blood got from the second group receptors averaged 6.8±1.7 g/l, the majority of the calves has immunoglobulins’ G content not exceeding 7.0 g/l. Sickness rate of the first and second groups calves averaged accordingly 10.0 and 40.9%. This high level of immunoglobulins’G conserved at time of cows life during three-four years.

These facts show the positive influence of the non-heating radiation on the immunological system of cow organisms.
3 Neurological Disease Treatment by Complex Spectrum

The problem of alternative treatment in medicine needs a constant research new kind of methods.

57 patients at the age of 48-72 years with chronic cerebral circulatory impairment/disturbances within the basin of the carotid and vertebral arteries were treated. The cerebrovascular atherosclerosis was the major factor causing chronic cerebral circulatory impairment in 37 patients. 21 patients had a long history of hypertension. The patients were treated with the complex therapy including vasoactive, hypotensive and antisclerotic drugs and non-heating complex spectrum. An average course of treatment was 8-10 days. The wave was exposed for 20-40 minutes depending on the individual adaptation response. The patients of the control group (23 men) were treated with the above medications and massage in the "collar" neck area. The shoulder joint contralateral to the site of the lesion was the area of exposure. In the patients with hypertension the non-heating complex spectrum wave doses was applied to the cervical sympathetic nodes as the most sensitive to the factors regulating vascular tonus. The neurological state of the patients was evaluated using the criteria developed for the treatment efficiency estimation.

Apart from neurological examination we performed Doppler ultrasonography of the major head and neck vessels. We also studied some parameters of the hemeostasis system and rheological properties of blood. In addition, the type of individual adaptation response was determined.

The neurological state was evaluated by calculating the numbers of the main parameters using the Scandinavia scale. The patients (89,3%) treated with the non-heating complex spectrum were found to have better results than those of the control group (70%).

Dopplersonography was conducted using "Vasoscan" ("Sonicaid" GB) that provided the spectral analysis of Doppler signals by fast Furiye transformation. The blood flow was studied in the common, internal and external carotid arteries, supraciliary and vertebral arteries. In all cases supraciliary hemodynamical test and external carotid artery branches compression tests were carried out.

The following parameters were processed by computer: peak systolic frequency (max A), peak diastolic frequency (max D), cerebral vascular resistance parameters (RP), pulsation index (PI), spectral broadening (SB). All patients showed signs of the cerebral blood flow insufficiency in the major cerebral arteries due to their stenosis or occlusion manifested mainly in the carotid artery. With this cerebro-vascular lesion the cerebral blood flow redistribution through the Circus of Willisii vessels as well as extra-intracranial anastomosis becomes impossible and an acute cerebral ischemia may develop.

The cerebral blood flow improved in 23 patients following the complex treatment including the non-heating complex spectrum therapy. The systolic blood pressure fell and the diastolic one raised in the patients with the arterial stenosis of less than 50%. In cases of the arterial stenosis of more than 50% the cerebral vascular resistance parameters (RP) went down.

In most cases the spectral broadening decreased by the end of the course of treatment indicating that the cerebral blood flow acquired the laminar characteristics. In addition, some hemeostasis parameters improved; in particular, the thrombocytes aggregation time decreased.
from 35.8±1.2 to 34.1±0.56, while disaggregation percentage increased from 8.7±2.8 to 13.2±3.2. A decrease in hematocrit level (from 46 to 40%) was also registered. The arterial blood pressure parameters were compared with the individual systolic and diastolic pressure parameters characteristic of the given age group and determined using the formulae: systolic blood pressure is equal to 102+ (0.6 x age in years) mm Hg; diastolic blood pressure is equal to 63+(0.4 x age in years) mm Hg. The evaluation of the hypotensive effect in the patients with accompanying hypertension revealed that there was a drop in the initially elevated blood pressure in 14 patients (84.4%). Since the non-heating complex spectrum therapy significantly effects compensatory adaptation mechanisms we could trace the adaptation response dynamics in the patients with chronic cerebral circulatory impairment. The type of individual response was determined using the lymphocytic segmental index (LSI), i.e. 0.3 and less was considered "stress", 0.31-0.50 - "training", 0.51 -0.70 - "low activity", 0.71 and higher -"high activity". The duration and frequency of electromagnetic fluctuations were chosen depending on the initial level of LSI. In the patients with "stress" the wave exposure was 30-40 minutes, while the millimeter wave frequency as part of complex spectrum which has an exposure less 53.67 GHz. In the "training" and "low activity" patients the less wave exposure and higher frequency were more preferable.

In the patients with accompanying hypertension the parameters of cerebral vascular resistance and hypertonus regressed. The platelet aggregation time decreased while the percentage of disaggregation in hemostasis system increased. The decrease in the hematocrit level was registered. The complex spectrum wave therapy had a considerable antihypertension effect when the sinocarotid area was irradiated.

The above treatment of patients with cerebral circulatory impairment showed the favourable effect on patients' adaptability: i.e. "stress" response significantly decreased and was followed by "high" or "normal" activity.

Today this researching are extended for treatment neuropathy different genesis. The first results brought positive effect of complex spectrum influence.

4 Experimental Animals Diseases Treatment

The scientific and practical research in the veterinary began with the treatment of mastitis. The problem concerning mentioned illness is still actual and difficult. This illness is considered to be one of the most spread at feeding time. The most spread form of the animal mastitis is mastitis without showed symptoms or closed form. According to the International milking federation, only 3 per cent of ill cows has showed symptoms and 52.0 per cent of ill cows doesn’t have any symptoms of mastitis.

For mastitis treatment we worked out special device. The milk gland processing was made in the morning and in the evening for 3 days. The sample of milk was taken before and after processing. Received results were processed with the help of the statistic and correlation analysis. The cows are continued to be observed and clinic examination and milk analysis are taken place.

During the microbiological examination of the cow’s milk gland was found out that 17-38 per cent of all the micro-organisms are of spherical form, 3-42 per cent — staphylococcus, 58...80 per cent — milk-lactic micro-organisms. During the examination of the cow’s without showed symptoms was found also putrefactive microflora.
After the first processing the composition of the Gram-negative forms of the micro-organisms decreased. The concentration of the micro-organisms was decreasing constantly and on the 3rd day the single Gram-positive forms of the micro-organisms appeared.

That means, that after processing of the cow’s milk gland with our devices positively influenced on the microbiological composition of the milk gland and promotes its recovery. *On the 4th day it is possible to receive milk of good quality. Same dynamic of microbiological data had got after EMW influence at treatment time of endometritis and stomach’s diseases.*

**5 Conclusion**

The method of the influence of the complex spectrum of electromagnetic wave weak doses for treatment were worked out. Prevental phenomena in case diseases. 57 patients with chronic cerebral circulatory impairment of in the carotid and vertebral arteries were treated with non-heating millimeter wave was researched. It was found that the patients treated with EMW had much better results (89.3%) than those of the control group (70%).

During the first 2 hours after the birth 60% transplanted calves got from the first group receptors has immunoglobulins’ G content exceeding 10g/l and 40% has immunoglobulins’ content between 8.8...10.0g/l. The immunoglobulins’ G content of the calves’ blood got from the second group receptors averaged 6.8±1.7 g/l, the majority of the calves has immunoglobulins’ G content not exceeding 7.0 g/l. Sickness rate of the first and second groups calves averaged accordingly 10.0 and 40.9%. This high level of immunoglobulins’ G conserved at time of cows life during three-four years.

Our experiments confirmed our hypothesis about parametric influence of the EMW on biosystems what means that regeneration processes take place in the cells.

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**References**


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Dielectric Properties of Human Semen at Microwave Frequencies

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ABSTRACT. The electromagnetic spectrum is being used in every field of diagnostic and therapeutics of modern medicine. This work details the effects on dielectric properties of human semen in vitro at S-band of microwave frequencies using cavity perturbation technique. Semen samples are collected from normal subjects and measurements are done. It is observed that conductivity of semen increases as time elapses, which could find applications in forensic medicine.

Keywords: dielectric properties, cavity perturbation, semen, forensic medicine

1 Introduction

Information regarding the dielectric properties of tissues is important for studies on the biological effects of radio frequencies and microwaves. The understanding and development of medical microwave techniques can be aided by knowledge of the microwave dielectric properties of human tissues. For example, tissue temperature pattern retrieval in microwave thermography is achieved using models of the underlying tissue structure, which depend particularly on the dielectric properties of the tissue [1, 2].

The dielectric parameters of various human tissues at different RF frequencies were reported by Gabriel, Cook and Land et.al [3-6]. Cook et al also conducted a microwave study of human blood using coaxial line and wave-guide methods [7]. Tissue samples of human brain at microwave frequencies were analysed using sample cell terminated transmission line methods [8]. Open-ended coaxial line method allows measurements of tissue samples over a wide range of frequencies [9].

Microwave medical tomography is emerging as a non-hazardous technique for imaging. Proper knowledge of body tissue dielectric properties at the lower microwave frequencies is important for both active and passive microwave imaging [10-12]. Studies on the variation of dielectric properties of body fluids and urinary calcifications at microwave frequencies have revealed that diagnosis is possible through cavity perturbation technique [13-14]. Here we report dielectric properties of semen at microwave frequencies. The conductivity of semen samples is found to increase with time.

2 Measurement Procedure

The measurement set-up consists of a transmission type S-band rectangular cavity resonator, HP 8714 ET network analyser. The cavity resonator is a transmission line with one or both ends closed. The length of the resonator determines the number of resonant frequencies. The transmission type resonator used in this experiment is excited in the TE_{10p} mode. The sample holder which is in the form of a capillary tube flared to a disk shaped bulb at the end is introduced into the cavity through the non-radiating cavity slot, which can facilitate the easy movement of the sample holder. The resonant frequency f_o and the corresponding quality factor Q_o of the cavity at each resonant peak with the empty sample holder placed at the maximum electric field are noted. The same sample holder containing a
known amount of sample is again introduced into the cavity resonator through the non-radiating slot. The resonant frequencies of the sample loaded cavity is selected and the position of the sample is adjusted for maximum perturbation (i.e. maximum shift of resonant frequency with minimum amplitude for the peak). The new resonant frequency $f_s$ and the quality factor $Q_s$ are noted. The same procedure is repeated for other resonant frequencies. Dielectric parameters were calculated using cavity perturbation technique.

3 Theory of Cavity Perturbation

When a material is introduced into a resonant cavity, the cavity field distribution and resonant frequency are changed which depend on geometry, electromagnetic properties and its position in the fields of the cavity. Dielectric material interacts only with electric field in the cavity. According to the theory of cavity perturbation, the complex frequency shift is related as \[ \Delta \omega = -\frac{(\varepsilon_r - 1) \int E_0 E^* dV}{2 \int |E_0|^2 dV} \] \[ \Delta \Omega = \frac{d \omega}{\omega} \approx \frac{1}{2} \left[ \frac{1}{Q_s} - \frac{1}{Q_0} \right] \] but \[ \frac{d \Omega}{\Omega} \approx \frac{d \omega}{\omega} + \left[ \frac{1}{Q_s} - \frac{1}{Q_0} \right] \] Equating (1) and (2) and separating real and imaginary parts we get

\[ \varepsilon_r' - 1 = \frac{f_o - f_s}{2 f_s} \left( \frac{V_c}{V_s} \right) \] \[ \varepsilon_r'' = \frac{V_c}{4V_s} \left( \frac{Q_o - Q_s}{Q_o Q_s} \right) \] Here, $\varepsilon_r = \varepsilon_r' - j \varepsilon_r''$, $\varepsilon_r'$ is the relative complex permittivity of the sample, $\varepsilon_r'$ is the real part of the relative complex permittivity, which is known as dielectric constant. $\varepsilon_r''$ is the imaginary part of the relative complex permittivity associated with the dielectric loss of the material. $V_s$ and $V_c$ are the volumes of the sample and the cavity resonator respectively. The conductivity can be related to the imaginary part of the complex dielectric constant as

\[ \sigma = \omega \varepsilon'' = 2 \pi f \varepsilon_0 \varepsilon'' \]

4 Biology of the Sample

Semen, which is ejaculated during the male sexual act, is composed of the fluid and sperm from the van der Feerans (about 10% of the total), fluid from the seminal vesicles (about 60%), fluid from the prostrate gland (about 30%), and small amounts from the mucous glands, especially the bulbourethral glands. Thus, the bulk of the semen is seminal vesicle fluid,
which is the last to be ejaculated and serves to wash the sperm out of the ejaculatory duct and urethra. The average pH of the combined semen is approximately 7.5, the alkaline prostatic fluid having neutralised the mild acidity of the other portions of the semen. The prostatic fluid gives the semen a milky appearance, while fluid from the seminal vesicles and from the mucous glands gives the semen a mucoid consistency. Also, the clotting enzyme of the prostatic fluid causes the fibrinogen of the seminal vesicle fluid to form a weak coagulum that holds the semen in the deeper regions of the vagina where the uterine cervix lies. The coagulum then dissolves during the next 5 to 15 minutes because of lysis by fibrinolysin formed from the prostatic profibrinolysin. In the early minutes after ejaculation, the sperm remain relatively immobile, because of the viscosity of the coagulum. However, as the coagulum dissolves, the sperm simultaneously become highly motile. Although sperm can live for many weeks in the male genital ducts, once they are ejaculated in the semen their maximal life span is only 24 to 48 hours at body temperature. At lowered temperatures, however, semen may be stored for several weeks; and when frozen at temperatures below –100°C, sperm have been preserved for years [16].

5 Results and Discussion

The microwave studies of the samples are done using cavity perturbation technique and the results are shown in Table 1 and in Figs. (1-2). Table 1 indicates the variation of dielectric constant of different semen samples at different time intervals after ejaculation. It is observed that the dielectric constant is consistent at all frequencies at different intervals of time after ejaculation. From Fig. 1 it is observed that the conductivity of the semen samples increases as frequency increases. This shows that semen is lossier at higher frequencies. This is due to absorption of electromagnetic energy by the sample under study. From Fig. 2 it is observed that the conductivity of semen increase as time elapses. This is due to the clotting enzyme of the prostatic fluid, which forms a coagulum in early stages after ejaculation, which makes the sperm remain relatively immobile, because of the viscosity of the coagulum. The conductivity is relatively less due this effect in early stages. As coagulum dissolves during the next 5 to 15 minutes, sperms become highly motile, which causes an increase in the conductivity. This has potential application in forensic medicine in that the elapsed time after ejaculation is directly related to the conductivity of semen.
Table 1 Variation of dielectric constant with frequency at different time intervals

<table>
<thead>
<tr>
<th>Frequency (MHz)</th>
<th>T = 5minutes</th>
<th>T = 15minutes</th>
<th>T = 30minutes</th>
<th>T = 45minutes</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>M23</td>
<td>M24</td>
<td>M29</td>
<td>M33</td>
</tr>
</tbody>
</table>

Fig. 1 Conductivity of semen
Fig. 2 Temporal behaviour of conductivity of M 24

6 Conclusion

The microwave study of the semen samples is done using cavity perturbation technique. This technique requires very low volume of sample and it is particularly applicable to samples like semen. The study shows that the dielectric constant of a given semen sample does not show appreciable variation with time or with frequency. Also it is observed that the conductivity of the semen sample increases as frequency increases, which indicate that semen is lossier at higher frequencies. The conductivity of semen also shows an increase with time, and this potentially finds application in forensic medicine to find the time of ejaculation after the sexual act.

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Reference


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Session 3

Applications of Model Systems, Mixing rules, Calibration, and Reconstruction Algorithm
Chairmen: I. Woodhead; D. Funk

1  The Relation between Fractal Dimension and Microwave Parameters
    A. Albadri; London, UK

2  A Unified Moisture Algorithm for Improved RF Dielectric Grain Moisture Measurements
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3  Development of TDR-Sensors for Moist Materials Using HFSS
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6  Enhanced Integral Equation Modelling for Moisture Sensors
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7  Spatial Time Domain Reflectometry with Rod Probes
    R. Becker, St. Schlaeger; Karlsruhe, Germany
The Relation Between Fractal Dimension and Microwave Parameters

Ali Albadri
Hydronix Ltd; Guildford; UK

ABSTRACT. Microwave techniques have been used to measure moisture content of particulate materials such as grains, seeds and pulverised minerals. The shapes of the particles have a significant impact on the bulk density of the material. The density of the material is affected by changes in the moisture content, and temperature of the material. In this study the fractal dimension has been used to quantify the geometrical arrangement for the particles and the spaces between them, it was determined from the topography of the particles. Measurable microwave parameters such as frequency, bandwidth, and amplitude were correlated to the fractal dimension after specific additions of quantities of water were added to the particulate material. Two models were suggested to determine the fractal dimension, one model was done to simulate spherical shape (with circular cross section) particles like lentil seeds, and another was to simulate oval (with elliptical cross section) shape particles like wheat grains.

Keywords: fractal dimension, moisture content, dielectric properties

1 Principles of the Fractal Dimension

There are objects which can not be described by known geometrical feature, because they are irregular. Mandelbort [1] proposed that the irregularity of such objects could be described as fractal. A fractal line has the property of being self similar to any scale (in some cases only within a defined scale). That is, at any magnification, the geometrical appearance of the line is similar but not necessary regular.

Consider an irregular line describing, for example a profile (see Fig. 1), and then

![Fig. 1 The interaction of the measuring unit with irregular profile](image)

measure the length of the line using a finite measurement step. The value so obtained is an approximate length, and would depend on the step size. A large step erodes the angular fluctuations of the line [2].

The apparent length of a fractal line between two points, $L_p$, is related to the size of the measuring step $\zeta$, and the fractal dimension $D$ is given by [2]:

$$L_p(\zeta) = N(\zeta)^{1-D}$$  \hspace{1cm} (1)

The fractal dimension may then be obtained from the slope of $\ln L_p(\zeta)$ plotted against $\ln(\zeta)$, noting that the fractal dimension $=D=1+$slope is greater than 1.

2 Particles Shapes and Microwave Parameters

Measurements for dielectrics of particulate materials and especially grains are dependent on various factors. The dominant factors are the amount of water in the grains, range of frequency used, and the temperature of the grains especially at extreme high or low temperatures. Variability in density has a substantial effect on the reliability and repeatability of the measurements that is on the errors in measuring the frequency or frequency shift or amplitude or phase shift or bandwidth.

Many studies have tried to eliminate the effect and the variability in the density of the particulate materials during static or on line measurements. But few of them have tried to determine the relationship between the shape of the particles and density, or even to assess the correlations between the shapes of the particles and the measured microwave parameters.

Single kernel (oval shape) and single soybean (disc shape) was studied using cavity microwave resonator [3]. The measurements involved measurement of the resonant frequency and the $Q$, quality factor, of the resonator. Samples of the soybean revealed that the resonant frequency shift $\Delta F$, and the change in the transmission factor $\Delta T$ were both linear function of the mass of water in the seed. The two variables are related to the dielectric property of the seed as follows;

$$\Delta F = (\varepsilon - 1)Kf_o(\nu_s/\nu_c)$$  \hspace{1cm} (2)

$$\Delta T = \varepsilon'K^2Q(\nu_s/\nu_c)$$  \hspace{1cm} (3)

Where $K$ is the shape factor, $f_o$ and $Q_o$ respectively are the resonant frequency and quality factor for the empty cavity, $\nu_s$ and $\nu_c$ are the volumes of the sample and cavity respectively.

It has been proven by Kraszwski [4] that the ratio of the parameters $\Delta F$ and $\Delta T$ is independent of size (volume) of the seed, and slight variation in the seed shape should not significantly affect the dependence of this ratio upon moisture content. As it was observed in the following equation;

$$\Delta F/\Delta T = \left(\frac{\varepsilon' - 1}{\varepsilon''}\right)(1/K)(f_o/2Q_o)$$  \hspace{1cm} (4)

Mohsenin [5] introduced shape classifications such as roundness and spherical ..etc for seeds. However, Nelson [6] calculated the three orthogonal dimensions (length, width, and thickness) for various seeds. Volume coefficient was determined as the ratio of the measured mean volume to the orthogonal dimension of the grain. The volume coefficient ranged from 0.222 for oil type sunflower seeds to 0.588 for sweet corn seeds. With use of the volume coefficient, seed density was estimated for a particular kind of seed from seed weight and dimensions.
However no attempts were tried to correlate the geometrical variables to the microwave parameters.

3 A Model to Determine the Fractal dimension of Circle and Elliptical Shape Particles

A box of 200mmx200mmx200mm and another 100mmx200mmx100mm respectively were assumed to uniformly contain 20mm diameter spherical shape particles in the former box and 10mmx20mmx10mm oval shape particles in the latter box (as in Fig. 2a and Fig. 2b).

Fig. 2a 200mmx200mmx200mm box contains 20mm spherical shape particles.

Fig. 2b 100mmx200mmx100mm box contains 10mmx20mmx10mm oval shape particles.

A perpendicular view was taken to one side of the box as in Fig. 3a and Fig. 3b. A circle with a diameter of 40mm was drawn at an arbitrary location on the view. Previous experimental trails proved to us that the diameter of the circle should be at least 2d, where d is the diameter.
of the spheres or $2L$, where $L$ is the longest dimension in the ellipse for the oval shape particles.

![Diagram](image)

Fig. 3a A perpendicular view to one side of the box (in Fig. 2a) which contains spherical shape particles.

![Diagram](image)

Fig. 3b A perpendicular view to one side of the box (in Fig. 2b) which contains oval shape particles.

Particles intersected by the circle have one part inside the circle and another outside the circle (see Figs. 3). The outside boundary of each particle represents a part of a boundary for a region. Continuous repeating this step with each intersected or touched particle until the circle is closed produces a contour that defines the boundary of that region.

A divider was opened to various step sizes ($\zeta$), then stepped along the profiles of the contours of the regions. The apparent length $L_p(\zeta)$ was measured as the product of the number of steps and the step size.
The spherical (with circle cross section) and oval (with elliptical cross section) geometries in Figs. 2 and Figs. 3 were assumed to simulate respectively spherical and oval grains or mineral particles. The spaces between the particles simulate the liquid medium (in this study is water) that contains the particles.

The volume fraction of the liquid medium increases as the volume of the box increase, however the volume fraction of the particles remains constant. This means that only the distances between the particles will increase. Numbers of dimensional increases in the volume of each box were drawn and laid down on 2D drawings as in Figs. 3. These increases simulate increases in the moisture content which is accommodated between the particles.

For each increase in the liquid percentage there will be a change in the contours that define the regions, as changes in the spaces between the particles causes changes in the particles compaction. Then the fractal dimensions were estimated from the 2D drawings. Each value for the fractal dimension was calculated from at least 20 measurements to reduce the error to less than 5%.

A percentage of the liquid medium might be absorbed by the particles depending on the chemical and physical compatibility between the two. However in this model this percentage has been ignored.

Fig. 4 and Fig. 5 show the liner relationships between $ln L_p(\zeta)$ and $ln(\zeta)$ of profiles (contours) of regions of the particles when they are mixed with different percentages of the liquid medium (water). The figures suggest that the fractal dimension decreases then increases as the volume fraction of the water increase.

![Fig. 4 The relationship between $ln L_p(\zeta)$ and $ln(\zeta)$ with D=fractal dimension and M=moisture content for spherical shape particles.](image-url)
Fig. 5 The relationship between $\ln(L_p(\zeta))$ and $\ln(\zeta)$ with D=fractal dimension and M=moisture content for oval shape particles.

4 Experimental Measurements

Open cavity resonator was connected to a metal box that contains electronics components as in Fig. 6. The electronics components work to process the signal coming from the resonator to deduce the resonant frequency, bandwidth and amplitude. The cavity window was insulated from the tested material by thin sheet (1mm=thick) of Nylon barrier, which was placed on the window for the cavity.

Fig. 6 Sketch shows the arrangement for the devices used to measure the frequency, and width, amplitude.

0.6 kg of lentil seeds and 1.3 kg of wheat grains were placed in separate plastic containers and left to dry out for 48 hr at 28°C. In individual steps, quantities of water were added to the materials to increase their moisture content. A digital weighting scale was used to measure the weight of material before and after each addition of water, thus the mass fraction of water (the percentage of water) was calculated from;
\[ m_w = \frac{m_t - m_m}{m_t} \]  

\( m_w \) = mass of water (g).  
\( m_t \) = total mass of the particulates material (g).  
\( m_m \) = mass of the dry particulates material (g).  

The particles and additions of water were manually mixed and left to stabilise for 1hr at 18°C. The frequency, bandwidth and amplitude measurements were done on the dry materials and on each wet material after each addition of water.  

At least 20 measurements for each of the frequency, bandwidth and amplitude were needed to reduce the error to less than 5%. The average of the 20 measurements was calculated, and then used in plotting the next Figures.  

When the percentage of water reached 40% it was not possible to notice any change in the measurements as the lentil seeds and wheat grains became saturated with water. The saturation state could be seen as significant accumulation of water on the bottom of the container.  

Photographs were taken to the top view of the material in the containers after each addition of water (see Fig. 7 and Fig. 8). Circles were drawn on each photograph to define boundaries of number of regions using the way described in the previous section. The fractal dimensions of the boundaries (profiles or contours) of the regions were determined then the values were averaged out to a single value for each moisture percentage. The error in the averaged values of the fractal dimensions was reduced to less than 5% by measuring high number of regions. Fig. 9 and Fig. 10 show the relationships between \( \ln L_p(\zeta) \) and \( \ln f(\zeta) \) for lentils seeds and wheat grains. Fig. 9 shows that the fractal dimension increases than decrease as the volume fraction of water increase. Fig. 10 shows that fractal dimension decreases than increases with increasing the water content.  

Fig. 7 Top view for the lentil seed .
Fig. 8 Top view for the wheat grains.

Fig. 9 The relationship between $\ln L_p(\zeta)$ and $\ln(\zeta)$ for lentil seeds.
5 Results and Discussion

The results from the model of the spherical particles (circular cross section) shows that the fractal dimension decreases as the moisture contain increase as shown in Fig. 11. Detailed study to the relationship indicates that there is a tendency for the values of the fractal dimension to change from low to high values then low again as the percentage of the liquid medium increases. The model for the oval shape particles (with elliptical cross section) shows clearly that the fractal dimension fluctuates, decreases then increases as the percentage of the liquid medium increase.

A. W. Kraszewski and S. O. Nelson [7] used equations 2 and 3 for moisture content determination on objects of variable size and shape. During the measurements, it was observed that when a corn kernel of arbitrary shape is rotated about the x-axis in the cavity,
both $\Delta F$ and $\Delta T$ follow $\cos^2\theta$ behaviour, where $\theta$ is the angle of rotation. For two kernels of different size and shape but similar moisture content, the measured values pass alternately through equal spaced region (90° apart) of weak and strong interaction with the electromagnetic field. It was reported that the amplitude of change for the spherical large kernel is smaller than those for a flak, disk-like, but smaller kernels.

Fig. 12 and Fig. 13 show respectively that the frequency decreases as the moisture content in the lentil seeds and wheat grains increase, while the bandwidth has a weak tendency to increase and the amplitude to decrease with increasing the moisture content. The detailed study of the three curves suggests that they have the tendency to fluctuate as the water content in the seed or grain increase.

![Graph showing the relationship between microwave parameters and moisture content for lentil seeds.](image1)

![Graph showing the relationship between microwave parameters and moisture content for wheat grains.](image2)

Fig. 12 The relationship between the microwave parameters and moisture content for the lentil seeds.

Fig. 13 The relationship between the microwave parameters and moisture content for the wheat grains.
Figure 14 and Figure 15 respectively show the change in the fractal dimension of lentil seeds and wheat grains against their moisture content. While the fractal dimension for the lentil seeds increases then decreases with increasing the moisture content, and then it tends to increase again at 40% water content, the fractal dimension for the wheat grains decreases then increases with increasing the moisture content, and it tends to decrease again at 40% water content.

Fig. 14 The change in the fractal dimension with the moisture content for the lentil seeds.

Fig. 15 The change in the fractal dimension with the moisture content for the wheat grains

The two figures suggest the way in which disc shape particles, like the lentil seeds, and oval shape particles, like the wheat grains, align and orientate themselves or the spaces between them change as their water content increase.

There are two explanations to the results in Fig. 14 and Fig. 15. The first one is that when the lentil seeds had low percentage of water, they tended disorientate themselves to be not well packed, hence the high fractal dimension. As the water percentage increases to 35% the seeds aligned and orientated themselves to a common orientation by packing themselves, hence the low fractal dimension. The fractal dimension for the grains of wheat show that it is low at low
water content and it increases when the water content increases to more than 28%. This could indicate that the grains disorientated themselves to be not well packed when the moisture content is more than 28%. The disorientation of the grains peaked at 34% water content then decreased at 40%. The second explanation is that the spaces between the grains or seeds change (increased) with increasing the moisture content. This explains the agreement in the fluctuated behaviour for the relationships in Fig. 14 and Fig. 15, and that in Fig. 11.

Fig. 16 shows that the frequency, amplitude and bandwidth fluctuate as the fractal dimension increase. Both the frequency and amplitude behave in a similar manner (increase then decrease), and both behave in opposite to the bandwidth. Fig. 17 for the wheat grains show different behaviour to that seen for the lentil seeds. It suggests that the frequency and bandwidth behave in a similar manner (decrease then increase) as the fractal dimension increase, but the amplitude behaves opposite to both.
6 Conclusions

This study is an attempt to use the concept of the fractal dimension as a tool to establish relationships between particles shapes, particles alignments (configuration), the spaces between the particles and the moisture content in the particulate material.

Theoretical model was proposed for spherical and oval shape particles to determine the fractal dimension, and to correlate the fractal dimension to the moisture content in the particulate material. The values for fractal dimension fluctuate as the moisture content increase. This fluctuation is caused by increase in the spaces (or the percentage of the liquid medium) between the particles.

Experimental measurements showed that microwave parameters like frequency, bandwidth, and amplitude fluctuate as the moisture content or the fractal dimension of the particles increase. It was observed that the fluctuation is dependent on the particles shapes, particles alignment and the spaces between them.

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A Unified Moisture Algorithm for Improved RF Dielectric Grain Moisture Measurements

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ABSTRACT. This paper presents a Unified Grain Moisture Algorithm, based on measurements of the real part of the complex permittivity of grain at 149 MHz. The main goal of the method was to enable different moisture meter models to provide equivalent moisture predictions without calibration development. The database that was used to create and test the method included 6,189 grain samples representing 53 grain types of U.S. grain that were collected over a period of six years. The overall standard deviation of differences with respect to the air oven method was 0.34 percent moisture. The algorithm is available as a public algorithm for commercialization by multiple manufacturers.

Keywords: radio-frequency, moisture, grain, unified

1 Introduction

Moisture content is one of the most important quality factors in marketing grain, since it determines both the quantity of dry matter traded and the storability of the grain. Grain moisture meters based on the RF dielectric method have been used widely for over fifty years because the technique is capable of reasonably good accuracy for all grain types (with appropriate calibrations), the time required for a measurement is compatible with grain producers' and handlers' needs, and the cost of the instrumentation is moderate. However, the RF dielectric method has been hampered by calibration cost and complexity associated with differences between grain types and from year to year for specific grain types. Furthermore, the lack of standardization of measurement frequency and sensing technology has severely limited calibration transferability among moisture meter models.

In response to these challenges and with the belief that it was possible to substantially improve the RF dielectric moisture method, two agencies of the US Department of Agriculture (USDA-Grain Inspection, Packers and Stockyards Administration (GIPSA) and USDA-Agricultural Research Service (ARS)) initiated a collaborative research project in 1995. Kurt C. Lawrence and Stuart O. Nelson (ARS) developed the calibrated dielectric test cell [1] that was subsequently used by GIPSA to measure the dielectric characteristics of thousands of grain samples.

GIPSA’s analysis of that data and other tests at lower frequencies [2] showed:

- The large dielectric loss peaks and steep slopes and the unusually high dielectric constant values observed in the kilohertz and low megahertz frequency regions are due to conductivity effects—Maxwell-Wagner relaxations and electrode polarization—and not to bound water. These conductivity effects are extremely sensitive to the distribution of moisture within kernels and subtle differences in kernel morphology—contributing significantly to moisture meter calibration instability within grain types and calibration diversity across grain types.

- Moving the measurement frequency for dielectric moisture meters from the 1 to 20 MHz range to about 150 MHz (specifically, 149 MHz), where dielectric loss appeared
to be minimized, dramatically reduces the influence of conductivity effects on moisture measurements and improves calibration accuracy and stability.

- For grain, the Landau-Lifshitz, Looyenga density correction as restated by Nelson [3] effectively normalizes grain samples to a common density—thereby minimizing density-induced errors from test cell filling methods, grain moisture level, and kernel density and shape.

- After density correction, the shapes of the dielectric constant (at 149 MHz) versus moisture curves for all grain types became geometrically similar and could be superimposed by three “unifying parameters.” After the unifying adjustments, all grain types could be described by a single calibration equation.

- The water in grain (for dry to moderately moist samples) appears to be “bound” in the sense that it does not freeze at 0 °C, but there seems to be a difference in the dielectric response of “tightly-bound” or “monolayer” water and the dielectric response of the rest of the water in grain.

- The dominant mechanism of conductivity in grain is probably percolating protonic conductivity along chains of hydrogen bonds rather than ionic (salt) conductivity.

Based on these insights, GIPSA developed the “Unified Grain Moisture Algorithm” (UGMA). In this method, many similar types of grains (such as wheat classes, rough rice classes, edible beans, processed rice, etc.) are grouped together to use exactly the same calibrations. Furthermore, distinctly different types of cereal grains and oilseeds are combined into a single calibration by means of unifying parameters as described below. The accuracy of the method, with a single unified calibration, was shown to be as good as or better than that achievable with current dielectric grain moisture meters using separate calibrations for each distinct grain type. [2]

GIPSA decided to publish the UGMA as a public algorithm rather than seeking patent protection and exclusive licensing. GIPSA did this to enable multiple manufacturers to design and produce grain moisture meters that would require little or no calibration development effort and would yield equivalent moisture measurement results for different meter models. Also, GIPSA has supported research at Corvinus University of Budapest to further refine the algorithm and answer several researchable questions that are of common interest to all participating manufacturers. Two other papers in these proceedings address some of these questions. [4, 5]

This paper provides the an overview of the Unified Grain Moisture Algorithm, descriptions of some of the enhancements that have been developed since the original publication, and performance data for over 6,000 samples representing 53 U.S. grain types and six crop years.

2 Materials and Methods

Obtaining thousands of grain samples that are representative of diverse growing regions, varieties, moisture levels, and crop years is very difficult. Furthermore, determining the “true” moisture contents of those samples by a well-controlled reference method is extremely expensive. This research overcame those limitations by using the same samples and reference analyses as the GIPSA Annual Moisture Survey. [6] This program involves collecting and testing about 1200 grain samples per year that represent all significant growing areas for all
US grain crops that are assigned to GIPSA for quality certification. The purpose of the Annual Moisture Survey is to ensure that official moisture meters (used by the US Official Inspection Service) are calibrated to provide the best accuracy possible with respect to the USDA air oven method. Thus, this RF dielectric research project was a logical and effective extension of the Annual Moisture Survey. Table 3.1, which shows the performance of the UGMA for six crop years, also identifies the grain groups and numbers of samples tested.

The dielectric measurement system consisted of a Hewlett-Packard HP-4291A RF Material/Impedance Analyzer and the previously mentioned parallel-plate transmission line test cell developed by Lawrence and Nelson. (Fig. 1) The HP-4291A is a one-port impedance analyzer that covers the range of 1 to 1800 MHz. [7] In this research, complex reflection coefficient data were recorded at 2 MHz intervals from 1 to 501 MHz.

The overall length of the open portion of the test cell was 600 mm. The spacing between the 90 mm high plates was adjusted to 31.4 mm to yield a characteristic impedance of very nearly 50 ohms. The grain-filled section (centered in the 600 mm section) was 152.4 mm long. Thin polystyrene plates were installed in the test cell to define the grain-filled section. A 6 mm thick PVC plate formed a sliding gate under the grain-filled section to permit emptying samples without moving the test cell. The purpose of the air-filled sections on either side of the grain section was to avoid effects caused by non-TEM modes that might be excited at the abrupt transitions at either end of the test cell. The test cell was terminated with a precision 50-ohm load (HP-909C).

Funk [2] provides details of the measurement method, which is summarized here. Sample temperature was measured before loading through a funnel. The test cell was always overfilled, and the excess grain was removed by striking off to achieve a constant grain volume. After the dielectric measurements, the measured sample portion was weighed.

Fig. 1 Transmission line test cell and HP-4291A RF Material/Impedance Analyzer

3 Results and Discussion

3.1 Test Cell Standardization

The measured complex reflection coefficient values at 149 MHz were converted to complex permittivity values by means of an equation derived from a signal flow graph model of the test cell. The signal flow graph and the iterative solver (written in Mathcad Version 2001) [8] were previously described.[2] Mathematical models based on ABCD matrices were also
developed and tested. The ABCD matrix approach was capable of modeling non-ideal test cell characteristics (load impedance, characteristic impedance, discontinuities, etc.) more easily than the signal flow graph approach, but both methods gave essentially equivalent results for the test cells that were used in this research.

Kurt C. Lawrence, who performed the original modeling, adjustment, and calibration of the test cell, provided measurements of several types of alcohols. The test cell parameters for the signal flow graph were determined by optimizing the agreement between the predicted complex permittivity (from complex reflection coefficient) and the known dielectric characteristics of the high-purity alcohols.[2]

Subsequent research [9] has shown that the data needed to be corrected for two effects that were caused by the test cell design. The presence of dielectric materials in proximity to the test cell (specifically the cell “gate”) caused the calculated dielectric constant of the empty test cell to be other than 1.000. To correct this, the empty-cell dielectric constant \( \varepsilon_{ec} \) (calculated from the empty-cell reflection coefficient) and an offset value \( corr \) were subtracted from the measured relative complex permittivity \( \varepsilon_{meas} \) for the sample. (Eq. 1) Secondly, the parallel-plate transmission line test cell did not support a true transverse-electric-magnetic (TEM) mode with grain in the test cell. An effective filling fraction \( FF \) [10] was needed to convert the measured relative complex permittivity to “actual” relative complex permittivity. The real part \( \varepsilon_r \) of the relative complex permittivity was used for further calculations.

\[
\varepsilon_r = \text{Re} \left( \varepsilon_{meas} - \varepsilon_{ec} - corr \right) FF^{-1} + 1
\]  

(Eq. 1)

Fig. 2a shows calculated \( \varepsilon_r \) versus reference moisture values for 6,189 samples that represent 53 different grain types and six crop years.

3.2 Density Correction

Funk [2] showed that the density-corrected dielectric constant is much more useful for grain moisture prediction than the uncorrected dielectric constant. The Landau and Lifshitz, Looyenga dielectric mixture equation as restated by Nelson [3] was found to be particularly effective for adjusting the dielectric constants of different grain samples to a common density (target density). This correction minimized errors and non-linearity caused by three major sources of density variations: moisture level, filling method, and sample-to-sample variation. Eq. 2 was used for density correction in the UGMA.

\[
\varepsilon_{dc} = \left( \varepsilon_{r} \left[ \left( \frac{\rho_{target}}{\rho_{sample}} \right) + 1 \right] \right)^{3}
\]

(Eq. 2)

where: 
\( \varepsilon_{dc} \) is the density-corrected dielectric constant
\( \varepsilon_r \) is the dielectric constant
\( \rho_{target} \) is the target density (67.4 kg/hl)
\( \rho_{sample} \) is the sample density (kg/hl)

A target density of 67.4 kg/hl was chosen because it was the average density of the samples tested. The choice of target density was arbitrary; trials with different values showed that the specific value did not affect the accuracy of the resulting calibrations. However, the target density determined the slope of the relationship between density-corrected dielectric constant
and moisture and affected the specific polynomial calibration coefficients that were found by regression. Fig. 2b shows the density-corrected dielectric constant versus (reverse temperature corrected) air oven values (Eq. 3) for the same 6,189 grain samples. A reduction in scatter and improved linearity (within groups) is evident.

Fig. 2 Visualizing the Unified Grain Moisture Algorithm. a) Dielectric constant versus reference moisture for 6,189 samples representing 53 types of U.S. cereal grains, oilseeds, pulses, and processed rice for 1998 through 2003 crop years. b) With density correction and reverse temperature correction (of moisture values). c) With slope (SP) adjustment. d) With offset (OP) adjustment. e) With translation (TP) adjustment. f) With best-fit 5th order polynomial calibration curve superimposed on the data.

3.3 Unifying Adjustments

For each grain group, the slope (% moisture per unit of density-corrected dielectric constant) was calculated for the subset of samples with reference moisture values between 10 and 20 %
moisture. (Grain types were initially assigned to groups based on similarities in chemical and physical characteristics. Group memberships were later adjusted as needed to minimize calibration error.) The samples below 10 percent moisture and above 20 percent moisture were excluded from the slope calculation because the “standard” curve shape is non-linear in those regions. The slope unifying parameter $SP$ (Eq. 4) was defined as the correction factor needed to adjust the slope of each grain group to 6.000 in the 10 to 20 % moisture range. The data for the different grain groups appeared to rotate about a point ($\%M = 5$, $\varepsilon_{dc} = 2.5$), so an offset unifying parameter $OP$ with a nominal value of 2.5 was hypothesized. Fig. 2c shows the data for the 6,189 samples with the slope unifying parameter applied. All grain groups are now parallel in the 10 to 20 percent moisture range.

The offset unifying parameter was iteratively adjusted for each grain group, resulting in Fig. 2d. A translation unifying parameter $TP$ was needed to align the curve shapes for the different grain groups in the low moisture region. Note that changing the translation parameter had the effect of translating the data for that grain group along the common (slope = 6.000) line. After applying the three unifying parameters to each grain group, the data appeared as in Fig. 2e. A fifth-order polynomial was fit to the data as shown in Fig. 2f.

$$M_{adj} = M_{ref} + K_{tc} \cdot (T - 25) + TP$$

$$\varepsilon_{adj} = (\varepsilon_{dc} - OP) \cdot SP + 2.5 + \frac{TP}{6}$$

$$M_{pred} = \left( \sum_{r=0}^{5} (\varepsilon_{adj} \cdot CC_r) \right) - TP$$

### 3.4 Temperature Correction

Temperature significantly influences the radio-frequency dielectric characteristics of grain; the dielectric constant generally increases with increasing temperature. Previous research [2] suggested a simple temperature correction function (Eq. 6) for reducing temperature-induced error in UGMA predicted moisture values.

$$M_{tc} = M_{pred} - K_{tc} \cdot (T - 25)$$

where $M_{pred}$ is the moisture value calculated from dielectric characteristics

- $K_{tc}$ is the temperature correction coefficient (% moisture per degree Celsius)
- $T$ is the measured sample temperature
- $M_{tc}$ is the predicted moisture content with temperature correction

A more effective (but more complicated) temperature correction function for the UGMA is described elsewhere in these proceedings. [5]

One of the research efforts at Corvinus University of Budapest involves predicting unifying parameter and temperature correction coefficient values from grain physical and chemical properties. Successful prediction of some or all of these parameters would reduce or eliminate the effort required to add more grain types to the UGMA. Space limitations prevent listing the specific unifying parameters and polynomial calibration coefficients. The most current values are available upon request from the authors.
3.5 Accuracy of the Unified Grain Moisture Algorithm

Fig. 3 and Table 3.1 present the results of the UGMA for the 6,189 samples. The overall prediction accuracy (standard deviation of error with respect to the air oven method) was 0.34 percent moisture. The error increases somewhat for high moisture grain; the grain groups that have very wide moisture ranges tend to have poorer overall prediction accuracy.

![Graph showing moisture prediction error](image)

Fig. 3 Moisture prediction error for 6,189 grain samples representing 53 US grain types and six crop years

Table 3.1 Grain samples tested 1998 – 2003 by grain group. Moisture ranges and standard errors of prediction

<table>
<thead>
<tr>
<th>Grain Group Name</th>
<th>Types in Group</th>
<th>Number of Samples</th>
<th>Minimum Moisture</th>
<th>Maximum Moisture</th>
<th>Error SEP (%M)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soy</td>
<td>1</td>
<td>795</td>
<td>6</td>
<td>26</td>
<td>0.18</td>
</tr>
<tr>
<td>Sorghum</td>
<td>1</td>
<td>216</td>
<td>5</td>
<td>25</td>
<td>0.22</td>
</tr>
<tr>
<td>Sunflower seed</td>
<td>2</td>
<td>564</td>
<td>4</td>
<td>25</td>
<td>0.38</td>
</tr>
<tr>
<td>Corn</td>
<td>3</td>
<td>1234</td>
<td>4</td>
<td>50</td>
<td>0.47</td>
</tr>
<tr>
<td>Oats</td>
<td>1</td>
<td>136</td>
<td>4</td>
<td>16</td>
<td>0.32</td>
</tr>
<tr>
<td>Wheat (except durum)</td>
<td>5</td>
<td>1232</td>
<td>7</td>
<td>26</td>
<td>0.22</td>
</tr>
<tr>
<td>Barley</td>
<td>2</td>
<td>383</td>
<td>5</td>
<td>21</td>
<td>0.28</td>
</tr>
<tr>
<td>Rough rice and durum</td>
<td>4</td>
<td>862</td>
<td>4</td>
<td>37</td>
<td>0.43</td>
</tr>
<tr>
<td>Peas</td>
<td>3</td>
<td>90</td>
<td>7</td>
<td>20</td>
<td>0.24</td>
</tr>
<tr>
<td>Mustard</td>
<td>2</td>
<td>39</td>
<td>5</td>
<td>13</td>
<td>0.29</td>
</tr>
<tr>
<td>Edible beans group 1</td>
<td>9</td>
<td>147</td>
<td>7</td>
<td>22</td>
<td>0.38</td>
</tr>
<tr>
<td>Triticale</td>
<td>1</td>
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<td>10</td>
<td>14</td>
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<tr>
<td>Processed rice</td>
<td>11</td>
<td>304</td>
<td>11</td>
<td>20</td>
<td>0.28</td>
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<tr>
<td>Long-grain proc. rice</td>
<td>2</td>
<td>56</td>
<td>11</td>
<td>21</td>
<td>0.23</td>
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<tr>
<td>Edible beans group 2</td>
<td>2</td>
<td>30</td>
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<td>17</td>
<td>0.41</td>
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<tr>
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<td>4</td>
<td>8</td>
<td>0.29</td>
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<tr>
<td>Safflower</td>
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<td>4</td>
<td>10</td>
<td>0.41</td>
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<tr>
<td>High-oil corn</td>
<td>1</td>
<td>33</td>
<td>9</td>
<td>29</td>
<td>0.18</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>53</strong></td>
<td><strong>6,189</strong></td>
<td></td>
<td></td>
<td><strong>0.34</strong></td>
</tr>
</tbody>
</table>
4 Conclusions

The Unified Grain Moisture Algorithm was shown to be applicable to many different grain types and provide good accuracy over wide moisture ranges. Research at Corvinus University of Budapest has improved the method and has made it more suitable for commercialization.

Acknowledgements

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References


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Development of TDR-Sensors for Moist Materials Using HFSS

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ABSTRACT. The spatial sensor characteristics of a 6cm TDR flat band cable sensor section was simulated with finite element modelling (High Frequency Structure Simulator-HFSS) under certain real conditions. The complex dielectric permittivity \( \varepsilon(\omega, \tau) \) or complex electrical conductivity \( \sigma(\omega, \tau) \) of saturated and unsaturated soils was examined in the frequency range 50MHz-20GHz at room temperature and atmospheric pressure with a HP8720D- network analyser. The simulation is performed with a \( \lambda/3 \) based adaptive mesh refinement at a solution frequency of 12.5GHz. The electromagnetic field distribution, S-parameter and step responses were examined.

Keywords: lossy dielectrics, finite element modelling, HFSS, dielectric spectroscopy, fractional relaxation

1 Introduction

Soil science, geophysical prospecting, agriculture, hydrology, archeology and geotechnical engineering have benefited greatly from developments in radio and microwave technology. Electromagnetic techniques are used to estimate soil and rock physical characteristics such as water content, density and porosity [0, 0, 0, 0]. Both invasive methods, such as time domain reflectometry [0, 0] and cross borehole radar [0], and noninvasive methods, such as capacity methods and ground penetrating radar [0, 1, 0, 0], are used. Common to all these techniques is the fact that electromagnetic wave interaction depend on dielectric properties of rock or soil deposit through which it travels, which are influenced by chemical composition, mineralogy, structure, porosity, geological age and forming conditions. Besides, several additions have an effect on the dielectric properties like ubiquitous water.

In particular, knowledge of the spatial and temporal variability of water saturation in soils is important to obtain improved estimates of water flow (and its dissolved components) through the vadose zone. Due to its accuracy and potential for automated measurement, TDR has become one of the standard methods to measure spatial and temporal variability of water contents in laboratory soil cores and experimental field plots [0].

In this study the spatial sensor characteristics of a 6cm TDR flat band cable sensor section was simulated with finite element modelling (High Frequency Structure Simulator-HFSS) under certain conditions: (i) in direct contact to surrounding material, (ii) with consideration of a defined gap of variable size filled with air or water and (iii) cable sensor pressed at a borehole-wall.

2 Materials and Methodology

Time domain reflectometry measures the propagation velocity of a step voltage pulse (typical values of a tektronix cable tester: rise time \( \sim 200ps \), sampling increment \( \Delta t \sim 20ps \)) with a bandwidth of around 20kHz to \( \sim 25GHz \) (Nyquist-frequency: \( f_{\text{Max}} = 0.5/\Delta t \)). The velocity of this signal is a function of the frequency dependent effective complex permittivity \( \varepsilon_{\text{eff}}(\omega) = \varepsilon_a(\omega) - i\varepsilon_a^*(\omega) \) of the material through which it travels with potential modification by conductive losses \( \varepsilon_{\text{eff}} = \varepsilon_a + \sigma_{\text{DC}} / (i\omega\varepsilon_0) \) due to a direct current electrical conductivity \( \sigma_{\text{DC}} \) [0].
It is often convenient to consider the analogy of propagation phase velocity \( v_p(\omega) \) and attenuation \( \beta(\omega) \) of an electromagnetic plane wave:

\[
v_p(\omega) = c \sqrt{2 \left( \sqrt{\varepsilon_{\text{eff}}^{\prime\prime}(\omega) + \varepsilon_{\text{eff}}^{\prime\prime}(\omega)} + \varepsilon_{\text{eff}}^{\prime}(\omega) \right)^{-1}}
\]

\[
\beta(\omega) = \omega \sqrt{\varepsilon_{\text{eff}}^{\prime\prime}(\omega) + \varepsilon_{\text{eff}}^{\prime\prime}(\omega) - \varepsilon_{\text{eff}}^{\prime}(\omega)} \cdot \left( c \sqrt{2} \right)^{-1}
\]  

where \( \omega = 2\pi f \) is the angular frequency and \( c \) the velocity of light [0, 0]. Hence any modulation of an electromagnetic wave in a real medium will propagate at a group velocity according to the Rayleigh equation [0]

\[
v_g = \frac{d\omega}{dk} = v_p \left[ 1 - f \frac{dv_p}{df} \right]^{-1}.
\]

Fig. 1 (top) Phase velocity \( v_p \), high frequency approximation \( v_p' \), corresponding group velocity \( v_g \) and (bottom) attenuation \( \beta \) according to (2.2). Also included corresponding high frequency approximation \( \beta_h \) of (left) a sand-bentonite mixture (SB 50/50) with different gravimetric water content \( c_w \) and bulk density \( D \) (see section 3, Tab. 1, Fig. 2). (right) Comparison of a millisil® quartz powder (median grain size 11 \( \mu \)m, water content \( c_w=25.47\text{wt.\%} \), bulk density \( D=1.55\text{g/cm}^3 \), \( \sigma_{DC}=0.012\text{S/m} \)), natural water (\( \sigma_{DC}=0.041\text{S/m} \)) and sand bentonite mixture SB 50/50-4 (c.f. Tab. 1).
The flat band cable of length $l$ consists of three strip conductors embedded in a polyethylene band $[0, 0]$. The effective group or phase velocity of the signal in a perfect dielectric (pure real $\varepsilon_r = \bar{\varepsilon} = \text{const.}$ without dispersion and conducting losses) surrounding the cable sensor is in principle only a crude approximation especially at frequencies $f < 1\text{GHz}$ and $f > 10\text{GHz}$ for real soils (c.f. Fig. 1)

$$v_p' = \frac{2l}{t} = \frac{c}{\sqrt{\varepsilon_r}}$$  \hspace{1cm} (2.4)

where $t$ is two way travel time. Considering anomalous dispersion equation (2.4) is referred to as a high frequency approximation of phase velocity (Fig. 1). In contrast the high frequency attenuation approximation for real soils works considerably well (c.f. Fig. 1)

$$\beta_h(\omega) = \frac{\omega \varepsilon_0 \varepsilon_{\text{eff}}}{\sqrt{\varepsilon_{\text{eff}}}} Z_0$$  \hspace{1cm} (2.5)

with impedance $Z_0$ and permittivity $\varepsilon_0$ of free space. We now consider the soil as a four-phase medium composed of: air, quartz grain, water and clay. In the particular case of spatial TDR the surrounding medium in the direction of the band cable is described by a relative effective permittivity $\tilde{\varepsilon}_{\text{eff}}(\omega, \tau, x)$. It depends on position $x$, angular frequency $\omega$ and contribution due to several relaxation processes via relaxation time $\tau(T, p)$ on absolute temperature $T$ and pressure $p$

$$\tau_i(T, p) = \frac{h}{k_B T} \exp \left( \frac{E_{a_i}(T, p)}{RT} \right).$$  \hspace{1cm} (2.6)

Herein, $h$ denotes the Planck-constant, $k_B$-Boltzmann constant, $\kappa_i \approx 1$ the transmission coefficient, $R$ gas constant and $E_{a_i}(T, p) = \Delta G_i(T, p) + T \Delta S_i(T, p)$ activation energy of the $i$-th process [0]. Dielectric loss spectra of saturated and unsaturated soils very often show a marked deviation from simple Debye-behaviour [0, 0, 0, 0]. Based on the theory of fractional time evolutions Hilfer [0] derived a Jonscher type function [0] for the complex frequency dependent dielectric permittivity of amorphous and glassy materials

$$\tilde{\varepsilon}_{\text{eff}, i}(\omega, \tau_i) - \varepsilon_\infty = \frac{\Delta \varepsilon_i}{(j\omega\tau_i)^{\alpha_i} + (j\omega\tau_i)^{\beta_i}}.$$  \hspace{1cm} (2.7)

with high frequency limit of permittivity $\varepsilon_\infty$, relaxation strength $\Delta \varepsilon_i$, angular frequency $\omega$ and stretching exponents $0 \leq \alpha_i, \beta_i$ similar to the familiar empirical Havriliak and Negami [0], Cole-Cole [2], Cole-Davidson [0] or Kohlrausch-Williams-Watts [0] dispersion and absorption functions. For the particular case $\alpha_i = 0$ and $\beta_i = 1$ (2.7) transforms to the Debye model.

3 Experiments

The complex dielectric permittivity $\tilde{\varepsilon}(\omega, \tau)$ of saturated and unsaturated soils was examined in the frequency range $50\text{MHz}$-$20\text{GHz}$ at room temperature and atmospheric pressure with a
HP8720D- network analyser. This was performed using a combination of open-ended coaxial-line (HP85070B) and coaxial transmission line technique (sample holder (7x16x100)mm³). Different natural and synthetic soils were investigated. Here, we present our results for synthetic soil SB50/50. It is a mixture of 50wt.% sand (grain size <2mm) and 50 wt.% bentonite (Calcigel®: 71wt. % Ca-dioctahedral smectite, 9wt.% illite/dioctahedral mica, 1wt.%kaoline, 1wt.% chlorite, 9wt.% quartz, 5wt.% feldspar, 2wt. % calcite, 2wt.% dolomite) with dry density \(D_{\text{dry}}=1.33\text{g/cm}^3\). The synthetic soil samples were incrementally wetted from air dry up to saturation with natural water and equilibrated 12h. After each dielectric measurement bulk density \(D\) as well as gravimetric water content \(c_w\) were determined.

Three relaxation processes are assumed to act in the investigated frequency-temperature-pressure range: one primary \(\alpha\)-process (main water relaxation) and two secondary \((\alpha', \beta)\)-processes due to clay-water-ion interactions (bound water relaxation and the Maxwell-Wagner effect). The effective permittivity of a multiphase mixture \(\tilde{\varepsilon}_{\text{eff}}\) can be determined by the complex relative permittivity of water \(\tilde{\varepsilon}_w\), bound water \(\tilde{\varepsilon}_b\), the contribution due to clay-water-ion interaction \(\tilde{\varepsilon}_{\text{Clay}}\) as well as the real and constant permittivity of quartz grain \(\varepsilon_{\text{Sand}}=4.5\) and air \(\varepsilon_{\text{air}}\). The dielectric relaxation behaviour of each process is described by a fractional relaxation model according to (2.7) considering relaxation time distributions \(H(\tau)\). This allows the complete spectrum to fit as a function of water content \(c_w\) and bulk density \(D\) with the use of a generalized dielectric response (GDR):

\[
\tilde{\varepsilon}_{\text{eff}}(c_w, D) - \varepsilon_{\infty} = \sum_{i=1}^{N} \Delta \varepsilon_i (c_w, D) \left[ \frac{1}{\omega \tau_i (c_w, D)} + j \frac{1}{\omega \sigma_i (c_w, D)} \right] - j \sigma_{DC}(c_w, D) \frac{\omega \varepsilon_0}{\omega \varepsilon_{\infty}}. \quad (3.1)
\]
A Levenberg-Marquardt algorithm [0] is used to find best GDR fitting parameters (Tab. 1).

Table 1 Parameters of the three relaxation processes from GDR-fitting \( (i = [\alpha, \alpha', \beta]) \); water content \( c_w \) and bulk density \( D \) of SB-50/50.

<table>
<thead>
<tr>
<th></th>
<th>SB50/50-1</th>
<th>SB50/50-2</th>
<th>SB50/50-3</th>
<th>SB50/50-4</th>
<th>SB50/50-5</th>
<th>SB50/50-6</th>
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<tr>
<td>( c_w ) [wt. %]</td>
<td>5,0</td>
<td>11,4</td>
<td>18,1</td>
<td>28,1</td>
<td>33,5</td>
<td>41,5</td>
</tr>
<tr>
<td>( D ) [g/cm³]</td>
<td>1,14</td>
<td>1,37</td>
<td>1,34</td>
<td>1,75</td>
<td>1,52</td>
<td>1,66</td>
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<tr>
<td>( \varepsilon_\infty )</td>
<td>2,3</td>
<td>2,9</td>
<td>3,6</td>
<td>12,6</td>
<td>12,6</td>
<td>13,6</td>
</tr>
<tr>
<td>( \Delta \varepsilon_\alpha )</td>
<td>0,7</td>
<td>1,5</td>
<td>3,5</td>
<td>9,9</td>
<td>13,7</td>
<td>17,8</td>
</tr>
<tr>
<td>( \tau_\alpha ) [ps]</td>
<td>20</td>
<td>18</td>
<td>16</td>
<td>16</td>
<td>18</td>
<td>15</td>
</tr>
<tr>
<td>( \alpha_\alpha ) (fixed)</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>( \beta_\alpha )</td>
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<td>1,23</td>
<td>1,1</td>
<td>1,12</td>
<td>1,14</td>
<td>1,07</td>
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<tr>
<td>( \Delta \varepsilon_\alpha' )</td>
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<td>0,33</td>
<td>0,7</td>
<td>0,88</td>
<td>1,83</td>
<td>1,63</td>
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<tr>
<td>( \tau_\alpha' ) [ps]</td>
<td>73</td>
<td>111</td>
<td>130</td>
<td>136</td>
<td>136</td>
<td>145</td>
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<td>( \alpha_\alpha' ) (fixed)</td>
<td>0</td>
<td>0</td>
<td>0</td>
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<td>0</td>
<td>0</td>
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<td>( \beta_\alpha' )</td>
<td>0,95</td>
<td>1,11</td>
<td>1,2</td>
<td>1,3</td>
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<tr>
<td>( \Delta \varepsilon_\beta )</td>
<td>0,96</td>
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<td>1,37</td>
<td>0,93</td>
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<td>1,32</td>
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<tr>
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<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
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<td>( \beta_\beta )</td>
<td>0,32</td>
<td>0,35</td>
<td>0,21</td>
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<tr>
<td>( \sigma_{DC} ) [mS/cm]</td>
<td>4E-4</td>
<td>0,074</td>
<td>0,73</td>
<td>2,8</td>
<td>4,21</td>
<td>4,84</td>
</tr>
</tbody>
</table>

4 HFSS Simulation

The transfer or scattering function \( S_{ij}(\omega) \) of the flat band cable section (Fig. 3) was simulated by finite element modelling (High Frequency Structure Simulator-HFSS) under certain conditions: (i) in direct contact to the surrounding material (air, water of various salinities, different synthetic and natural soils (sand-silt-clay mixtures)), (ii) with consideration of a defined gap of various size (total high 2mm, 3mm or 5mm) filled with air or distilled water and (iii) cable sensor pressed at a borehole-wall.

Fig. 3 Model geometry of (left) flat band cable surrounded by saturated and unsaturated soil with a gap filled with air or water and (left) cable sensor pressed at a borehole-wall.
The simulation is performed with a $\lambda/3$ based adaptive mesh refinement at a solution frequency of 12.5GHz with an interpolating sweep in frequency range 1MHz-12.5GHz with extrapolation to DC. The electromagnetic field distribution, S-parameter and step response (200ps rise time) of the structure were computed in reflection and transmission mode.

Fig. 4 Electric field distribution @12.5GHz for the investigated flat band cable surrounded by air, sand-bentonite-mixture (SB_50/50-4) with $c_w=28,14$wt. % and $D=1.79$g/cm$^3$ as well as a defined 3mm air or water gap. (left) Cross section, (right) longitudinal section of middle conductor.

Fig. 5 (left) Input return loss magnitude or reflection coefficient $S_{11}$ and forward transmission or transmission coefficient $S_{21}$ as well as (right) TDR-waveform in reflection and transmission mode for simulated flat band cable structure, surrounded by air and a sand bentonite mixture of various water contents and bulk densities (see Tab. 1).
5 Discussion and Conclusion

The simulation adequately reproduces the spatial and temporal electrical and magnetic field distribution. High-lossy soils cause in dependence of increasing water content $c_W$ and bulk density $D$ a decrease of TDR signal rise time as well as a strong absorption of multiple reflections (Fig. 5, 6 and Tab. 2). Air or water gap work as quasi wave-guide, i.e. the influence by surrounding medium is strongly reduced (Fig. 4, Tab. 2). Appropriate TDR-travel-time distortions can be quantified (Tab. 2, Fig. 7). Further investigations in combination with reconstruction algorithms have to point out to what extent the accuracy of water content profiles can be determined in high lossy materials.

Fig. 6 TDR-waveform in (left) reflection and (right) transmission mode simulated for case (iii) cable sensor pressed at a borehole-wall with various borehole fillings compared to the flat band cable sensor.

Table 2 Simulation results of the different configurations obtained from TDR-data (c.f. Fig.).

<table>
<thead>
<tr>
<th>Configuration</th>
<th>$t_{onset}$ [ns]</th>
<th>$t_{rise}$ [ns]</th>
<th>$\varepsilon_{eff}$ (2.4)</th>
<th>$v_{eff}$ (2.4) [cm/ns]</th>
<th>$Z'_{\text{cable}}$ [Ω]</th>
</tr>
</thead>
<tbody>
<tr>
<td>flat band cable</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>air</td>
<td>0.386</td>
<td>0.533</td>
<td>1.334</td>
<td>25.892</td>
<td>83.8</td>
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<td>SB 50/50-0</td>
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<td>0.559</td>
<td>1.727</td>
<td>22.749</td>
<td>84.1</td>
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<td>0.630</td>
<td>1.941</td>
<td>21.461</td>
<td>85.1</td>
</tr>
<tr>
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<td>0.501</td>
<td>0.663</td>
<td>2.246</td>
<td>19.952</td>
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<td>4.113</td>
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Fig. 7 Real effective relative permittivity $\varepsilon_{\text{eff}}$ according to equation (2.4) plotted against TDR rise time $t_{\text{rise}}$ (in reflection mode) for all sensor configurations and investigated cases.

Acknowledgement

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Modelling of Electromagnetic Wave Propagation along Transmission Lines in Inhomogeneous Media

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ABSTRACT. Time domain reflectometry (TDR) is a well-established method for the measurement of moisture in various materials, especially soils. Standard waveform analysis usually provides the average water content along the length of the TDR probe, while more sophisticated methods try to reconstruct the spatial water content profile. A reconstruction algorithm has been developed which uses one- or two-sided reflection data to calculate capacitance and conductivity distributions from which water content profiles can be derived. Several examples demonstrate the performance of the algorithm under various conditions in lossless and lossy materials.

Keywords: time domain reflectometry, spatial resolution, water content profile, reconstruction

1 Introduction

Knowledge of moisture content is essential to many applications in hydrology, agriculture, and civil engineering. One of the standard measurement methods is time domain reflectometry (TDR) providing automated moisture monitoring with high accuracy. TDR probes usually consist of two- or three-wire transmission lines which are embedded in the material under test (Fig. 1). A fast rise time voltage step propagates through the system being partly reflected at the coaxial cable to probe transition and totally reflected at the end of the probe. These reflections show up in the TDR trace from which the wave velocity can be derived. Together with an electrical equivalent circuit of the transmission line and the dielectric characteristics of the material under test the mean water content is calculated.

Fig. 1 Basic TDR set-up and typical TDR trace (sum signal). Oscilloscope and pulse generator are usually integrated in a single TDR instrument.

For many applications this standard waveform analysis is not sufficient. Instead of the mean water content spatial distribution of moisture is required. One approach has been developed by Hook et al. [1] dividing the transmission line into several sections by remote-controlled switches. Others change the diameter of the probe to create a series of characteristic reflections. Besides the coarse local resolution these method may fail at layered materials with
high conductivity. More sophisticated methods consist of modelling the wave propagation along the transmission line in inhomogeneous media and solving the inverse problem in order to retrieve the water content profile. These methods are usually based on the following electrical equivalent circuit of a very short or infinitesimal piece of the transmission line (Fig. 2). Each section of the line may have different line parameters according to the water content of the surrounding material.

$$\begin{align*}
\text{RL} & \quad \text{GCV}(x) \\
I(x) & \quad \text{V}(x) \\
I(x+\Delta x) & \quad \text{V}(x+\Delta x)
\end{align*}$$

Fig. 2 Equivalent circuit of a short section of a transverse electromagnetic (TEM) transmission line. V(x) and I(x) are the voltage and the current at the beginning and at the end of the section.

The response of the transmission line to an incident step impulse can be calculated in the time domain [2] or in the frequency domain [3,4]. Typical relations between the electrical equivalent circuit and the water content are given by Fig. 3a,b. The capacitance of the transmission line depends on the relative dielectric permittivity \(\varepsilon'\) of the surrounding material which is related to the water content as well.

![Fig. 3](image_url)

Fig. 3 (left) Examples for water content to permittivity [5] and (right) capacitance to permittivity relations [2]

### 2 Step Response of Inhomogeneous Transmission Lines

Fig. 4 shows several examples of water content profiles along a transmission line and the corresponding responses to step-like electrical pulses. Low water content sections are identified by high reflected voltages and vice versa. For more complicated and/or continuous profiles the step response is influenced by many multiple reflections. Then more sophisticated methods are required to analyse the waveform and reconstruct the water content profile.
Fig. 4 Examples for water content profiles (left) and step response (right) of a 2 m long transmission line embedded in lossless material.

In case of lossy materials the analysis of the waveform is complicated. The simulations in Fig. 5 assume that the conductivity is proportional to the capacitance. This is often a reasonable approximation, because losses are mainly due to soluted ions in the water fraction. Increasing water content raises capacitance and conductivity at the same time. In the step response the losses reduce the reflected voltage until the partial reflections from the transitions between different water content sections are barely visible. Then, amplitude resolution of the TDR instruments and noise are limiting factors for any analysis method.
Fig. 5 Examples for the water content profiles (left) and step response (right) of a 2 m long transmission line embedded in a lossy material.

3 Reconstruction Algorithms

The reconstruction of line parameters has already been investigated both in time and frequency domain. The methods presented by Lundsted and He [6] and Schlaeger et. al [7] are computationally fast algorithms in the time domain for frequency independent line parameters, where the latter is especially suited for automated calculations and batch processing. Frequency domain algorithms [3,4] are computationally much slower, but include frequency dependent line parameters. In order to account for arbitrary dispersive line parameters a new reconstruction algorithm in the frequency domain has been developed. It is also suited for dielectric coated transmission lines with sophisticated electrical equivalent
circuits. Furthermore high frequency models of the coaxial cable to probe transition may be included for a more accurate description of wave propagation.

Fig. 6 shows the block diagram of the reconstruction algorithm. The TDR signal (step response) is calculated from assumed water content profiles and compared with the measurement. An initial water content profile is iteratively adjusted until a sufficient match between calculated and measured TDR signal is achieved. The algorithm has been implemented in Matlab using the Optimization Toolbox [8]. For each iteration the frequency domain response $S_{11}(f)$ has to be calculated and transformed into the time domain which is computationally slow, but a single transformation of the measured/simulated signal and comparison in the frequency domain has not been successful.

Fig. 6 Block diagram of the reconstruction algorithm

4 Reconstruction Examples

Fig. 7 shows an example for a reconstruction of a water content profile in lossless material ($G=0$). The true profile consists of several sections with water contents between 10% and 35%, whereas the initial profile for the reconstruction algorithm is constantly 5%. The corresponding step responses are clearly different at the beginning. After several iterations true and reconstructed signal are getting closer as well as true and reconstructed water content profile until the algorithm converges. Increasing the spatial resolution further improves accuracy (Fig. 8) at the cost of computation time.
Fig. 7 Reconstruction example in lossless material. Left side: water content profiles, right side: step response.
Fig. 8 Reconstruction example in lossless material. Left side: water content profiles, right side: step response. Spatial resolution is doubled compared to Fig. 7.

The reconstruction algorithm is well suited for lossy materials if the relation between capacitance and conductivity is known, even in the case of noisy signals. Fig. 9 shows a reconstruction example for this case. The noise is low-pass filtered by the algorithm according to the spatial resolution.

Fig. 9 Reconstruction example in lossy material with noisy step response. Left side: water content profiles, right side: step responses. Capacitance $C$ is assumed to be proportional to conductivity $G$.

If the relation between capacitance and conductivity is not known the algorithm has to reconstruct both profiles which doubles the number of unknowns. This is hardly possible from one-sided measurements. From two-sided measurements of the step response it is possible to retrieve both profiles, which can be later transformed into water content again. Fig. 10 shows on the left a capacitance and a conductivity profile. The corresponding step responses from the left and from the right side of the transmission line are shown on the right. Both have the same time to the hard reflection at the open end of the line, but the partial reflections are different of course.
Fig. 10 (left) Capacitance and conductivity profile for a 2 m long transmission line embedded in lossy material. (right)step responses from the left and the right end of the line. The other end is always left open.

The reconstruction results in Fig. 11 show a sufficient match between true and reconstructed profiles. A finer spatial resolution would even give better results at the cost of computation time.

Fig. 11 (left) True and reconstructed capacitance profile. (right) True and reconstructed conductivity profile.

The disadvantage of two-sided measurements is the requirement of two connections at both ends of the transmission line of course. Therefore standard TDR probes are replaced e.g. by flat band cables which require the development of new installation procedures in the field.

5 Conclusion

Reconstruction of water content profiles is essential for advanced measurement tasks in hydrology, agriculture and civil engineering. A reconstruction algorithm has been developed which is suited both for lossless and lossy materials. It is based on a frequency domain optimization and accounts for dispersive materials. Using one-sided measurements capacitance respectively water content profiles can be retrieved when the electrical loss mechanism is known. Otherwise two-sided measurement are required for calculating capacitance and conductivity profiles separately. Several simulation examples have shown the performance of algorithm under various conditions. Practical applications in the field will
suffer from the limited amplitude resolution of TDR instruments and noise. Further investigations will be conducted to quantify this influence on the reconstruction accuracy.

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The Theoretical Model of the Microwave Complex Permittivity of Grain

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ABSTRACT. The new theoretical model of calculation of the grain complex dielectric permittivity depending on its moisture in the microwave region is submitted. In this model the permittivity of a grain material is determined on the basis of the Reynolds-Hough’s formula for heterogeneous mixtures, in which the uncertain complex kernel form factors are introduced. The value of these factors is selected by the condition of the best approach between the theoretically derived values of dielectric permittivity of a grain material and experimental data.

Keywords: dielectric permittivity, grain, moisture.

1 Introduction

The non-contact electrodynamic methods are widely applied for moisture content measurement in a grain during harvesting, storage, purification and conversion [1-4]. Its work principle is based on the effect of the essential difference between the dielectric permittivities of water and grain dry substance in the microwave region. For effective application of these methods it is necessary to know the functional dependence of grain materials dielectric permittivity on their moisture, which is of a rather complex nature. The study of such dependence was very attentive during long time [5-7]. However the majority of articles in the given field are of experimental character, and the known theoretical models are usually reduced to mathematical approximations of the empirically established dependencies of grain dielectric permittivity on moisture, frequency of radiation, temperature etc. [7]. The physical model of grain material dielectric permittivity calculation for microwave region has not been developed so far. This work is an attempt to construct such physically proved model for a wide region of grain moisture change.

2 Theoretical Bases of Model

From the physical-chemical point of view a grain is characterized as the structured heterogeneous system, which consists of kernel solid organic substance, air between grains, air microcapillaries in them and also of water absorbed by grains which partially fills these microcapillaries [8]. To model the dielectric properties of such systems rather effective theoretical methods have been developed [9]. They allow to take into account localization of every component in the volume of a mixture. These methods use the simplifying assumption of rather low frequency of penetrating electromagnetic radiation, when its wave length considerably exceeds the sizes of separate dispersed particles in a mixture (in this case it is grains). It allows to consider them as the particles of a small electrical size, and to consider interaction between the field and them in electrostatic approach [10]. Thereby it make it possible to describe the dielectric property of separate particles and all mixture with the help of the simple analytical formulas.
As is known from experience, a grain substance well absorbs water: even at large values of moisture content, more than 20 %, water is accumulated only inside the grains and does not form separate particles outside their limits. It means that the space between grains is filled only by air, and the grain material as a whole can be considered as a disordered mixture of grains and air gaps between them. The dielectric permittivity \( \varepsilon \) of such mixture can be described by the known Reynolds-Hough’s formula [9]:

\[
\varepsilon = 1 + (\varepsilon_g - 1) v K ,
\]

where \( \varepsilon_g \) is the dielectric permittivity of separate grains, \( v \) is their volume concentration in the entire volume, \( K \) is the ratio of the average values of electrical field intensity inside the grains and in the entire volume of a material. The concentration of grains \( v \) can be determined through measurements of the material bulk density \( \rho \) and kernel density of separate grains \( \rho_g \):

\[
v = \rho / \rho_g .
\]

For the calculation of mixtures dielectric properties the \( K \) value is usually defined at the assumption that every dispersed particle of a mixture is immersed into continuous infinite equivalent media with a certain effective dielectric permittivity [9]. For the disorder mixtures with high component concentration (as in our case) the last is supposed to be equal to the dielectric permittivity of all mixture \( \varepsilon \). Then the general expression for \( K \) value is possible to be written down as:

\[
K = \frac{A}{1 + B \varepsilon_g / \varepsilon}
\]

with some factors \( A \) and \( B \), which are determined by the form of grains. Till now relations (1) and (3) were used for theoretical modeling of such heterogeneous systems, for which the factors \( A \) and \( B \) are beforehand known [9]. They are systems with a regular form of dispersed particles as an ideal sphere (\( A=3/2, B=1/2 \)) or cylinder (\( A=2, B=1 \)) [10]. But such approach does not allow practically to take into account deviations from regular form for mixture particles. To overcome this restriction, we shall use other approach, at which the value of factors \( A \) and \( B \) is not determined beforehand, but is established subsequently by comparison of theoretical results and experiment. It is possible yet to assume that the value \(|A|\) will be in a range of approximately from 3/2 up to 2, and \(|B|\) from 1/2 up to 1, because the form of the grains is characterized by an intermediate status between a sphere and a truncated cylinder.

The substitution (3) in (1) gives the equation relatively to the mixture dielectric permittivity \( \varepsilon \), which solution is:

\[
\varepsilon = C + \sqrt{C^2 + B \varepsilon_g} ; \quad C = [(A v - B) (\varepsilon_g - 1) + 1 - B] / 2 .
\]

To calculate the dielectric permittivity of separate grains \( \varepsilon_g \) it is necessary to take into account the features of moisture absorption by a grain. The observations show that at small values of moisture their kernel density \( \rho_g \), and also bulk density \( \rho \) of the entire grain material with the increase of moisture increase a little, and then begins to decrease [6]. These changes approximately correspond to the following dependencies (fig. 1):

\[
\rho_g = \{ a_g + b_g (1 - \tau_g (1 + \tau_g^2)^{-1/2}) \} / [1 + s_g (0.3 - w)^2] ; \quad \tau_g = (w - w_{0g}) / \Delta_g ;
\]

\[
\rho = \{ a + b (1 - \tau (1 + \tau^2)^{-1/2}) \} / [1 + s (0.3 - w)^2] ; \quad \tau = (w - w_0) / \Delta ,
\]

\[
\rho_g = \{ a_g + b_g [1 - \tau_g (1 + \tau_g^2)^{-1/2}] \} / [1 + s_g (0.3 - w)^2] ; \quad \tau_g = (w - w_{0g}) / \Delta_g ;
\]

\[
\rho = \{ a + b [1 - \tau (1 + \tau^2)^{-1/2}] \} / [1 + s (0.3 - w)^2] ; \quad \tau = (w - w_0) / \Delta ,
\]
where \( w \) is the moisture content of a grain material, which is defined by the ratio of water weight in it \( m_w \) to the general weight of all material \( m \):

\[
w = \frac{m_w}{m},
\]

(6)

\( a_g, b_g, s_g, w_{0g}, \Delta_g, a, b, s, w_0, \Delta \) are the factors determined by properties of grain substance and external physical conditions, in particular by temperature. In particular for a grain of a soft winter wheat at temperature \( t=20^{\circ}C \)

\[
a_g = 1.1; \quad b_g = 0.11; \quad s_g = 1.4; \quad w_{0g} = 0.17; \quad \Delta_g = 0.06;
\]

(7)

\[
a = 0.634; \quad b = 0.11; \quad s = 0.9; \quad w_0 = 0.15; \quad \Delta = 0.08.
\]

Thus the density of grains of zero moisture is \( \rho_{g0} = 1.167 \, \text{g/cm}^3 \), and the density of an absolutely dry grain is \( \rho = 0.778 \, \text{g/cm}^3 \).

Fig. 1 Dependencies of the bulk density of soft winter wheat \( \rho \), its average kernel density \( \rho_g \) and the ratio of the kernel volume increase on the volume of absorbed water \( \Delta V_g/V_w \) (8) on moisture content \( w \) at temperature \( t=20^{\circ}C \).

If the moisture, absorbed by grains, only filled microcapillaries inside them and displaced air from there, as in cellulose fibers of paper materials [11], kernel density would be increase with the increase of moisture, as it occurs at its small values. But it decreases further which informs about the increase of the kernel volume and appearance of other moisture absorption mechanisms. The only alternative is to assume, that particles of absorbed water are built between particles of grain organic substance, changing structure of the last and thereby increasing their volume.

With the help of expressions (5), (6) it is possible to estimate the relative value of kernel volume change \( \Delta V_g \) in comparison with absorbed water volume \( V_w \). The dependence of the ratio

\[
\frac{\Delta V_g}{V_w} = \frac{V_g - V_{g0}}{V_w} = \frac{1}{V_w} \left( \frac{1 - C_v}{w} \right) = \frac{\rho_w}{\rho_g} \left( \frac{1}{\rho_{g0}} - \frac{1 - w}{\rho_g} \right)
\]

(8)
on moisture content \( w \) for parameters of density (7) is shown in fig. 1. Here \( V_g \) and \( V_{g0} \) are volumes of moist grains and dry grains without water, \( \rho_w = 1 \text{ g/cm}^3 \) is the density of the last,
\[
v_w = \frac{V_w}{V_g} = w \frac{\rho_g}{\rho_w}
\]
is the water content in grains volume,
\[
C_v = \frac{V_{g0}}{V_g} = \frac{(m - m_w) / \rho_{g0}}{m / \rho_g} = \frac{\rho_g}{\rho_{g0}} (1 - w)
\]
is a factor describing relative increase of the grain volume. It is clear from fig. 1, that the value of ratio (8) grows with the increase of moisture and it reaches a unit at \( w = 25 \% \). Hence, in the region of large moisture the specific mechanism of water absorption by grains is shown which is accompanied by their volume increase. It is obvious, that the indicated features of moisture absorption process in a grain characterize its behavior at a physical level as a biological object.

We shall consider each separate grain as a system of three small-dispersed well hashed phases: water, solid organic substance and air capillaries. With regard to a such system the logarithmic Lichtenecker’s formula is correct [9]:
\[
\ln \varepsilon_g = v_m \ln \varepsilon_m + v_w \ln \varepsilon_w,
\]
where \( v_m \) is the volume of grain substance without water related to complete volume of grains, \( \varepsilon_m \) and \( \varepsilon_w \) are the dielectric permittivities of this substance and water. Since the dielectric permittivity of air is equal to a unit the appropriate component in (11) turns to zero. In the absence of water we would have for the same values:
\[
\ln \varepsilon_{g0} = v_{m0} \ln \varepsilon_m,
\]
where \( \varepsilon_{g0} \) is the dielectric permittivity of absolutely dry grains, \( v_{m0} \) is the concentration of grain substance in dry grains. Then in the right part of (11) the first component can be written as \( C_v \ln \varepsilon_{g0} \), where \( C_v = v_m / v_{m0} = V_{g0} / V_g \), and in view of relations (9), (10) formula (11) for the kernel dielectric permittivity will be transformed to a kind:
\[
\varepsilon_g = \exp \left( \rho_g [(1 - w) \rho_{g0}^{-1} \ln \varepsilon_{g0} + w \rho_w^{-1} \ln \varepsilon_w] \right).
\]
The unknown value of the dielectric permittivity of dry grains \( \varepsilon_{g0} \) together with the grain form factors \( A \) and \( B \) is established on the basis of experimental data.

3 Determination of Model Parameters from Experiment

The experimental foundation of grain dielectric permittivity was carried out by the moisture measurement microwave device for loose materials [4]. Its basic element is a volume coaxial cylindrical microwave resonator connected by means of a ring slot with a cylindrical region, where the investigated material was located. In the experiment the frequency \( f \) and the Q-factor of free resonant oscillations were directly measured by putting in it various moisture grain samples. Thereby the complex wave number \( k \) of resonant oscillations was found:
\[
\Re k = 2 \pi f / c; \quad | \Im k | = \Re k / 2Q,
\]
where \( c \) is the speed of light in a vacuum. Moreover the results of theoretical research were used, which describe the functional dependence of this number on complex permittivity of a
dielectric placed in the resonator. It appears that for the coaxial resonator the given dependence is very close to linear [4]. With its help the grain dielectric permittivity was found for test samples of various moisture, measuring the value of its content directly by a thermogravimetric method (by weight). The values of grain permittivity for soft winter wheat found in this way at frequency 3.6 GHz are shown by points in fig. 2.

![Fig. 2 Dependencies of real and imaginary parts (dielectric constant and loss factor) of the grain dielectric permittivity $\varepsilon$ and the kernel dielectric permittivity $\varepsilon_g$ on moisture $w$ for soft winter wheat at frequency 3.6 GHz and $t=20^\circ$C.](image)

For the determination of the form factors $A$ and $B$ and the dielectric permittivity of dry grains $\varepsilon_{g0}$ the appropriate theoretical dependencies were calculated by formulas (3) - (5), (7), (12) at various values of these parameters. Their value was selected by a condition of the minimal deviation of the received theoretical curves from experimental points (fig. 2) and additional condition of minimizing the factors $A$ and $B$ variations with moisture. In addition the dielectric permittivity of water was assumed equal: $\varepsilon_w = 76.8 + 16i$ [12]. It appears that the optimum correspondence between the theory and experiment takes place in the case when

$$\varepsilon_{g0} = 2.6 + 0.28i; \quad A = \frac{\alpha_a + B_a f(w)}{1 + w\gamma_a}; \quad B = \frac{\alpha_b + B_b f(w)}{1 + w\gamma_b},$$

where the moisture content $w$ is expressed in absolute units;

$$f(w) = 1 + \sigma (1 + \sigma^2)^{-1/2}; \quad \sigma = (w - 0.16) / 0.035;$$

$$\alpha_a = 1.6 + 0.1i; \quad \beta_a = 0.5 + 0.06i; \quad \gamma_a = 3; \quad \alpha_b = 0.6; \quad \beta_b = 0.06; \quad \gamma_b = 2.2.$$ 

The dependence of factors $A$ and $B$ (13) on moisture is shown in fig. 3. The existence of such dependence is caused by a biological nature of the studied object. One of these factors turn out to be complex, which can be explained by losses of electromagnetic field energy owing to its scattering on the surface of the grains. It arises because the condition of a little electrical size of grains satisfies roughly (a wave length, which is equal to approximately 8 cm, is only 16-20 times higher than the grain size), and also because there are various nonuniformities of their surface.
Fig. 3 Dependencies of the grain form factors $A$ and $B$ (13) on moisture $w$ for soft winter wheat at frequency 3.6 GHz and $t=20^\circ$C.

The numerical results submitted in fig. 2 as a whole are in agreement with the experimental data on grain permittivity of other authors [1, 5, 6]. Small divergences from these data are probably caused by the circumstance, that another region of microwave radiation was used and the grain of other kind was investigated (hard winter wheat) in the referred works.

4 Conclusion

It should be noted that the grain materials are very sensitive to external influences. Therefore it is necessary to take into account the dependencies of the dielectric permittivities and especially of grain substance density on temperature and other external conditions during the use of the suggested theoretical model. For example, the small external pressure or even repeated shake of a grain is sufficient to change the parameters of density (5), (7). Thus parameters (13) which are interpreted as the grain form factors, can change too, and in general the grain can become anisotropic as a whole owing to the occurrence of the primary spatial orientation of the grains-ellipsoids.

The complex grain form factors $A$ and $B$ (13), which define the ratio of the average values of an electrical field intensity inside grains to that in all volume of a material (3), play an important part in the considered model. They are used as free parameters for the agreement of the theoretical calculation with the experiment. It can be possible to complicate relation (3) by adding in its right part one more or even two similar component with new factors. The more so that the grain form is close to the form of a general ellipsoid, and for the last the ratio $K$ of field values inside and outside contains three different components [10]. However then the relation (1) turns to the cubic and even quartic equation in the material dielectric permittivity $\varepsilon$, from which it is all the same necessary to select the equation of lower order to find its unique physically acceptable solution. Therefore complication of dependence (3) by introduction of additional components in this case is inexpedient, but then the factors $A$ and $B$ lose the strict sense of dispersed particles form factors.

It is obvious, that a similar technique can be used to model the dielectric properties of other heterogeneous mixtures, in which dispersed particles have varied forms. The method of a
dispersed particles form factors variation offered in the present work, opens wide possibilities for physical modeling of dielectric properties for such complex heterogeneous systems, which defy theoretical study till now.

At last, we shall note one more circumstance. The theory suggested above considers only the bound water (specifically bound by a substance of grains) if to use this term as alternative to a free condition of water as liquid. But it is thought to have the same physical properties (density, dielectric permittivity), as free water, even if it is introduced between the particles of the organic substance in grains. However in some works (see for example [13]) the concept of the bound water is applied to the characteristic of its special condition, when it forms transitive layers on a surface of solid phase and its physical properties strongly differ from the properties of usual free water. In the submitted model, as well as in the model of a paper dielectric permittivity [11], the presence of such water condition is not taken into account, but it does not affect the good agreement of its results with experimental results of other authors. From here it is possible to derive a conclusion, that in usual heterogeneous systems (grain, paper) the particular bound water is not formed at all or its quantity is not enough to appreciably influence the macroscopic characteristics of such heterogeneous systems.

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Enhanced Integral Equation Modelling for Moisture Sensors

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ABSTRACT. Integral equation (IE) methods are viable for modelling the interaction between microwave signals and porous materials for measurement of moisture content. Practitioners of IE methods frequently use delta basis functions to represent the field in each cell, but the inherent approximations limit accuracy. Although higher order basis functions provide one alternative, here we retain the geometric and cell density advantages of delta functions, but address accuracy by correcting for the field integration errors of the conventional moment method. Our approach circumvents the errors that arise when assuming that the field of a cell is adequately represented by a source at its centre, and has resulted in close agreement between empirical results and the model. The new technique retains the rapid calculation and suitability for use in dielectric tomography and for modelling the spatial response of moisture sensors.

Keywords: TDR, heterogeneous, dielectric, integral equation.

1 Introduction

TDR (time domain reflectometry) is used extensively for measurement of $\theta$, the volumetric moisture content in soil, and is applicable but less widely used in other materials such as grains, powders, and minerals. For measurement of $\theta$, a short open-ended parallel transmission line or waveguide, typically 300 mm long, is buried in the material under test. The travel time of a pulse with very short risetime (typically < 300 ps) is measured and provides the mean propagation velocity $v_p$, on the line of known length. Since most biological and composite materials make negligible contribution to the permeability of the region, $v_p$ indicates the mean relative permittivity $\varepsilon_r$ of the material surrounding the waveguide. When the loss tangent is small, and the relative permeability is one, $\varepsilon_r$ may be obtained using:

$$\varepsilon_r \approx \frac{c^2}{v_p^2} \tag{1}$$

where $c$ is the velocity of light. Since $\varepsilon_r$ for most organic and mineral materials is typically in the range of three to five whereas that of water is typically 80, $\varepsilon_r$ of a material forms a useful surrogate for its moisture content. Frequently, empirical calibration techniques are used since practical dielectric models are usually unable to account for the subtle interactions between water molecules and the material that affect the water’s polarisability. For example, Topp et al [1] developed a polynomial relating the measured $\varepsilon_r$ to the moisture content of soil. This calibration is applicable to quite a wide range of soil types (and hence orders of magnitude variation in particle size with their attendant variable interactions with water molecules) and typically has an accuracy of better than 2% in $\theta$ over the range 5 to 50%.

We have previously shown [2] how an integral equation (IE) method can be used to model the field distribution around a TDR waveguide immersed in an arbitrary permittivity (and hence...
moisture content) distribution, and lead to a prediction of \( v_p \). The model has also been used to invert a set of data to provide a measure of moisture distribution [3], and here an IE method confers significant advantages. IE methods enable the problem to be solved once for a given permittivity distribution, and for any field distribution representing different positions of the waveguide, resulting in just one forward calculation per inverse iteration. This contrasts with DE methods where one solution is required for each waveguide position. The approach is thus generally more favourable than the otherwise more rapid DE methods, especially when coupled with rapid IE approaches such as the pseudo 3-D method [4].

When employing IE methods, delta basis functions and point matching are normally used. In the case described here, the depolarisation field in every discretised cell is calculated from the sum of the field contribution from the centre of all other cells. Higher order basis functions may also be used, but there is a compromise between the additional complexity and accuracy of the higher order basis functions, compared with gaining increased accuracy through finer discretisation but retaining delta basis functions. Point matching methods have been used in our work but their use does introduce an error which we have addressed by an alternative formulation. First we describe our existing method, and then detail the alternative approach.

2 Integral Equation and Discretisation

The polarisation of a discretised zone or cell within a dielectric material may be represented by a dipole at its geometric centre. In most dielectric materials, there is no net polarisation until generated by an external or impressed field. When applied to this quasi-static electric field problem where the material is considered lossless, the method of moments may be considered as the summation in each cell, of the electric field contributions due to the polarisation in all other cells. The potential \( \phi_p \) at point \( p(x,y,z) \) generated by polarisation \( P \), is:

\[
\phi_p = \frac{\vec{P}_r \cdot \hat{r}}{4\pi\varepsilon_0 r^2}
\]

(2)

where \( \hat{r} \) is a unit vector pointing from the centre of the cell to \( p \) [5]. In Cartesian 3-space:

\[
\phi_p = \frac{\vec{P}_r \cdot (\vec{x} + \vec{y} + \vec{z})}{4\pi\varepsilon_0 r^3}
\]

(3)

where \( \vec{x}, \vec{y} \) and \( \vec{z} \) are the rectangular components of \( \vec{r} \). The potential arising from many cells is:

\[
\phi_p = \iiint \frac{\vec{P}_r \cdot \hat{r}}{4\pi\varepsilon_0 r^2} dv
\]

(4)

where \( dv \) is the differential volume over which each \( \vec{P}_r \) applies. Reverting to the single dipole case, its electric field is the space rate of change of potential \( -\text{grad} \phi_p \) so that:

\[
E_{px} = -\frac{\vec{P}}{4\pi\varepsilon_0 r^5} \left[ \hat{x}(r^2 - 3x^2) - \hat{y}(3xy) - \hat{z}(3xz) \right]
\]

(5)
with corresponding equations for $E_{py}$ and $E_{pz}$. The above may be combined in an integral
equation describing the electric field $E_p$ at a point $p$:

$$E_p(x,y,z) = -\nabla \left( \oiint \frac{P^\wedge}{4\pi \epsilon_0 r^2} \, dv \right)$$

(6)

The polarisation region may now be discretised, and following the method of moments [6],
we calculate the matrix of polarisation vectors $P(x,y,z)$ using:

$$L(P) = -E_i(x,y,z)$$

$$= E_p(x,y,z) - \frac{P(x,y,z)}{\epsilon_0 \chi(x,y,z)}$$

(7)

where $L$ is a linear operator, $E_i$ the external impressed field and $\chi(x,y,z)$ the electric
susceptibility ($\epsilon_r(x,y,z) - 1$). Eqn (7) is converted to matrix form and solved for the vector of
polarisations $P$, and the electric field strength in each cell is recovered from the polarisation:

$$E(x,y,z) = \frac{P(x,y,z)}{\epsilon_0 \chi(x,y,z)}$$

(8)

The inputs required for the method are: a vector comprising sets of three elements describing
the impressed field, a matrix describing the permittivity within each cell, and the dimensionality of the problem. While the above method applies to any impressed field
distribution, in this case $E_i$ is the vector of impressed field components due to the waveguide,
where the transverse field is described by the $x$-$y$ plane, and the $z$ axis is parallel to the
waveguide rods. To obtain the potential difference between the two rods and hence determine
line capacitance, the matrix $E$ is integrated along a path between the rods in a transverse
plane. Then to obtain $v_p$ for the lossless case, the standard transmission line formula is used:

$$v_p = \frac{1}{\sqrt{LC}} = \frac{\pi \int E(x,y,z) \, dl}{q \mu \cosh^{-1}\left( \frac{b}{a} \right)}$$

(9)

Here $dl$ is the length element of the numerical integration (the cell length in this discretised
case), $q$ the same initial line charge density that defined the impressed field, $\mu$ the total
permeability, $b$ the transmission line rod spacing, and $a$ the rod diameter.

3 New Field Formulation

Implicit in the above formulation of the point matching method described by Eqn (6) is the
$1/r^3$ dependence for calculation of the far field contribution from the dipole at the centre of
each cell. While this approximation is valid for distant cells, the assumption for the near field
introduces errors, since point matching assumes that the total field contribution from the cell
may be adequately represented by a dipole at the centre. The near-field due to polarisation in
real materials is more accurately represented by a uniform distribution of dipoles, so the standard approach has been altered to correct for the assumption.

Consider the field in one 2-D square cell, due to dipoles uniformly distributed over an adjacent (non-diagonal) cell, with total polarisation moment $P$ distributed in the $x$ direction as polarisation density $P/x$. The field due to the polarisation is:

$$E_p \approx \int_{x/2}^{3x/2} \frac{P dr}{x r^3} = \frac{16}{9 x^3} P$$

(10)

If $k$ represents a factor to correct for distance between the source and field points, then equating the field in Eqn (6) with the resultant of Eqn (10), and with the convention that the dimensions are referred to cell centres:

$$\frac{P}{(kx)^3} = \frac{16P}{9x^3}$$

(11)

provides a correction $k$ of 0.825. Similarly, the next adjacent cell is located in the interval $(1.5x, 2.5x)$ to give $k=0.95$, and for the interval $(2.5x, 3.5x)$, $k=0.98$. Reformulating Eqn (11) for the slightly more involved diagonal cell integration gives $k=0.908$ for the nearest diagonal cell.

Implementation of the improved method may be simply achieved by testing the distance $r$ from field to source cell, and applying correction $k$ for the appropriate interval in $r$. In this manner, a suitable correction may also be applied to diagonal cells (eg the first diagonal is incorporated with the second rectangular correction) without incurring additional computational burden.

3 Verification

The IE model was verified by comparison with actual propagation times measured by a Hewlett Packard 54121T digitising oscilloscope connected to a 300 mm waveguide in proximity to a water bath. A 1:4 balun, constructed in a manner similar to [7], but using grade S3 ferrite toroids, coupled the 54121T TDR channel to the balanced waveguide which comprised two 6.5 mm diameter brass rods space 60 mm apart.

A time marker comprising a HP5082-3188 PIN diode (on-resistance $R_o = 0.6 \Omega$ at a diode current $I_d =10 \text{ mA}$, and reverse bias capacitance $C_r <1 \text{ pF}$ at 20 V) 300 mm from the open-circuited ends of the waveguide was used in a manner similar to [8]. A bias network (Fig.1), enabled the PIN diode to be switched on for a reference measurement. Contrary to the findings of [8] for the lower sensitivity measurements in soil, the effect of changing the reverse voltage of the diode was apparent to 20 volts, although the forward current had little effect once the diode was forward biased. Consequently, plus 5 V was applied to $V_s$ (Fig.2) to select the reference measurement mode ($I_d \approx 10 \text{ mA}$) and minus 20 V to measure total propagation time, $t_p$. The variable resistance was used to balance, as far as practicable, the sensitivity of each PTL rod.
Fig. 1 Circuit of balun, PIN diode bias and balancing network.

In this instance, the waveform with the diode shorted was subtracted from that with the unshorted diode, cancelling perturbations due to changes in cable impedance and spurious reflections from connectors. The HP54121T was normalised with the reference plane located at the SMA connector between the cable and the balun.

The point of reflection was determined from the intersection of tangents to the maximum slope of the returned edge and the preceding plateau in a manner similar to that used by [9] and [10] (Fig 2).

Fig. 2 Difference waveform and tangents for HP54121T measurements.

Waveform data retrieved from the HP54121T were smoothed and differentiated using 25 point routines [9]. The intersection between the tangents to the maximum positive slope and the immediately preceding stationary point defined the turning points, and the time difference represents propagation time $t_p$ for the 300 mm section of the waveguide.
A rectangular thin walled plastic container 150 by 500 by 80 mm filled with water formed a dielectric body. The waveguide was positioned near the water and used computer readable position sensing with 1 mm precision to record relative positions. The position was defined as the (x, y) distances (mm) between a top edge of the container and the geometric centre of transmission line. A ‘distant’ separation provided a reference reading to correct the small difference between measured and modelled values of \( t_p \) for air (where \( v_p = c \)). Model predictions were calculated using 5 mm cubic cells and a quasi 3-D approach that included the influence of the neighbouring cells in the z direction within the 2-D (xy) matrix [4].

### 4 Results and Discussion

Table 1 shows the comparison between the quasi 3D IE method, and the improved method that compensates for the near-field of all cells within 3 cell dimensions of each source cell. A correction of 6 ps was subtracted from the model predictions to account for the discrepancy between measured and modelled results when the waveguide was positioned far from the water bath (Table 1).

<table>
<thead>
<tr>
<th>Measurements</th>
<th>Quasi-3D Model</th>
<th>Improved Quasi-3D model</th>
</tr>
</thead>
<tbody>
<tr>
<td>Position (mm)</td>
<td>( t_p ) (ns)</td>
<td>Predicted ( t_p ) (ns)</td>
</tr>
<tr>
<td>0, 5</td>
<td>1.077</td>
<td>1.060</td>
</tr>
<tr>
<td>0, 10</td>
<td>1.043</td>
<td>1.037</td>
</tr>
<tr>
<td>0, 20</td>
<td>1.026</td>
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<tr>
<td>0, 30</td>
<td>1.005</td>
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<tr>
<td>75, 5</td>
<td>1.252</td>
<td>1.153</td>
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<tr>
<td>75, 10</td>
<td>1.095</td>
<td>1.087</td>
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<td>1.028</td>
<td>1.038</td>
</tr>
<tr>
<td>75, 30</td>
<td>1.014</td>
<td>1.022</td>
</tr>
<tr>
<td>distant</td>
<td>1.002</td>
<td>1.008</td>
</tr>
</tbody>
</table>

The better agreement between measured data and the improved model demonstrates the improved accuracy that can be obtained with the point matching method, provided field integration errors are accounted for. Although there remains some consistent difference for the asymmetric case (\( x=0 \), Fig 3), this is probably an empirical error since there were asymmetric readings (averaged for Table 1) due to imperfectly balanced signals from the balun (an optimal balun would provide a perfectly balanced signal without bandwidth limitations). Hence, it is considered that further improvement in model accuracy would require validation using more accurate measurement techniques. This would include attention to the high sensitivity of readings to distance between the waveguides and the water, when the distance is small. The improved model results in a better-conditioned field matrix than the
conventional approach since there is more complete integration of the anomalous field. Using the conventional but quasi-3D approach, it was necessary to regularise the field matrix prior to solving, to obtain a stable solution. The improved model provided well-conditioned field matrices, avoiding the need for any regularisation.

Fig.3 Field distribution for asymmetric positioning ($x=0$) of waveguide with respect to water bath.

5 Conclusions

We have described an IE method for determining the electric field distribution in a low loss, inhomogenous dielectric material given a pre-determined impressed field, $E_i$, and for a parallel waveguide, to calculate propagation velocity. Improvement to the conventional IE model arising from more accurate integration of the near field in the IE method provided a more stable solution that agrees more closely with experimental results using water as a dielectric body.

A current direction of our research work is enhanced accuracy and convergence rate for our techniques to non-invasively measure moisture distribution [3]. The technique described here will be used to further enhance model accuracy in that application.

References


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Spatial Time Domain Reflectometry with Rod Probes

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ABSTRACT. Monitoring of transient soil moisture profiles yields valuable insight into hydrological processes in the vadose zone. A recently developed reconstruction algorithm allows deriving water content profiles along extended moisture probes from Time Domain Reflectometry (TDR) signals in time domain. This procedure, which we call Spatial TDR, has originally been designed for and applied to flat band cables buried in soil and connected from both sides. However it is often desirable to use standard TDR rod probes which are easy to handle and less invasive. Therefore a solution is proposed how the Spatial TDR method can be extended to common coated rod probes. Electromagnetic simulations of a 3-rod-probe in lossless and lossy soils are conducted to prove the concept.

Keywords: time domain reflectometry, water content profile, inverse problem, Spatial TDR

1 Introduction

The spatial distribution of soil moisture and its evolution in time is valuable information for many investigations in hydrology, agriculture, and soil science. However the monitoring of a sufficient number of soil moisture profiles can be expensive, laborious, and invasive, especially if the profiles are determined point-wise by a large amount of single probes buried in soil. A recently developed reconstruction algorithm (Schlaeger, 2002) allows computing complete soil water content profiles along single moisture probes from time domain reflectometer (TDR) samples in almost real-time. This method leads to a reduction of probes accompanied by a higher spatial resolution of moisture profiles at the same time. The whole technology of soil moisture profile retrieval is called Spatial TDR. This method will be examined more closely throughout the article. First we introduce the basic concept of the method with emphasis on its algorithmic core and the initial calibration of the applied coated 3-rod-probe. Then we assess the theoretical accuracy of the yielded moisture profiles by means of in depth electromagnetic (EM) field simulations of the TDR process. Finally we realized the virtual experimental setup of the EM simulation in a laboratory experiment in order to compare reconstructed water content profiles with results from oven drying method in a real soil to demonstrate the capability of Spatial TDR under field conditions.

2 Methods

2.1 The Inverse Problem

A TDR instrument located at $x_0$ emits a voltage step pulse $V^m_1(t,x_0)$ via a feeding cable into a waveguide (moisture probe) buried in soil. When the propagating EM wave hits the junction between cable and probe it is generally split. Part is reflected and traveling backwards toward the TDR instrument where it is being sampled, part is transmitted into the waveguide, interacting with the surrounding soil. When the pulse reaches the probe end it is reflected again and the reflected signal is also sampled by the TDR device.
Hence $V_i(m)(t,x_0)$ (input, measured) excites the system probe/soil which reacts with voltage waves whose superposition $V_o(m)(t,x_0)$ (output, measured) is sampled by the TDR instrument. The TDR signal $V_o(m)(t,x_0)$ is sometimes referred to as reflectogram. The elapsed time between first and second main reflection showing up in the reflectogram is the pulse travel time forth and back the moisture probe. This travel time can be transformed into average soil moisture by appropriate calibration functions and/or mixing rules. This is generally what common TDR signal evaluation does.

But the TDR signal contents more information. The reflectogram, especially the part between first and second main reflection at the probe’s beginning and end is a finger print of the dielectric profile along the waveguide, which is mainly ruled by the water content. Unfortunately the moisture distribution cannot be calculated directly from the TDR signal but has to be estimated indirectly. The basic idea of Spatial TDR is to transform the sampled voltage $V_o(m)(t,x_0)$ into the soil moisture profile $\theta(x)$ along the probe by means of inverse modeling. The essence of the approach is to simulate the propagation of the TDR signal along the waveguide in time domain by employing a numerical model (forward problem).

From an electrical point of view buried waveguide and surrounding medium together form a non-uniform transmission-line (Lundstedt, 1995). Under certain conditions the wave propagation in time domain within this system under test (SUT) can be modeled as a partial differential equation (PDE), which in literature is referred to as telegraph, telegrapher’s or transmission-line equation. This simplified model assumes that the relevant properties of the transmission-line can be described by bulk electronic parts like resistors, inductors, and capacitors (see Fig. 1). Among the conditions for this electronic circuit model to hold the most important are: wave modes other than the transversal-electromagnetic (TEM) mode may be neglected, and frequency dependence of transmission-line properties may be neglected. The first condition requires a well-behaving waveguide with little distortion on the signal propagation; the second is only met, if the losses in the SUT are not too large.

Fig. 1 The simplified moisture probe model consisting of bulk electronic parts. Above: coated 3-rod-probe as an example for a moisture probe (TDR waveguide); below: equivalent circuit of the transmission line.

Schlaeger (2002) derived the following wave equation from the circuit model for describing the propagation of a voltage pulse $V(x,t)$ along the buried waveguide:

$$\left(L'(x)C'(x)\frac{\partial^2}{\partial t^2} + L'(x)G'(x)\frac{\partial}{\partial t} + \frac{\partial L'(x)}{\partial x} + \frac{\partial L'(x)}{\partial x} - \frac{\partial^2}{\partial x^2}\right) V(x,t) = 0. \quad (1)$$
Capacitance $C'(x)$ and effective conductance $G'(x)$ are influenced by the soil water content distribution $\theta(x)$ along the waveguide. Inductance $L'(x)$ is a function of the transmission-line only and piecewise constant for coaxial cable and moisture probe. The spatial derivative of $L'$ in (1) describes the change of inductance between coaxial cable and probe. Resistance $R'$ along the waveguide has been neglected. All parameters are given per unit length.

Strictly spoken the equivalent circuit of Fig. 1 is not totally correct, because the conductor $G'$ should be enclosed by two capacitors due to the rod coating. Therefore $G'$ is not the real ionic conductance of the soil but a kind of correcting parameter in the determination of $C'$. According to former results we assume that this simplification does not have a large influence on the results.

Eqn. (1) is solved numerically with appropriate initial and boundary conditions to simulate a TDR measurement $V_0(t,x)\|C',G'$ for given $C'(x)$ and $G'(x)$. The result of the simulation is compared to the TDR measurement. An optimization algorithm is used to modify the electrical parameters $C'(x)$ and $G'(x)$ along the simulated moisture probe until the simulated TDR reflectogram $V_0(t,x)$ matches the measurement $V_0(t,x)$ sufficiently well. The final parameter distributions resulting from the simulation are the best estimate of the electric properties along the real probe in soil.

### 2.2 From Capacitance to Dielectric Permittivity

To derive the volumetric water content profile $\theta(x)$ the dielectric permittivity profile $\varepsilon(x)$ of the soil/water/air mixture has to be extracted from the capacitance profile $C'(x)$ first. The dielectric permittivity expresses the strength of interaction between electric field and material. In our case of a moisture probe with simple geometry (Fig. 2) it is possible to find a simple parametric form of the relationship between $\varepsilon$ and $C'$:

$$1/C'(x) = 1/(\varepsilon(x)C'_1) + 1/C'_2$$

(2)

![Fig. 2: Total Capacitance $C'$ of a 3-rod-probe as a function of the soil’s dielectric permittivity $\varepsilon$. (a) Segment of three parallel rods encompassed by soil; light gray: PVC coating; dark gray: metallic core; (b) Equivalent circuit. $C'_1$, $C'_2$: constant capacitance parameters determined by the probe’s geometry.](image)
2.3 From Dielectric Permittivity to Water Content

The second step performs the transition from dielectric permittivity to water content. An empirical relationship between $\varepsilon$ and $\theta$ often used in TDR applications was found by Topp, et al. (1980). We use a simpler but less general empirical formula, which we derived from laboratory experiments with loamy sand:

$$\theta(\varepsilon) = 30.1 \cdot \varepsilon^{0.31} - 41.1 \, \% \text{vol}$$

(3)

2.4 The 3-Rod-Probe and its Parameters

The 3-rod-probe under investigation has been designed by W. Schädel (Schädel, 2005). Each rod consists of a stainless steel core of 6 mm diameter with a 1 mm thick PVC coating. The rods are 30 mm apart. They are screwed into the probe head which connects them to a 50 $\Omega$ coaxial cable (type RG213).

According to (1) and (2) it is necessary to determine the three parameters $C'_1$, $C'_2$, and $L'$ for the rod probe. This can be done empirically by measuring TDR pulse propagation velocities $v_i = v(\varepsilon_i)$ for two different media with dielectric permittivity $\varepsilon_1$ and $\varepsilon_2$, respectively. The pulse propagation velocity along the coated probe rods is:

$$v(\varepsilon) = 1/\sqrt{L'C'(\varepsilon)}.$$  

(4)

The well-known formula $v = c_0 / \sqrt{\varepsilon}$ is only valid for uncoated rods. The pulse velocity is determined empirically by measuring the time span $\Delta t$ between the first two main reflections in the TDR reflectogram:

$$v = 2l / \Delta t,$$

(5)

with $l$ being the probe length. Combining (4) for the two materials one yields:

$$C'_1 = (\varepsilon_2 - \varepsilon_1) / (\varepsilon_2 \varepsilon_1 (v_1^2 - v_2^2) \cdot L'), \text{ and } C'_2 = (\varepsilon_2 - \varepsilon_1) / ((\varepsilon_2 v_2^2 - \varepsilon_1 v_1^2) \cdot L').$$

(6)

A third independent information is needed to get $L'$. The rod impedance can be used to express $L'$:

$$Z(\varepsilon) = \sqrt{L'C'(\varepsilon)}.$$  

(7)

The impedance $Z(\varepsilon)$ of the rod probe buried in material with dielectric permittivity $\varepsilon$ generally differs from the known impedance $Z_0$ of the connecting cable. The impedance mismatch leads to a partial reflection of the incident excitation pulse at the junction of cable and probe. The amplitude of incident and reflected signal are denoted by $A_I$ and $A_R$, respectively. Then the reflection coefficient yields:

$$r(\varepsilon) = A_R / A_I = (Z(\varepsilon) - Z_0) / (Z(\varepsilon) + Z_0),$$

(8)
which can be determined experimentally from TDR measurements. Substitution of (4) into (7) and (7) into (8) and solving for \( L' \) yields:

\[
L' = \frac{(1 + r(\varepsilon))}{(1 - r(\varepsilon))} \cdot \frac{Z_0}{v(\varepsilon)}.
\]

Eqns. (6) and (9) are sufficient to determine \( C'_1, C'_2, \) and \( L' \) for the coated 3-rod-probe from TDR reflectograms.

2.5 Empirical Relationship between Total Capacitance and Effective Conductance

The wave equation (1) needs two parameter distributions \( C'(x) \) and \( G'(x) \). These parameter distributions could be found simultaneously by inverse modeling, if two independent TDR measurements were available for the same moisture probe. This has been done earlier with special 3-wire flat band cables connectable to the TDR instrument from both sides (Scheuermann et al., 2001). However rod probes in their current design allow measurements from one side only. This problem could be overcome if a relationship between \( C' \) and \( G' \) existed. It is reasonable to assume such a relationship, since both parameters are mainly linked by the soil moisture: higher water content leads to higher dielectric permittivity and higher conductivity. Hakansson (1997) proposes the following empirical formula:

\[
G'(C') = \begin{cases} 
G'_{\infty} \cdot (1 - \exp(-(C'' - C''_0)/C'_d)), & \text{if } C'' \geq C''_0, \\
0, & \text{if } 0 \leq C'' \leq C''_0.
\end{cases}
\]

With this relationship a given capacitance profile can be transformed into an effective conductance profile. Both parameter distributions are inserted into (1), which is solved numerically. It will be shown in later in EM simulations, that the above parametric form fits the findings sufficiently well.

2.6 Electrodynamic Simulation of the TDR Measuring Process

To test the Spatial TDR method together with the 3-rod-probe several TDR reflectograms are simulated with Microwave Studio (MWS), an EM simulation tool based on the full wave solution of Maxwell’s equations. Fig. 3 shows the virtual experimental setup.

Fig. 3: Microwave Studio model of the 3-rod-probe with simplified probe head. Probe length: 600 mm, mutual rod spacing: 30 mm, outer rod diameter: 8 mm, coating thickness: 1 mm.
The moisture probe is embedded in a virtual material with three layers, the properties of which can be modified separately.

In the numerical model the 3-rod-probe is embedded in material, whose dielectric permittivity and ionic conductance can be modified. A voltage step pulse of 1 Volt amplitude and 1 GHz bandwidth is fed into the probe. The pulse propagates along the probe and interacts with the predefined material. The voltage at the input is recorded over time, which yields the TDR reflectogram.

The simulated TDR reflectograms are used for three purposes: 1. determination of the probe parameters according to (6) and (9), 2. determination of the empirical $C'\cdot G'$-relationship (10), and 3. generation of test reflectograms to assess the quality of the Spatial TDR algorithm.

### 3 Results

#### 3.1 Determination of Probe Parameters

To simulate the TDR procedure with the numerical model (Fig. 3) an excitation pulse $V_{I}^{(s)}(t)$ has to be defined first. A step pulse with a frequency bandwidth of 1 GHz and amplitude 1 (in arbitrary units) is shown in Fig. 4, dotted line. To determine the inductance of the probe a measurement in air ($\varepsilon=1$) is simulated. The reflected portion resulting from the simulation run is denoted by $V_{R}^{(s)}(t)$ in Fig. 4 (The TDR instrument’s recording $V_{O}^{(s)}(t)$ is a superposition of incident and reflected pulse, i.e. $V_{O}^{(s)}(t)=V_{I}^{(s)}(t)+V_{R}^{(s)}(t)$). The reflection coefficient (8) is found by scaling $V_{I}^{(s)}(t)$ until it matches the first rising edge and the following plateau of $V_{R}^{(s)}(t)$. According to Fig. 4 the reflection coefficient reads: $r(\varepsilon=1)=0.535$.

![Fig. 4: TDR simulation of the 3-rod-probe in air (\varepsilon=1). $V_{I}^{(s)}(t)$: predefined excitation pulse, $V_{R}^{(s)}(t)$: reflected signal, $r\cdot V_{I}^{(s)}(t)$: excitation pulse scaled by reflection coefficient $r=0.535$.](image)

The pulse travel time between first and second reflection (steep edges) of $V_{R}^{(s)}(t)$ was determined by the tangent fitting method (Heimovaara and Bouten, 1990): $\Delta t(\varepsilon=1)=4.364$ ns. With an effective probe length of $l=0.605$ m (the field reaches a few millimeters beyond the rod’s end into the soil) the pulse propagation velocity (5) yields: $v(\varepsilon=1)=2.773\cdot10^8$ m/s= $0.925\cdot c_0$, with the speed of light $c_0=2.997\cdot10^8$ m/s.
The input impedance of the coaxial probe feeding (left boundary condition in Fig. 3) is $Z_0=50 \, \Omega$. Substitution into (9) yields the impedance: $L'=595 \, \text{nH/m}$.

In order to derive $C'_1$ and $C'_2$ from (6) the pulse velocity in another material must be determined. We chose a soil with 13 %vol volumetric water content (Tab. 1).

![Fig. 5: Two reflectograms to determine the pulse propagation velocity in two different media. Material 1: air, $\varepsilon_1=1, \sigma_1=0 \, \text{mS/m}, \Delta t_1=4.364 \, \text{ns}$; material 2: soil ($\theta=13 \, \text{%vol}$), $\varepsilon_2=6.8, \sigma_2=23 \, \text{mS/m}, \Delta t_2=10.11 \, \text{ns}$. Substitution into (6) yields: $C'_1=22.9 \, \text{pF/m}$ and $C'_2=486 \, \text{pF/m}$. With these parameters the capacitance model (2) allows to calculate the dielectric permittivity profile from any capacitance profile resulting from the Spatial TDR algorithm.

### 3.2 Empirical Relationship between Capacitance and Effective Conductance

To check and parameterize the proposed $C'\cdot G'$-relationship (10) the MWS model (Fig. 3) was fed with homogeneous soil of different volumetric water content. The water content influences the material’s dielectric permittivity $\varepsilon$ as well as its ionic conductivity $\sigma$. For each material realization a TDR signal was simulated with MWS. These reflectograms were used in the reconstruction algorithm under the constraint of constant $C'$- and $G'$-distribution.

Two examples of reflectograms and their corresponding reconstruction are displayed in Fig. 6, left. Fig. 6, right, shows the resulting $C'\cdot G'$-pairs and the fitted model (10) with parameters: $C'_{\theta}=65.24 \, \text{pF/m}, C'_{\sigma}=20.75 \, \text{pF/m}, G'_{\sigma}=27.83 \, \text{mS/m}$. The proposed model is in very good accordance with the results of the reflectogram simulations.
3.3 Test of the Spatial TDR Method

To assess the quality of the algorithm which determines the water content profile from a TDR reflectogram by inverse parameter estimation, the MWS model (Fig. 3) is fed with three soil layers of different moisture. Tab. 1 shows the applied soil parameters.

Table 1: Dielectric permittivity $\varepsilon$ and ionic conductivity $\sigma$ of a soil for different volumetric water contents $\theta$. The parameters were derived from loamy sand in laboratory experiments. For the sake of simplicity the moisture states are named dry, wet, and moist, respectively.

<table>
<thead>
<tr>
<th>Moisture state</th>
<th>$\theta$ %vol</th>
<th>$\varepsilon$ -</th>
<th>$\sigma$ mS/m</th>
</tr>
</thead>
<tbody>
<tr>
<td>‘dry’</td>
<td>0.5</td>
<td>2.9</td>
<td>0</td>
</tr>
<tr>
<td>‘moist’</td>
<td>8</td>
<td>4.9</td>
<td>14</td>
</tr>
<tr>
<td>‘wet’</td>
<td>13</td>
<td>6.8</td>
<td>23</td>
</tr>
</tbody>
</table>

Several different soil moisture sequences were realized in the MWS model. Two examples with the predefined material combinations dry/moist/wet and wet/moist/dry are shown in Fig. 7. With each sequence two cases were realized: one with and the other without consideration of ionic conductivity (lossy and lossless case). Fig. 8 displays the corresponding TDR reflectograms simulated with MWS.

Each simulated reflectogram together with the excitation pulse was fed into the Spatial TDR algorithm and a reconstruction process was conducted to retrieve the soil moisture profiles, which should match the predefined as close as possible. Fig. 8 shows the reflectograms approximated by the algorithm. The resulting water content profiles are given in Fig. 7.
Fig. 7 Moisture profiles, predefined in the MWS model, and reconstructed by means of the Spatial TDR algorithm. Material sequences dry/moist/wet (left) and wet/moist/dry (right). For each material combination the lossless and lossy case has been considered.

Fig. 8 TDR reflectograms simulated by MWS and the corresponding signal approximations resulting from the reconstruction algorithm. Material combination dry/moist/wet (left) and wet/moist/dry (right). Energy losses due to ionic conductance lead to a strong falling trend of the TDR signals.

With the material sequence dry/moist/wet (Fig. 7, left) the reconstructed soil moisture profiles are close to the preset. In the lossless case the deviation between the reconstruction and the target is less than 1 % vol. When the soil’s ionic conductivity is considered the error is a little larger. The negative volumetric water content in the first layer with dry soil is due to the low dielectric permittivity slightly exceeding the domain of the moisture calibration curve \( \theta(\varepsilon) \).

When the predefined profile is reversed, i.e. starts with a wet layer, the reconstruction results oscillate stronger around the setpoint values. The lossy case again produces larger deviations than the lossless. It is remarkable that the comparatively low quality of the former cannot be explained by an insufficient TDR reflectogram reconstruction. In this particular case the reconstructed TDR signal matches the target reflectogram very well (Fig. 8, right, dashed).
The overall accuracy of Spatial TDR with coated rod probe is sufficient for many applications in soil science. Becker (2004) showed in a lysimeter experiment with 1 m³ loamy sand that the method is capable of tracking transient soil moisture profiles under irrigation with high spatial and temporal resolution.

In another laboratory experiment he compared profile reconstruction and gravimetric water content from oven drying. Even in this field application accompanied with much higher calibration effort and uncertainty the average error was rarely above 2 %vol.

4 Conclusion

The Spatial TDR method derives water content profiles from TDR reflectograms along single probes connected to the TDR instrument from both sides. The method is also applicable to coated rod probes connected from one side only. In this case a special relation between capacitance and effective conductance ($C′-G′$-relation) of the probe/soil system must be utilized.

Based on TDR reflectograms simulated with an electromagnetic field solver we explained how to calibrate the moisture probe and how to parameterize the $C′-G′$-relationship. These procedures can also be used with real measurement data.

We used the same simulation framework to assess the quality of the reconstructed soil moisture profiles. The deviation with respect to a predefined water content profile is mostly below 1 %vol and rarely above 2 %vol. Spatial TDR is capable of monitoring transient soil moisture profiles with high spatial and temporal resolution which has already been supported by irrigation experiments.

References


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Session 4

Electromagnetic Wave Propagation in Lossy Dielectrics Related to Sensors
Chairmen: M. Kummer, A.S. Sovlukov

1 Electromagnetic Wave Propagation in Polarizable Wet Media
   H. Bolvin and A. Chambarel; Avignon, France

2 Radiofrequency Measurement of a Lossy Dielectric Liquid Content in Water
   V. Y. Fateev, A. S. Sovlukov; Moscow, Russia

3 Measurement of Moisture Content in a Highly Electrical Lossy Material using Time Domain Reflectometry
   St. Schlaeger; A. Scheuermann; R. Becker, A. Bieberstein; Karlsruhe, Germany

4 Some Topics of Measurement of Complex Permittivity for Lossy Dielectric Materials with Wide-Range of tanδ
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5 Material Parameter Measurement of Soils and Liquids with a Waveguide Setup
   M. Pauli, T. Kayser and W. Wiesbeck; Karlsruhe, Germany
Electromagnetic Wave Propagation in Polarizable Wet Media

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ABSTRACT. The Time Domain Reflectometry -TDR- probe is a technique applied to soil moisture measurement. This probe is a wave guide composed of two or three parallel metallic rods stuck in the ground, between which one applies a step of electric field. The main objective of this paper is to contribute to the interpretation of the signals obtained at the entrance of the probe. The theoretical approach is formulated using 2D Maxwell equations and we propose to take into account the transient polarization of the medium. We have simulated the wave guide in this medium thanks to the Finite Element Method.

Keywords: Time Domain Reflectometry, Maxwell’s equations, electric polarization, Finite Element method.

1 Introduction

Water resource management for plants is becoming an increasingly acute problem. This is associated with pollution phenomena usually caused by fertilizers. It is therefore essential to have a precise idea as to the soil’s moisture content, both at surface and underground levels. A number of current research programs are investigating technologies usually based on electrical measurements. Resistive [1] and capacitive [2] probes can be offered as examples. The latest generation of instruments [3] is based on Time Domain Reflectometry (TDR). The TDR probe is a wave guide composed of two or three parallel metallic rods stuck in the ground. At initial time, a step of electric field is applied between the electrodes and the intensity is measured at the entrance of the wave guide [4].

We present a model of electromagnetic wave propagation in a wave guide containing a complex medium: in practice, the complex medium represents the soil.

This work is a contribution to the interpretation of the TDR (Time Domain Reflectometry) probe signals. The electric permittivity is frequency-dependent: this is not appropriate for the impulse mode of TDR probe. In relation with the characteristic time of the TDR signal, it is not possible to neglect the transient polarization. In fact we must take into account the inertia of elementary charges. For this reason we built a model based on electric polarization $\vec{P}$ of the medium.

The theoretical approach is formulated using 2D Maxwell equations. They are solved in the time domain using the Finite Element Method [5]. Solutions are performed numerically using a semi-implicit method. Moreover, for this study, we developed efficient C++ Object-Oriented Programming for the Finite Element code called FAFEMO (Fast Adaptive Finite Element Modular Object) [6]. This constitutes a general Finite Element solver for the Maxwell equations.
2 General Presentation of the Model

2.1 Geometrical Model of the 2D TDR Probe

The 2D wave guide (Fig.1) is represented by two parallel plate electrodes and the electric conductivity of these is infinite.

In our example we study the transverse magnetic-mode. The electromagnetic field is:

$$\begin{align*}
\vec{H} &= \begin{bmatrix} H_x \\ 0 \\ 0 \end{bmatrix} \\
\text{and} \\
\vec{E} &= \begin{bmatrix} 0 \\ E_y \\ E_z \end{bmatrix}
\end{align*}$$

Fig. 1 Geometrical model

2.2 Maxwell’s Equations

Considering the usual notations, the Maxwell’s equations are formulated as follows [7]:

$$\overrightarrow{\text{curl}} \, \vec{E} = - [\mu] \frac{d\vec{H}}{dt}$$  \hspace{1cm} (1)

$$\overrightarrow{\text{curl}} \, \vec{H} = \frac{\partial \vec{D}}{\partial t}$$  \hspace{1cm} (2)

$$\text{div} \, \vec{D} = \rho$$  \hspace{1cm} (3)

$$\text{div} \, ([\mu] \, \vec{H}) = 0$$  \hspace{1cm} (4)
2.3 Polarization Model

We present a first level of polarization model in the case of a dielectric medium. Electric polarization $\vec{P}$ of the medium is defined by the usual relation [7]:

$$\vec{D} = \varepsilon_0 \vec{E} + \vec{P} \quad (5)$$

The Maxwell-Ampere equation (2) becomes:

$$\nabla \times \vec{H} = \frac{\partial \vec{D}}{\partial t} = \varepsilon_0 \frac{\partial \vec{E}}{\partial t} + \frac{\partial \vec{P}}{\partial t} \quad (6)$$

by introducing a conduction current:

$$\vec{J}_c = \frac{\partial \vec{P}}{\partial t} = N \vec{V} \quad (7)$$

where $N$ is the volumic electric charge density in the medium.

We have used a classical model in which an electric charge is fastened elastically to the nucleus, obeying the following non dimensional equations:

$$\frac{d\vec{V}}{dt} + \frac{\tau}{\tau_0} \vec{V} + \omega^2 \tau_0 \vec{X} = \vec{E} \quad \text{with} \quad \frac{d\vec{X}}{dt} = \vec{V} \quad (8)$$

Dielectric loss is defined by a macroscopic viscous model [7].

In Eq.(8), $\frac{\tau}{\tau_0}$ represents a loss coefficient; $\frac{\tau_0}{\tau}$ is the non dimensional relaxation time of the water molecules ($\tau = \frac{L}{c}$ is the characteristic time of the TDR probe).

$\omega^2 \tau^2$ is a phase coefficient: $\omega$ is the angular frequency for maximum absorption by water molecules ($\sim 3$ GHz).

The non-dimensional Maxwell’s equations become respectively:

$$\nabla \times \vec{E} = -\frac{\partial \vec{H}}{\partial t} \quad (9)$$

$$\nabla \times \vec{H} = \frac{\partial \vec{E}}{\partial t} + N \vec{V} \quad (10)$$

$$\text{div}(\vec{E} + \vec{P}) = 0 \quad (11)$$

$$\text{div}(\vec{H}) = 0 \quad (12)$$
2.4 Boundary Conditions

The boundary conditions on the electric conductor - i.e. electrodes - can be written :

\[ \vec{E} \times \vec{n} = 0 \]  

(13)

At initial time, we apply a constant electric potential difference between the electrodes. Far from the electric line, the other boundary conditions must be defined as :

\[ \vec{E} \times \vec{n} = 0 \]

In all cases of TDR procedure, the electromagnetic wave never reaches that boundary because we only study the first reflection in the wave guide for the detection of electric properties. On the electrodes, we can calculate line current \( I \) by the Ampere theorem with the superficial current’s density [7].

3 Finite Element Formulation

The Galerkin Finite Element method is applied to Maxwell’s equations (9) and (10), and to equation (8). We use vector \( \{ \delta \vec{H}, \delta \vec{E}, \delta \vec{V}, \delta \vec{X} \} \) and the weighted residual method can be written :

\[ \int (\delta \vec{H} \cdot \frac{\partial \vec{H}}{\partial t}) d\Omega = -\int (\delta \vec{H} \cdot \text{curl} \vec{E}) d\Omega \]  

(14)

\[ \int (\delta \vec{E} \cdot \frac{\partial \vec{E}}{\partial t}) d\Omega = \int (\delta \vec{E} \cdot \text{curl} \vec{H}) d\Omega - \int (\delta \vec{E} \cdot N \vec{V}) d\Omega \]  

(15)

\[ \int (\delta \vec{V} \cdot \frac{\partial \vec{V}}{\partial t}) d\Omega = \int (\delta \vec{V} \cdot \vec{E}) d\Omega - \int (\frac{\tau}{\tau_0} \vec{V} \cdot \vec{E}) d\Omega - \int (\delta \vec{V} \cdot \omega^2 \vec{X}) d\Omega \]  

(16)

\[ \int (\delta \vec{X} \cdot \frac{\partial \vec{X}}{\partial t}) d\Omega = \int (\delta \vec{X} \cdot \vec{V}) d\Omega \]  

(17)

After discretization of domain \( \Omega \) with \( ne \) elements \( \Omega_e \) and using Lagrange’s polynomial base the general formulation of the differential system is :

\[ \sum_{ne} \{ \delta u_e \} \cdot \left( \left[ m_e \right] \frac{du_e}{dt} \right) + \left[ k_e \right] \{ u_e \} - \{ f_e \} = 0 \]

(18)

where \( u_e = (H_e, E_e, V_e, X_e) \)

\[ \left[ k_e \right] \] is the electric stiffness matrix and \( \{ f_e \} \) the elementary electric load.

In this formulation (18), the physical parameters appear as elementary properties. Their values are distributed to each element. After a classical assembling operation, the differential system is as follows [6]:
\[
\frac{d}{dt} \{U\} = \{F\} - [K] \{U\}
\]  

(19)

or

\[
\frac{d}{dt} \{U\} = [M]^i \{\Psi(U,t)\} \quad \text{where} \quad \{\Psi(U,t)\} = \{F\} - [K] \{U\}
\]  

(20)

4 Numerical Resolution

4.1 Semi-Implicit Method

The corresponding \( k \) order algorithm is as follows:

\[
t_n = 0 \\
\text{while} \quad \left( t_n \leq t_{\text{max}} \right) \\
\quad \{\Delta U_n^i\} = \Delta t \beta \{M\}_{n+\theta} \cdot \Psi_{n+\theta} + \sum_{i=1}^5 \alpha_i \{M\}_{n+1-i} \cdot \Psi_{n+1-i} \\
\quad \left\{U_{n+1}\right\} = \left\{U_n\right\} + \left\{\Delta U_n\right\} \\
\quad t_{n+1} = t_n + \Delta t_n \\
\text{end while}
\]

where \( \theta \) is a upward time-parameter.

4.2 The Code and the Automatic Multigrid System (AMS)

We use efficient C++ Object-Oriented Programming for the Finite Element code called FAFEMO (Fast Adaptive Finite Element Modular Object) developed by A. Chambarel [6]. In this context, our numerical calculus uses a technique called the AMS. For all iterative or step-by-step processes, an expert system chooses the unknown degrees of freedom for the update of the solution, and the size of the unknown vector is optimized.

4.3 Space Discretization

The 2D model is presented in Fig. 1. We discretized the domain with triangular linear elements. They are described in literature references [8]. We refined the meshing near the electrodes (Fig. 2), so we have 13,985 triangular elements and 7,300 nodes. 51,100 differential equations are generated by the above Finite Element process. For numerical quadrature points, the nodes of the elements are chosen [6]. So mass matrix \([M]\) (Eq.19) is diagonal. Then, inversion is an easy procedure. Under these conditions we have tested a semi-implicit method. We have used a \textit{matrix-free technique} : the mass matrix and the stiffness matrix are never built ; only the elementary matrices (Eq.18) are calculated.
4.4 Numerical Results

The harmonic mode allows the determination of the coefficients of differential equation (8) because the frequency dependent laws are known for water (Table 1).

Table 1 Coefficients of differential equation (8)

<table>
<thead>
<tr>
<th>N</th>
<th>$\omega^2 \cdot \tau^2$</th>
<th>$\tau / \tau_0$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$10^6 - 3 \cdot 10^6$</td>
<td>$1.10^5$</td>
<td>0 - 2500</td>
</tr>
</tbody>
</table>

Usable signal $I = f(t)$ is presented on Fig. 3 for an electric charge density $N = 1.10^6$, and for different values of loss coefficient $\tau / \tau_0$: we notice a transient polarization connected to soil moisture which strongly decreases with the loss coefficient.
In Fig. 3, we also obtained the equivalent relative permittivity of the medium \((\varepsilon_r \sim 12)\) using transverse magnetic mode equations [7]. In practice the wave is close to a plane wave of electromagnetic field in the guide. The approximate formulation for an infinite guide (with a 2.a width) gives the following expression:

\[
\left| \frac{H_z}{E_y} \right|_{n,\omega} \approx \sqrt{\frac{\varepsilon}{\mu}} \sqrt{1 - \frac{1}{\varepsilon - \lambda^2}} \approx \sqrt{\frac{\varepsilon}{\mu}}.
\]

Fig. 4 presents the relative permittivity of the wet medium according to electric charge density \(N\).

Electric charge density \(N\) allows the determination of a relative permittivity of the medium at low frequency. So we can accede to the soil moisture with Topp formula [9].

On Fig. 5 we can see the propagation of the magnetic field in the wave guide, for growing values of electric charge density \(N\) for the same non-dimensional time. We notice a decrease in the celerity of the electromagnetic wave in the guide, if \(N\) increases. This decrease of the celerity is coherent with an apparent value of permittivity.

Fig. 5 Magnetic field in the wave guide for growing values of electric charge density \(N\) at \(t=4\).
5 Conclusion

We present a general Finite Element formulation for 2D Maxwell’s equations in the case of a propagation phenomenon in a TDR probe stuck in a complex medium as a soil. The aim is to contribute to the interpretation of the probe signals: we take into account the electric polarization of the dielectric medium. So we present a first level of polarization model and we validate the numerical method. This model can be adapted to other types of polarization or with conduction, according to soil characteristics.

References


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Radiofrequency Measurement of a Lossy Dielectric Liquid Content in Water

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ABSTRACT. Radiofrequency resonant TEM transmission line-based sensors for determination of a lossy dielectric liquid content in water are proposed and studied. Among them are modified two-wire line sensor, coaxial line sensor, and also coaxial line sensor with U-like inner conductor. It is shown that the latter sensor is preferable for monitoring considered liquids. Specific peculiarities of these sensors are described. Radiofrequency measuring devices containing these sensors are considered. Results of experiments with the sensors on some water-contained lossy dielectric liquids are presented. They proved effectiveness of suggested approach for measuring concentration of various two-component lossy dielectric liquids.

Keywords: radiofrequency, lossy dielectric liquid, water, concentration

1 Introduction

Concentration of various mixtures that are two-component liquids, in particular of water-contained mixtures is the parameter needed to be determined with high accuracy in many branches of industries (food, chemical, etc.); for ecological purposes there is a need also to determine presence and content of impurities in water. Radiofrequency (RF) and microwave measurement methods and sensors are used for solution of many such problems [1-4]. Various RF and microwave measuring cells for studies of dielectric liquids are known [1-9]. However some of practically important problems are characterized by the need to measure accurately concentration of two-component liquids with one or both components being lossy dielectrics, for instance of alcohol-water mixtures (solutions). In addition, there is also a need to measure very high degree or very low degree of a liquid content in water (content of one of components in a water-contained mixture is less than 1%). This problem is actual and not properly solved up to now. Solution of such measurement problem is considered in this paper using RF measurement techniques.

2 Radiofrequency Sensors

Design principles of RF sensors are suggested for highly accurate concentration measurement of water-contained lossy dielectric liquids. Several schemes of RF concentration measuring devices were designed and models of them were manufactured. Experiments with these designed devices were done for some water-contained solutions. These experiments with RF sensors showed that RF resonator sensors with dielectrically coated conductors are efficient for monitoring of water-contained liquids with high dielectric losses.

2.1 RF Resonant Two-Wire Line Sensor

Radiofrequency resonant concentration sensor may be done as the section of two-wire TEM transmission line (Fig. 1). It contains the probe consisting of two dielectrically coated metal plates surrounded by metal cylinder (screen), screened two-wire TEM-line switched at its
upper end on the winding of inductance coil. This inductance coil has also two coupling loops. They serve for inductive coupling of the resonator with network analyzer via connection cables providing excitation of electromagnetic oscillations in the TEM-line section and pickup of informative signals. The measured parameters are resonant frequency \( f \) and quality factor \( Q \) of the RF resonator. The coupling loops should have minimal mutual coupling, therefore they are located at the opposite ends of the winding. The probe is immersed into a monitored liquid (lossy dielectric). So measured resonant frequency of RF resonator depends on permittivity/loss tangent of this liquid.

![Fig. 1 RF resonant measuring device with two-wire probe](image)

RF probe contains two dielectrically coated metal plates fixed at the dielectric support. These metal plates are inside the metal cylinder (screen) with through holes providing free access of a monitored liquid into the space between the plates.

Experimental data related monitoring of alcohol-water mixture and to this sensor are shown in Fig. 2. It follows that relative change of resonant frequency \( f \) is 4% due to the change of measured concentration \( C \) (content of alcohol in water) in the range 9,2 \( \div \) 15,1 %; measurement inaccuracy if not more than 1%. Quality factor \( Q \) is rather high in the whole measurement range (much more than needed minimal value \( Q = 10 \) for providing resonant measurements [1]).

2.2 RF Resonant Coaxial Line Sensor

RF resonant sensor represents the section of coaxial transmission line with the probe at its lower end (Fig. 3). This probe is coaxial capacitor. Its inner rod has hermetic dielectric coating. This coating is needed for the quality factor increase of such RF resonator with the probe containing a monitored lossy dielectric liquid. Outer cylindrical conductor is electrically connected with outer conductor of the coaxial transmission line. Upper end of the
line section is loaded by winding of inductance coil. Other components of this scheme (coupling loops, network analyzer) are similar to the appropriate components in Fig. 1.

![Graph showing the dependence of resonant frequency and quality factor on concentration for RF resonant two-wire sensor.](image)

**Fig. 2** Dependence of resonant frequency and quality factor on concentration for RF resonant two-wire sensor

Sensor construction with the following parameters was used in experiments: length of coaxial TEM-line section 100 mm; diameters of inner (rod) and outer (screen) conductors 1 mm and 10 mm accordingly; probe length 50 mm; diameters of the probe’s inner and outer conductors 12 mm and 23 mm accordingly; thickness of dielectric (Teflon) coating on the probe’s inner conductor 1 mm.

![RF resonant measuring device with coaxial probe.](image)

**Fig. 3** RF resonant measuring device with coaxial probe

Received with this RF sensor experimental data for alcohol-water mixture with concentration $C$ (content of alcohol in water) change within the range 9.2 ÷ 15.1% are shown in Fig. 4. Relative change of resonance frequency $f$ versus concentration $C$ is 4%; measurement...
inaccuracy is not more than 1%. Quality factor $Q$ is rather high in the whole measurement range.

![Graph](image)

Fig. 4 Dependence of resonant frequency and quality factor on concentration for RF resonant coaxial sensor

2.3 Optimization of RF Resonant Sensor Construction

The above-considered construction of the coaxial sensor can be improved in order to meet technological needs under sensors’ production: to use standard isolated wires as dielectrically coated inner conductor of coaxial sensor. However it is not always possible because in this case special hermetization of wire ends is needed in order to exclude electric contact of this wire and a monitored liquid being a lossy dielectric. In practice such operation is the additional and non-desirable task under production of sensors, moreover if it is serial.

A TEM-line section with U-like conductors, in particular coaxial line with dielectrically coated U-like inner conductor (Fig. 5) is preferable to use as RF sensor taking into account manufacturing/exploitation factors. This conductor is surrounded by metal tube (screen). Measuring volume is hermetically fixed by dielectric support (slab) at the upper end of the transmission line section. This section is connected to the electronic unit providing excitation of electromagnetic oscillations in such TEM-line resonator and determination of its basic resonant frequency $f$ (informative parameter). Electronic unit can contain self-oscillator where the TEM-line resonator determines its frequency that is informative parameter in this case and is registered being related to measured concentration.

A standard isolated wire may be used as U-like inner conductor. In this case there is no need for doing hermetization of the ends of this coated conductor. Notice that for optimization of sensor characteristics it is often needed to choose thickness of this coating in the dependence on parameters of lossy dielectric liquids. However choice procedure of wire coating is rather difficult operation because assortment of industrially manufactured wires and dielectric tubes is limited. In addition there is a need to do manipulations with a sensor during experiments (change of holes’ diameters in the dielectric support where coated wires are come through; change of these wires).

Use of U-like inner conductor in the sensor gives ability to avoid these problems. The above said optimization can be provided electrically by switching on some reactive impedance $Z$ (lumped capacitance, inductance) between one of U-like isolated wire ends and metal tube...
(Fig. 5). In particular such end load may be equal zero ($Z = 0$) or infinity ($Z = \infty$) under short-circuiting or switching off appropriate end of the inner conductor and metal tube, accordingly.

End load $Z$ may be changed rather easily in experiments with minimal difficulties and time. Sometimes it may be of practically interest to excite electromagnetic oscillations in the sensor as in the section of screened symmetrical two-wire line; in this case it is shortcircuited at the end that is opposite to the end where excitation of oscillations takes place. Such line section is quarter-wavelength resonator. Metal tube (screen) is connected to the metal body of electronic unit and is not connected electrically to the considered section of two-wire line. This way of connection can be used under exploitation of the sensor in environment conditions characterized by the presence of disturbing strong electromagnetic fields (working welding apparatus, equipment for RF heating, etc.). Such disturbances influence on both conductors of the symmetrical two-wire line; therefore they are compensated in electronic unit and don’t influence on its operation. According to the other way it is also possible to connect together both ends of inner U-like conductor and also electronic unit; metal tube is here as outer conductor of such coaxial line. In this case the coaxial line section is: half-wavelength resonator under excitation of electromagnetic oscillations through lumped electric capacitance of small value ($\sim 1 – 10$ pF); quarter-wavelength resonator if wounding (inductance $\sim 1 – 10$ $\mu$H) is connected to the end where oscillations are excited. Higher value of resonator quality factor is advantage of such way of connection in comparison with the above-considered ones in cases where RF sensor size is rather significant (under monitoring of averaged liquid concentration values in large-sized tanks – cisterns, etc.). It can be explained by twice shortening of inner conductor electrical length resulting in the appropriate decrease of ohmic losses in the line conductors (in the other connection ways of line conductors such losses can decrease quality factor of the resonator up to an inadmissibly low value).

3 RF Concentration Measuring Device

Differential scheme of RF concentration measuring device is used in measurements (Fig. 6). It has two channels with measuring and reference sensors: in one of them RF sensor is filled in by a studied two-component liquid, in the second (reference channel) similar RF sensor is filled in by only one of liquid components of the studied mixture. Each RF sensor is switched on into frequency-determining circuit of appropriate self-oscillator contained in measuring head of each sensor. Electronic unit contains other components of the scheme in Fig. 6. In
each channel high frequency output signal of self-oscillator comes via connection cable to the appropriate frequency divider (its output frequency is ~ 10-100 kHz) in electronic unit and then to one of the ports of frequency mixer. Difference of frequencies from its output is fixed by digital or analog (using digital-analog transducer) register (frequency difference meter); its data are related to measured concentration values.

Photo of RF concentration measuring device is shown in Fig. 7. This device (its scheme is in Fig. 6) was used in experiments for measurement of a lossy dielectric liquid content in water. Two glasses are filled in by a monitored liquid (left glass) and with water (right glass). Similar RF sensors are placed in these glasses. They are connected with electronic unit (right). As both measuring and reference channels are at the same environment then influence of possible disturbances results in similar frequency changes of self-oscillators in the differential scheme of the device. Therefore concentration measurement accuracy is not dependent on these disturbing factors.

Experiments with different lossy dielectric liquids confirm effectiveness of such device for concentration measurement. So, TEM-line section had the following parameters in experiments: length 100 mm; inner diameter of metal tube from stainless steel 20 mm; diameter of inner U-like copper conductor with coating 2 mm, without coating 1,2 mm;
Polyvinyl chloride was used as coating material. Both parts of U-like conductor were symmetrically located in the metal cylinder relative to its longitudinal axis; space between these parts was 7 mm. One of the line section ends was connected to electronic unit while its other end was open ($Z = \infty$). Resonant frequency of empty sensor was 13 MHz. In experiments were studied water-alcohol mixture (solution) and smoking fluid used in food industry. Components of these liquids are lossy dielectrics. When the sensor was immersed into water then resonant frequency became 9 MHz. So, it was studied dependence of resonant frequency $f$ on concentration $C$ of carbon compounds (contained in smoking fluid) in water within the range $0 \div 2\%$; appropriate change (decrease) of $f$ was about 3% (~270 kHz) relative to its value when only water ($C = 0$) was present. Such experiments confirm ability, first, of highly accurate concentration measurement of two-component lossy dielectric liquids and, second, of low concentration measurement for such liquids.

4 Conclusions

The received results can be applied for highly accurate monitoring of various binary mixtures (solutions) with any dielectric losses of their components. Suggested RF devices are especially attractive if their serial production is planned. Based on the ideas considered it is also possible to design and product RF sensors for on-line monitoring of lossy dielectric liquids flowing in pipelines.

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Measurement of Moisture Content in a Highly Electrical Lossy Material using Time Domain Reflectometry

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ABSTRACT. Water content profile monitoring is of great importance for investigating transient hydraulic processes in porous media. Recently developed algorithms make it possible to obtain moisture profiles along extended flat band cables with Time Domain Reflectometry (TDR) measurements from both sides. However with these transmission lines the precise travel time measurement is complicated especially in highly electrically conductive media due to the imperfect TDR pulse reflection at the end of the transmission line and the strong signal dissipation. In order to improve the TDR pulse reflection switches were installed at each end of the transmission line. A full-scale dike model was equipped with flat band cables to test this new design in lossy material. The set-up of the measuring system for the dike model and first measuring results are presented. The different materials of the dike model are characterised electrically and calibration functions are shown.

Keywords: spatial TDR, transmission line design, calibration, lossy material

1 Introduction

With conventional TDR systems normally the mean volumetric water content is measured with a simple TDR step pulse and a total reflection at the end of the sensor using two or three wire transmission lines which are furthermore mostly limited in length. For longer transmission lines a new insulated flat band cable has been developed [1, 2]. Especially when water content distributions along extended transmission lines are to be measured taking into account the conductive properties of the soil, measurements are necessary on both ends of the flat band cables. A three step algorithm has been developed recently for reconstructing the soil moisture profile from this kind of measurements [3]. This new TDR system consisting of two-sided step pulse measurements with spatial analysis has already proved its effectiveness in different applications [4, 5] and is introduced as spatial TDR.

This type of measurement of the spatially distributed water content is straightforward, especially in soils like sand, which present no problem from an electrical point of view. However in highly electrically conductive soils the travel time analysis is much more complicated due to imperfect TDR pulse reflection at the end of the transmission line as a result of strong signal dissipation along the sensor. In order to improve the TDR pulse reflection switches were installed at each end of the transmission line.

The following contribution presents a modified set-up of two-sided measurements using flat band cables in order to ease travel time analysis of TDR traces. In order to test this new measurement set-up in electrical lossy material a full-scale dike model was equipped. The different materials of the dike-model are characterised electrically and first measurements are presented.
2 Spatial TDR with Modified Transmission Lines

The measurement set-up of the modified transmission line using flat band cables with switches on both sides is shown in Fig. 1. With a multiplexer for high-frequency signals it is possible to change between both sides of the transmission line. The switches are controlled separately with a relay device using 12 VDC. Both the multiplexer and the relay device are controlled via a conventional PC.

![Diagram of TDR measurement set-up](image)

Fig. 1 Operating mode of the TDR measurement set-up with switching at the transmission line ends (schematic description)

With this simple modification of the measurement set-up it is possible to measure TDR pulses under different electrical boundary conditions at the end of the sensor so that the travel time can be determined with simple signal processing methods. Another reason for the application of switches is that it is also possible to measure TDR step pulses as input-signals necessary for the reconstruction algorithm to obtain moisture profiles. Fig. 2 shows TDR traces on a 2 m long sensor measured from both sides in electrical lossy material under different electrical boundary conditions. In order to calculate the spatially distributed water content along a transmission line the following switch combinations are necessary for both sides of the sensor:

- **Switch I open:** Input-signal as TDR pulse launched into the sensor.
- **Switch I closed, switch II open:** Output-signal 1 with a total reflection at the end of the sensor.
- **Switch I closed, switch II closed:** Output-signal 2 with a reflection at the end of the sensor for a 50 Ω boundary condition.
Especially from the measurement above on output-signal 2 it is easy to see that a calculation of the travel time could be quite complicated using conventional methods e.g. with the tangent method [6]. But using the information of output-signal 1 the travel time can be calculated very easily and accurately via subtraction of the output-signals. The subsequent procedure for calculating the spatial water content distribution can be found in [3] and [4].

3 Dike Model and Instrumentation

3.1 Dike Model

In order to investigate the suitability of recycling material for dike construction a full-scale dike model was constructed by the Institute of Water Resources Engineering at the University of Darmstadt. Here the dike model was divided in two sections using a vertically embedded clay-seal (cf. Fig. 3) so that two different recycling materials could be tested as supporting shell. In both sections the dike has a zoned cross-section with a clayey material as a sealing (cf. Fig. 4). In one section the supporting shell consists of building rubble and in the other section of slag from refuse incineration. From the grain size distributions of Fig. 5 it is obvious that both materials contain a high amount of gravel. The building rubble even contains about 50 Mass-% gravel of 1 cm in diameter and higher.

Regarding the dimensions of the dike it is about 3 m in height from the base up to the crest and about 55 m in length. On the waterside of the model there is a sheet pile wall enfolding a basin. The inside of the sheet pile wall as well as the base of the dike model are sealed using a plastic sheet. As a result water infiltrating the dike is retained on the plastic seal and flows to a drainage blanket on the landside toe.
3.2 Instrumentation

The dike model is equipped with twelve switch-controlled flat band cables; five in the sealing element and seven in the supporting shell. In order to avoid changing materials along one transmission line, three profiles were divided into two ranges (at $y = 10$ m, $11$ m and $12$ m, cf. Fig. 4). In the middle of each section of the dike model one cross-section was equipped with flat band cables (cf. Fig. 3). In addition the model was equipped with observation wells in order to measure the water level at specific points.

The TDR-device (TDR 100 from Campbell Scientific), one multiplexer and the relay device are located in a weatherproof box on the crest of the dike right in the middle of the dike model above the vertical clay seal (called the main junction box, cf. Fig. 3). From here the flat band cables in both cross-sections as well as the switches are activated using further multiplexers and electrical points in the tributary junction boxes of the cross-sections. A conventional PC in the monitoring and data collection container takes over the control of the whole
measurement system. This measurement system is the first of this kind. At present another improved version is in operation on a dike model in Karlsruhe.

![Grain size distribution of the supporting shell materials](image)

**Fig. 5 Grain size distribution of the supporting shell materials**

### 4 Results

#### 4.1 Material Calibration

Laboratory calibration measurements were carried out in order to estimate both dielectric and electric properties of the soil. Because of the high amount of gravel in each recycling material it was necessary to separate grain sizes larger than 1 cm in diameter. The determination of the relative permittivity occurred using a two wire TDR probe with insulated metallic forks and a Tektronix 1502B cable tester as a TDR device. For the measurement of the specific conductivity the electrical resistivity method was used [7].

For the calibration measurements the soil sample was inserted into a PVC-tube (approx. 25 cm in height and 10 cm in diameter) and compacted with a specific water content. At the open ends of the tube electrodes were located over the whole surface of the sample for the power supply. Using the measured potential loss at specific points the conductivity was determined. Subsequently the TDR measurements were carried out repetitive on the undisturbed soil sample with different positions of the TDR probe. Finally the water content of the soil sample was determined with the oven drying method.

The results of the calibration measurements for both recycling materials are shown in Fig. 6. In the left graph the relationship between relative permittivity and volumetric water content is presented. In addition the well known calibration function according to Topp [8] has been entered in the graph. As can be seen both recycling materials seem to have a high relative permittivity in a dry state. Up to a volumetric water content of 25 Vol-% the measured
relative permittivity corresponds closely to the relationship given by Topp. But for higher values of the volumetric water content the relative permittivity of both materials differs greatly from Topp’s curve. One reason for this behaviour was found in the electric property of the soil which is defined in the relationship between saturation and specific conductivity shown on the right side of Fig. 6. Measurements on sand (rhombus) and clay (triangle) are also marked in the graph. Here both recycling materials show a much higher specific conductivity for higher saturation values than natural soils. Nevertheless calibration functions were derived from the measurements of the relative permittivity in order to obtain somewhat realistic water contents on the dike model.

Fig. 6 Relationship between relative permittivity \( \varepsilon \) and volumetric water content \( \theta \) (left) and between saturation \( S \) and specific conductivity \( \sigma \) (right) of the supporting shell materials (result of laboratory experiments)

4.2 Measurements

A simulation of a flood event was carried out in September 2003 on the dike model. Fig. 7 shows the results of TDR measurements as water content distribution in comparison with water level measurements presented as triangles in the supporting shell as well as in the sealing. The positions of the flat band cables are shown by the dots and the values beside them give the measurements at these points. For a better visualisation the reconstructed water content profiles are interpolated between the flat band cables.

The upper graph in Fig. 7 shows the water content distribution in cross-section A with slag from refuse incineration as material for the supporting shell. On the one hand reasonable measurement values of the water content can be seen here since the porosity of the material is about 38%. On the other hand it can be seen that the transition of the saturated zone (dark grey colours with water contents of 35 Vol-% and higher) to the unsaturated zone above (light grey colours with water contents less than 35 Vol-%) corresponds well to water level measurements. So far it has not been possible to give any accuracy of the measured water contents, since there are no measurements from independent methods for comparison. But the spatial resolution of the TDR measurements seems to have a satisfactory accuracy of a few centimetres.
The lower graph in Fig. 7 shows the water content distribution in cross-section B with building rubble as material for the supporting shell. The reconstructed water content profiles show suboptimal results, so that very high values of the water content (up to 51 Vol-%) appear in the saturated zone whereas in the unsaturated zone even negative values are shown. The reason for this lies mainly in the high amount of gravel in the soil. For determining the calibration function in the laboratory the gravel was separated. Nevertheless the TDR measurements in the building rubble also show that the transition from the saturated to the unsaturated zone corresponds well with the water level measurements.

Fig. 7 Distribution of volumetric water content in cross-section A with slag from refuse incineration as supporting shell (above) and in cross-section B with building rubble as supporting shell (below) as results of spatial TDR measurements

5 Summary and Conclusion

This contribution deals with spatial water content measurements using TDR in high electrical lossy earth materials. For this purpose a new measurement set-up for transmission lines based on flat band cables was developed. Switches at the ends of the transmission line make it possible to take TDR measurements under different electrical boundary conditions. So travel time analysis is possible with simple signal processing methods. The measuring set-up was installed and tested on a full-scale dike model constructed with recycling materials.

Laboratory experiments show here that both materials used for the supporting shell of the dike model have very high specific conductivities for increasing water contents. However it was possible to obtain satisfying results for the TDR measurements at least for slag from refuse incineration.
The measurements on the dike model have pointed out clearly some unanswered or inaccurate aspects concerning spatial TDR measurements especially in electrical lossy media and in soils with a high amount of gravel:

- Investigation of electric and dielectric properties of lossy soils especially regarding frequency-dependent behaviour.
- Enhancement of calibration methods especially for electrical lossy soils and for soils with a high amount of gravel.
- Development of new mixing rules particularly for electrical lossy soils.
- Investigations on the determination of the accuracy of spatial TDR measurements in such materials.

Among other things these questions will be the subject of investigation of the newly formed scientific workgroup SMG (Soil Moisture Group – Forschergruppe Feuchtemesstechnik) at the University of Karlsruhe in the future.

Acknowledgement

The investigations on the dike model concerning the suitability of recycling materials for dike construction were carried out by the Institute of Water Resources Engineering at the Technical University of Darmstadt in cooperation with the Institute for Road and Railway Engineering at the University of Karlsruhe. The authors would like to express their gratitude to both co-operation partners for permission to publish these results.

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Some Topics of Measurement of Complex Permittivity for Lossy Dielectric Materials with Wide-Range of tanδ

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ABSTRACT. Real electric materials may be described either as non-ideal (i.e. lossy) dielectrics or as non-ideal (i.e. \( \kappa < \infty \)) conductors. The transition range \(0.01 < \tan \delta < 100\) (high-loss range) sometimes requires special attention. In this range remarkable effects are
- appearance of auxiliary resonant frequencies in limited ranges of \( \varepsilon'_r \) and \( \tan \delta \), depending on the kind and amount of coupling and
- transitions between waveguide modes (e.g. from \( E_{01} \) to TEM-mode in a circular cavity with a centered rod of lossy dielectric).

A few examples and their effect on e.g. calibration functions will be discussed.

Keywords: dielectrics, high losses, sensor, calibration

1 Introduction

Resonators and waveguides – filled with or coupled to the test material – are the main types of sensors for measuring complex permittivity (and further for moisture) in RF- and microwave-range. The corresponding sensor parameters (resonant frequency \( f_r \) and quality factor \( Q \) or phase constant \( \alpha \) and attenuation constant \( \beta \), resp.) depend clearly on \( \varepsilon'_r \) and \( \tan \delta \).

Both said effects (see abstract above), depending also on the dimensions and position of the probe, lead to ambiguous solutions and to non-unique calibration functions. Such ambivalence and other possible deformations of the calibration functions and its graphic plots can be found in an adequate model by means of a detailed inspection of the characteristic equation (see Fig.3) over the complete range of interested values of at least frequency, \( f_r \) and \( \tan \delta \). These deformations may be avoided or at least shifted to a range out of interest by correspondingly modifying the test equipment (i.e. modifying the coupling of the probe).

A few typical situations will be demonstrated in the paper: shunt resonator with coupling, single mode TEM-resonator, and two other objects from literature.

Some parts of the following text are overtaken from a mathcad-worksheet.

2 Propagation Constant and Wave Impedance

The following values hold for unlimited, linear, dispersionless, isotropic and lossy media with \( \mu = \mu_0 \).

The losses are described by

\[
\tan \delta \left( f, \varepsilon'_r, \varepsilon''_r, \kappa \right) := \frac{\varepsilon''_r + \frac{\kappa}{2\pi \varepsilon_0} \cdot \frac{1}{f}}{\varepsilon'_r}
\]

(tande stands for tangens delta epsilon).
2.1 Propagation Constant

From Collin [2] we get\(^1\) for waveguides with assumed ideal walls, but filled with lossy dielectric material, the propagation constant \(\gamma\) as

\[
\gamma = \beta + \alpha = \left[ (k_c^2 - \omega^2 \mu_0 \varepsilon)^2 + (\omega^2 \mu_0 \varepsilon \tan \delta_t)^2 \right]^{1/4} e^{j\theta/2}
\]

(2)

with \(\theta = \tan^{-1} \frac{\omega^2 \mu_0 \varepsilon \tan \delta_t}{k_c^2 - \omega^2 \mu_0 \varepsilon}\) (3)

The following pictures show in normalised form (divided by the critical wavenumber \(k_c\)) the corresponding real and imaginary parts for some values of tande.

![Fig. 1 Attenuation constant (left side) and phase constant (right side) of lossy dielectrics](image)

From Fig. 1 it is easy to see that

- at any lossy case there is no rigid cut-off behavior; waves are propagating also at \(f < f_c\), but with rather high attenuation (see curves for tande > 0.01),
- at high losses fields are controlled by skin effect (see curves for tande > 1)

2.2 Wave Impedance

The wave impedance of a plane wave in an unlimited lossy dielectric medium, divided by its no-loss value, is the complex wave impedance (relative)

\[
ZF_{rel}(f, \varepsilon', \varepsilon'', \kappa) = 1/ \sqrt{1 - j \cdot \text{tande}(f, \varepsilon', \varepsilon'', \kappa)}
\]

(4)

\(^1\) In [2], p.341, eq.40, the exponent at the bracket term should be read 1/4 instead of 1/2
$ZF$ is the main part of the waveguide characteristic impedances which additionally depends on frequency and geometrical dimensions.

![Complex wave impedance](image)

Fig. 2 The relative complex wave impedance of a lossy dielectric material

This impedance shows nearly pure real values for very low and for very high values of $\tan\delta$. At about $\tan\delta = 2$ there is a smooth maximum of the imaginary component of this impedance.

### 3 Resonant Circuits with Lumped Elements

#### 3.1 Capacitively Coupled

Fig. 3 Shunt resonator, capacitively coupled with probe admittance

The characteristic equation of this shunt resonator is

$$Y_{\text{total}} = 0 \quad (5)$$

It leads via complex frequency to the usually measured resonant frequency and Q-factor. In this case after some intermediate steps one gets with the following components of the probe admittance

$$BP\left(f, \varepsilon'_r, \tan\delta, CP\right) := 2 \cdot \pi \cdot f \cdot CP \cdot \varepsilon'_r \quad (6)$$

$$GP\left(f, \varepsilon'_r, \tan\delta, CP\right) := 2 \cdot \pi \cdot f \cdot CP \cdot \varepsilon'_r \cdot \tan\delta \quad (7)$$

the total circuit admittance
\[ Y(f, \varepsilon_r, \tande, \text{CP}, C1, \text{Ck}, L0, G0) := G0 + \frac{1}{j \cdot 2 \cdot \pi \cdot f \cdot L0} + \text{Y2}(f, \varepsilon_r, \tande, \text{CP}, C1, \text{Ck}) \] 

(8)

The evaluation of eq. (8) with regard to the parameters of the circuit (or sensor) give the following diagrams:

(const. values: \(L0 = 1\, \text{nH}; G0 = 0.5\, \text{mS}; C1 = 6 \times 10^{-3}\, \text{nF}; \text{Ck} = 30 \times 10^{-3}\, \text{nF}; \text{CP} = 30 \times 10^{-3}\, \text{nF})

Fig. 4 Resonant frequency vs. \(\tande\) (param.: \(\varepsilon_r\))

Fig. 5 Q-factor vs. \(\tande\) (param.: \(\varepsilon_r\))

Fig. 6 Resonant resistance vs. \(\tande\)

Fig. 7 Complex frequency for \(\tande = 0 \ldots 10\)

Typical results for this capacitive coupling are
- rapid decreasing of sensitivity concerning permittivity for \(\tande > 0.2\)
- double-valued data for evaluating \(\tande\)
- no secondary resonances and
- easy to get the locus curve for the complex frequency \(f_k\) from the characteristic equation.
The connection between the complex frequency data and the usual resonator data is given by the following relations (with $f_k = f_{kre} + j^* f_{kim}$):

- the frequency without losses and probe: $f_o = f 0(L0, C1, Ck)$
- the actual resonant frequency $f_r(\varepsilon', \tan\delta) = f_{kre}(\varepsilon', \tan\delta)$
- the actual Q-factor $Q_r(\varepsilon', \tan\delta) = \frac{f_o}{2 \cdot f_{kim}(\varepsilon', \tan\delta)}$

### 3.2 Inductively Coupled

Fig. 8 Shunt resonator, inductively coupled with probe admittance $Y_P$

In this circuit the elements $L_k$, BP and GP form a secondary resonant circuit leading to a secondary loop in the locus curve of the total admittance (see Fig. 9, parameter $\varepsilon_r$). Any point of intersection with the horizontal line “susceptance = 0” represents a point of resonance. In this example with $\varepsilon_r = 9.25$ there are three such points for about $7 < \varepsilon_r < 10$. This range depends also on $\tan\delta$ and on the initial coupling factor $L_k / L_1$.

**Fig. 9 Admittance locus curves**

**Fig. 10 Secondary resonance**

The sequence of the kind of resonance of this three points along the frequency scale in Fig. 10 is shunt – series – shunt. The reason for this effect of secondary (etc.) resonance is the distribution of electrical and magnetic field energy in different space volumes (or different lumped elements) and its interaction with each other and this with pronounced frequency dependence. It leads to the generalized resonance condition

$$\tilde{W}_{electric}(\omega_r) = \tilde{W}_{magnetic}(\omega_r)$$ (9)
The shunt resonator (Fig. 8) is only a simple example, but it is good for demonstrating the main effect at least in limited frequency ranges. In practice, working with waveguides and its several modes and/or discontinuities, one has to expect a diversity of similar effects as above. Probably some of the so called “outliers”, reported on in the literature, may result from such an effect.

4 Single-Mode TEM – Resonator

The following arrangement (Fig 10) may be driven either as a TEM-section, connected with a network analyser, or, with short circuits at both ends, as a resonator, loaded by a probe.

Here it is advisable to formulate the over-all scattering matrix with considering the three lengths, the complex permittivity, and the frequency as six variables and only the diameters and the conductivity of the transmission line as three specific constants. By using a slider (e.g. at mathcad software), each one for any variable, it is easily possible to change any variable at will and to observe the result nearly immediately. Some typical results are

- in low-loss case: increasing the real permittivity leads mainly to growing multiplicity of resonances with 1/Q proportional to \( \tan \delta \).

- in medium-loss case: increasing attenuation in the probe section lead to separation of the sections L1 and L3, which now act as two resonators, coupled with each other via the probe. This includes the suppressing of any resonances by matching the impedances at certain small frequency ranges.

- for high values of \( \tan \delta \): the sections L1 and L3 are nearly completely decoupled; the remaining two resonators are mainly influenced via Q(\( \tan \delta \)).

Obviously, using a network analyser for this test set of Fig. 10 offers much more versatility for the data acquisition.
Fig. 12 Resonant frequency and Q-factor versus complex permittivity (from [3])

Fig. 13 Complex wavenumber versus conductivity (from [11])
5 Two Other Objects

In Fig. 12 is shown a kind of calibration diagram resulting from a circular cylindric probe in the centre of a circular cylindric waveguide. With increasing tande changed the waveguide mode from H011-mode in cylindric waveguide continuously to H011-mode in a coaxial transmission line.

Fig. 13 represents the locus curve of the complex wavenumber (proportional to a complex cut-off frequency) for a rectangular waveguide with an insert with conductivity (proportional to tande) from zero to infinity.

Both objects expose effects comparable to those of the simple circuits plus additionally the influence of other modes, inclusive plus transitions from one low-loss-mode to an other mode at higher values of tande.

6 Conclusions

The examples as above - in spite of being rather simple - show some inconvenient situations in procedures for the measurement of complex permittivity and of moisture, resp. Their physical background is the simultaneous presence of electric and magnetic energy (or conduction and displacement current) and its interaction with each other. To avoid (or to take into account) such inconvenient situations one should establish a proper (computer-) model of the real object and analyse in all relevant aspects.

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Material Parameter Measurement of Soils and Liquids with a Waveguide Setup

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ABSTRACT. The dielectric parameters of different soils and sand as well as of some liquids are determined in dependency of moisture content and temperature in a frequency range from 2 – 3 GHz. In this paper a waveguide measurement technique is used for the determination of these material parameters. The method is based upon the measurement of transmission and reflection coefficients in a rectangular waveguide with a Vector Network Analyzer (VNA). The dielectric properties of the material under test (MUT) are then calculated from the measured scattering parameters. To allow for measurements of liquid and granular materials the MUT is placed between two dielectric walls within the waveguide. In contrast to several other techniques this measurement setup is also suited for high loss materials.

1 Introduction

For investigations of wave propagation in soils it is necessary to have a detailed knowledge of the dielectric properties of the different soils. These material parameters depend highly on the moisture content of the soils, but also on the soil type, the temperature, the frequency and the salinity [1]. The soil type depends on the region and the depth. For this research, soils of the rhenish massif, a mountainous region in Germany, were investigated. The soil samples are taken from different depths and horizons. In this way it is possible to determine the material parameters of a complete soil profile at a certain position.

There exist several well-known ways for measuring the dielectric properties of materials. The use of a resonator that is detuned by the inserted MUT is very popular [2]. This method provides a very high accuracy since it depends on the measurement of the resonance frequency, however it suffers from several disadvantages. The range of the dielectric constant has to be known in advance for the proper dimensions of the resonator, as the measured result is only valid around the resonance frequency. Furthermore, this method is not suited for materials with high losses, because the quality factor is rather low.

Another way to determine material properties is based on the measurement of transmission and reflection. This can either be done with a waveguide [3] or with a free-space measurement setup [4]. The free-space measurement is mainly suited for higher frequencies because of the antenna and sample size. With this method the sample has to be placed in the far field of the antenna and the sample has to be large enough to avoid unwanted contributions of diffraction.

The advantage of waveguide measurements is that the propagation mode as well as the boundary conditions are exactly known and can therefore be described analytically. If the complex scattering parameters are known, it is possible to calculate the permittivity and the losses of the material under test. A new computation algorithm for the complex permittivity $\varepsilon_r$ from the scattering parameters $S_{11}$ and $S_{21}$ is outlined in this paper.
2 Applicator Design

For measurements in the frequency range from 2 – 3 GHz a standard S-band waveguide with dimensions of \( a = 86.36 \) mm and \( b = 43.18 \) mm is used. The applicator has an overall length of 220 mm and consists of brass. Two dielectric walls are inserted into the waveguides cross-section as can be seen in Fig. 1. The dielectric walls are made of PTFE (Polytetrafluoroethylene, Teflon). They are inserted into the waveguide with such pressure that air gaps between the dielectric walls and the waveguide walls are avoided. It is taken care that the surface of the dielectric plugs is perpendicular to the propagation direction of the electromagnetic wave inside the waveguide.

The material under test (MUT), that can either be granular or liquid, is placed between the two dielectric walls by opening one sidewall of the waveguide. The whole applicator is arranged in a way that the sidewall of the waveguide forms the top cover of the applicator. Hence, air gaps between the MUT and the critical walls (bottom and top wall) are avoided. The electrical field is nearly zero at the sidewalls for the considered TE\(_{10}\) mode and for this reason a potential air gap is not critical. Nevertheless, it is taken care that air gaps are minimized between the MUT and the sidewall that forms the top cover of the applicator. After filling the waveguide is closed. The material combination of brass and PTFE allows for measurements over a broad temperature range as well as for determination of material parameters of solvents or other corrosive liquids.

3 Determination of the Dielectric Material Parameters

3.1 Wave Propagation in a Multi-Layered Medium

Inside the applicator a five-layered structure has to be considered. This structure is illustrated in Fig. 2.

Layer 1 and layer 5 consist of air \( (\varepsilon_{r1} = \varepsilon_{r5} = 1) \), medium 2 and medium 4 consist of PTFE \( (\varepsilon_{r2} = \varepsilon_{r4} = 2.105 – j \cdot 4.21 \cdot 10^{-4}) \), and layer 3 represents the MUT. \( d_i \) is the thickness of the different layers and \( k \) is the direction of propagation of the waves where the index \( f \) means forward direction (+z-direction) and index \( r \) means the backward propagation (-z-direction), respectively.
Fig. 2 Structure with five layers inside the applicator

To derive the transmission and reflection coefficients, the forward and backward travelling waves have to be superposed with respect to the boundary conditions. At each boundary the electromagnetic wave is partially reflected and transmitted. Amplitude and phase of the reflected and transmitted waves depend on the dielectric properties of the walls and the material under test, respectively as well as on their electrical lengths. In this approach a TE\textsubscript{10} mode is assumed. Higher-order modes can exist inside a transition region from the coaxial feed to the waveguide. By choosing a proper length (20 cm) of the coaxial-waveguide-adaptors it is ensured that higher modes are substantially attenuated. The E-field vector is tangential to the boundaries and has therefore to be continuous at each boundary. The tangential component of the H-field also has to fulfil these conditions, and with this the E-field and the H-field can be described by the following equations

\[
E_{f,r} \cdot e^{-\gamma_{d1}} + E_{i,r} \cdot e^{\gamma_{d1}} = E_{i+1,r} + E_{i+1,f} \quad (1)
\]

\[
H_{f,r} \cdot e^{-\gamma_{d1}} - H_{i,r} \cdot e^{\gamma_{d1}} = H_{i+1,r} - H_{i+1,f} \quad (2)
\]

The E-field is depending on the dielectric parameters in the following way

\[
E_i = -j \frac{\omega \mu_0}{\omega^2 \varepsilon_0 (\varepsilon_{r,i}^\prime - j \varepsilon_{r,i}^\ast) \mu_0 + \gamma_i^2} \cdot \frac{\pi}{a} \cdot H_0 \sin \left( \frac{\pi x}{a} \right) \quad (3)
\]

\(\gamma_i\) represents the complex propagation constant in the different layers and is defined as

\[
\gamma_i = \sqrt{\left( \frac{\pi}{a} \right)^2 - \left( \frac{\omega}{\varepsilon_0} \right)^2 \left( \varepsilon_{r,i}^\prime - j \varepsilon_{r,i}^\ast \right)} \quad (4)
\]

where \(\omega\) is the angular frequency, \(a\) is the width of the waveguide, and \(c_0\) is the speed of light in vacuum. The material parameters are denoted by the complex \(\varepsilon_{r,i}\).

The complex reflection coefficient \(S_{11}\) at port 1 can be determined as the ratio of the reflected E-field to the incident E-field in region 1

\[
S_{11} = \frac{E_{1,r}}{E_{1,f}} \quad (5)
\]

and the complex transmission coefficient \(S_{21}\) between port 2 and port 1 is defined as the ratio of the transmitted E-field in region 5 to the incident E-field in region 1 assuming no backward propagation in region 5 (\(k_{5,f} = 0\)).

\[
S_{21} = \frac{E_{5,f}}{E_{1,f}} \quad (6)
\]

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3.2 Computation of the Complex Permittivity

The procedure described in 3.1 allows for the forward calculation of the complex scattering parameters $S_{11}$ and $S_{21}$ in dependency of the dielectric properties and the thickness of the different layers. Since with the measurement setup the complex scattering parameters $S_{11}$ and $S_{21}$ of the applicator are determined, it is necessary to compute the dielectric material parameters of the MUT from the $S$-parameters.

\[
S_{11} - S_{11}^\text{meas} = \frac{E_{1,r}}{E_{1,f}} - S_{11}^\text{meas} = 0 \tag{7}
\]

\[
S_{21} - S_{21}^\text{meas} = \frac{E_{5,f}}{E_{1,f}} - S_{21}^\text{meas} = 0 \tag{8}
\]

If the incident E-field $E_{1,f}$ is known, the $E_{1,r}$ and $E_{5,f}$ are determined to fulfil equations (7) and (8) by variation of the dielectric parameters of the MUT. The backwards calculation of the material parameters is ambiguous if only magnitude or phase are of the measured $S$-parameters is considered. Hence, magnitude as well as phase of $S_{11}$ and $S_{21}$ is taken into account. This is accomplished by solving a set of non-linear transcendental equations for magnitude, real part and imaginary part of each measured $S$-parameter. The first step is an initial guess of the starting value after which an iterative loop starts until a solution for $\varepsilon'$ and $\varepsilon''$ of the material under test is found. For every solution the residuum of real part, imaginary part and magnitude is calculated. The correct material parameters are obtained when the square sum of these residues is minimum.

4 Measurement Setup

The measurement setup consists of an Agilent E8357A vector network analyser (VNA), two coaxial-waveguide-adaptors and the applicator. The system is shown in Figure 3.

![Fig. 3 Measurement setup](image)

4.1 Calibration

Prior to the measurements a TRL (transmission, reflection, line) waveguide calibration is performed by measuring three standards: short, through and a quarter-wavelength delay line. This allows for determining the $S$-parameters of the applicator itself. The reference planes are
located at the ports of the applicator as shown in Fig. 3. In this way it is not necessary to know the S-parameters of the coaxial-to-waveguide adaptors since they are calibrated out. The accuracy of the calibration is verified by measuring the well-known materials air ($\varepsilon_r = 1$) and distilled water at $25^\circ C$ [1] ($\varepsilon_r = 78-j9$). The accuracy found to be in a range of 3.5%.

4.2 Soil Samples and Sample Preparation

Table 1  Properties of the soil samples

<table>
<thead>
<tr>
<th>Labno.</th>
<th>Horizon</th>
<th>Depth [cm]</th>
<th>Stone [%]</th>
<th>Sand [%]</th>
<th>Silt [%]</th>
<th>Clay [%]</th>
<th>Density [g/cm$^3$]</th>
<th>$\varepsilon$ meas. (dry)</th>
</tr>
</thead>
<tbody>
<tr>
<td>44421</td>
<td>Ap</td>
<td>0 – 20</td>
<td>13.53</td>
<td>27.65</td>
<td>53.46</td>
<td>18.88</td>
<td>1.54</td>
<td>2.89 – 0.17i</td>
</tr>
<tr>
<td>44422</td>
<td>Bv</td>
<td>20 – 45</td>
<td>10.17</td>
<td>22.42</td>
<td>46.86</td>
<td>30.72</td>
<td>1.48</td>
<td>2.68 – 0.12i</td>
</tr>
<tr>
<td>44423</td>
<td>II Al-sSew</td>
<td>45 – 73</td>
<td>24.46</td>
<td>29.04</td>
<td>61.76</td>
<td>9.20</td>
<td>1.66</td>
<td>2.92 – 0.17i</td>
</tr>
<tr>
<td>44424</td>
<td>III Bt-sSd</td>
<td>73 +</td>
<td>19.62</td>
<td>26.12</td>
<td>51.00</td>
<td>22.88</td>
<td>3.31</td>
<td>3.12 – 0.24i</td>
</tr>
<tr>
<td>44393</td>
<td>Ah</td>
<td>0 – 7</td>
<td>23.84</td>
<td>17.14</td>
<td>58.86</td>
<td>24.00</td>
<td>1.06</td>
<td>2.22 – 0.05i</td>
</tr>
<tr>
<td>44394</td>
<td>Al</td>
<td>7 – 40</td>
<td>27.92</td>
<td>16.85</td>
<td>61.09</td>
<td>22.06</td>
<td>1.21</td>
<td>2.68 – 0.07i</td>
</tr>
<tr>
<td>44395</td>
<td>II Bt</td>
<td>40 – 55</td>
<td>24.07</td>
<td>18.22</td>
<td>58.56</td>
<td>23.22</td>
<td>1.35</td>
<td>2.96 – 0.19i</td>
</tr>
<tr>
<td>44396</td>
<td>III ilCv</td>
<td>55 +</td>
<td>89.28</td>
<td>38.85</td>
<td>45.64</td>
<td>15.50</td>
<td>2.66</td>
<td>3.06 – 0.06i</td>
</tr>
</tbody>
</table>

The soil samples are taken from the rhenish massif in Germany. The samples corresponding to laboratory numbers 44421 – 44424 form one complete profile from a lower region whereas the numbers 44396 – 44396 represents the second one from another location at a higher region. In Table 1 detailed information about the samples is given. Stones and particles with a size larger than 2 mm build up the skeleton of the soil. The sum of the fractions of sand, silt and clay is considered to be 100% of the soil mass. More information about the definition of soils properties can be found in [5].

Each soil sample was dried and pulverised prior to the measurements. In order to achieve the different gravimetric moisture contents distilled water was added to the samples. The volumetric moisture content can easily be derived from the gravimetric moisture content using equation (9) where $\rho$ is the density of the dry soil and the liquid, respectively.

$$ r_{vol} = \frac{\rho_{dry\ soil}}{\rho_{liquid}} \cdot r_m $$

$$ r_{vol} = \frac{\text{volume of liquid in sample}}{\text{volume of test sample}}, \quad r_m = \frac{\text{mass of liquid in sample}}{\text{mass of dry soil in sample}} $$

5 Results

The dielectric properties of the soil samples are investigated as a function of frequency and gravimetric moisture content. Additionally, a sample of sand is measured for different moisture contents at a frequency of $f = 2.45$ GHz and the measured results are compared to calculated values.

The liquids acetone, ethanole, isopropanol as well as benzin, diesel and motor oil are measured for different temperatures in a frequency range from 2 – 3 GHz.
In Fig. 4 it can be seen that the real part of the soils’ permittivity is nearly independent from frequency but is strongly influenced by the moisture content. The difference in the permittivity is nearly linear by increasing the moisture content up to 20% but shows a larger difference when the moisture content is further increased. This could be due to the saturation of the soil. The imaginary part shows a stronger variation over frequency. Especially for a moisture content of 30% a resonant behaviour can be observed.

The measured permittivity of sand for different moisture contents is compared to the values calculated by the formula of Peplinski et al. [6, 7]. The results show a good agreement and are illustrated in Fig. 5.

The variation of the dielectric material parameters of soil samples over the depth was also investigated and the results are shown in Fig. 6. The bright bars show the samples 44421 – 44424 whereas the darker bars indicate samples 44393 – 44396 (cf. Table 1). It can be observed that the imaginary part of the permittivity varies much more with depth than the real part.
In a second measurement campaign different liquids have been investigated. The results for some alcohols are shown in Fig. 7. The temperature is varied in a range from 20°C to 70°C. Additionally, the permittivity of benzine, diesel and oil is measured. In case of acetone and benzine the boiling point is 55°C and around 45°C, respectively. It is shown, that alcohols have far higher permittivity and losses compared to fuel and oil (Fig. 8). This is due to the non-polar structure of alkanes, which are essential parts of fuels and oils.

The permittivity of ethanol is illustrated in Fig. 9. The real part decreases with increasing frequency. Furthermore, it drops with higher temperatures. The losses show a frequency dependent behaviour in a temperature range from 20°C to 40°C but are nearly constant over frequency at higher temperatures.

Fig. 6 Permittivity of dry soil samples as function of depth

Fig. 7 Permittivity of different alcohols as function of temperature

Fig. 8 Permittivity of fuels and motor oil as function of temperature
6 Conclusions

In this paper a waveguide measurement technique for determination of dielectric properties of granular materials and liquids is presented. The theoretical approach is illustrated and the algorithm for the determination of the material parameters is described. With this setup extensive measurements of soils and liquids have been performed. It has been show that the waveguide measurement setup is also well suited for the determination of materials with high losses.

Acknowledgement

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Session 5

**Measurement Methods**
Chairmen: T. Lasri, J. Sachs

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High Moisture Content Measurement Using Microwave Free-Space Technique

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ABSTRACT. High moisture content measurement in actual production line is required. In this paper, two methods use the microwave free-space technique are proposed for density-independent high moisture measurement. The first method determine the moisture content by measuring microwave attenuation and phase shift at 9 GHz caused by material under test, and the second method is based on measuring microwave phase shifts at 12 GHz and 9 GHz. The results of the experiments on sawdust sample are given. The standard error of calibration for the moisture determination by the first method is 12.9% in the moisture content range from 190% to 350%, and that by the second method is 8.9% in the same moisture range.

Keywords: microwave, high moisture content, free-space, density-independent

1 Introduction

Measurement of high moisture content is required in the actual production line. In green tea production line in Japan, fresh tea leaf is usually in the moisture content range from 350 to 400% on a dry basis. The tea leaf must be steamed and dried for 8-9 steps to be the green tea products with 4% moisture content. It is necessary to know the moisture content of tea leaves in each producing process. Methods to measure the moisture content range from 4 to 350% are required. Additionally, the methods are expected to be able to determine the moisture content independent of sample density because variation of sample density is the main error source in the moisture measurement by the microwave techniques [1], [2], [3], [4], [5]. A method using the microwave free-space technique now is widely applied in the actual production line [2]. The method is able to measure the moisture content of tea leaf ranging from 30 to 180% on dry basis independent of sample density. It is necessary to develop a method that is able to measure high moisture content above 180% independent of density.

In this paper, two methods using the microwave free-space technique are proposed to measure the high moisture content range from 190% to 350% on dry basis. The methods measure attenuation and phase shift of a microwave signal transmitted through a layer of material under test. The moisture content is determined using the measured attenuation and phase shift. The experimental results carried out with samples of sawdust are presented.

2 Principle

The moisture content on a dry basis, $M$, is usually defined as following, where $W_w$ and $W_d$ are the masses of water and dry material, respectively.

\[
M = \frac{W_w}{W_d} \times 100\%
\]  

(1)

The block diagram of the moisture measurement setup using the microwave free-space technique is shown in Fig. 1. Material under test is inserted between two horn antennas, and a microwave signal is transmitted through the material. The interaction between the microwave
and the material causes the amplitude of microwave to be attenuated, and the phase angle to be changed. The attenuation and phase shift are mainly caused by the water contained in the material, and are also affected by the dry material [1], [3], [5].

In general, the attenuation $\Delta A_1$ and the phase shift $\Delta \Phi_1$ at frequency $f_1$ can be expressed in following functional forms [1][6],

$$\Delta A_1 = \Psi_{a_1}(M, \rho) \cdot d$$

$$\Delta \Phi_1 = \Psi_{\phi_1}(M, \rho) \cdot d$$

where $\rho$ is the dry material density, $d$ is the material thickness, $\Psi_{a_1}(M, \rho)$, $\Psi_{\phi_1}(M, \rho)$ are functions of $M$, $\rho$. At another frequency $f_2$, the phase shift $\Delta \Phi_2$ similar with Eq.3 can be obtained,

$$\Delta \Phi_2 = \Psi_{\phi_2}(M, \rho) \cdot d$$

The first method we proposed in this paper uses Eq.2 and Eq.3 to determine the moisture content. Taking ratio of attenuation to phase shifts obtains,

$$\frac{\Delta A_1}{\Delta \Phi_1} = \frac{\Psi_{a_1}(M, \rho) \cdot d}{\Psi_{\phi_1}(M, \rho) \cdot d} = \Psi(M, \rho) \equiv R_{\rho}(\rho) \cdot \Psi_{\phi}(M, \rho)$$

where $\Psi(M, \rho)$ is a function of the density and the moisture content, $R_{\rho}(\rho)$ is a function of the density, $\Psi_{\phi}(M, \rho)$ is a function of the moisture content. It is reasonable to assume that $R_{\rho}(\rho)$ is a constant for small density variation of the same material [4], [5]. Thus the moisture content can be density-independent determined in the form,

$$M = g_1(\frac{\Delta A_1}{\Delta \Phi_1})$$

Similar to the first method, the second method is obtained using Eq.4 and Eq.3 to determine the moisture content [1], [6],

$$M = g_2(\frac{\Delta \Phi_2}{\Delta \Phi_1})$$

The two methods have been used for density-independent moisture measurement in the range from about 4% to about 40% [1], [5], [6]. In this paper, the methods will be tested for the high moisture range from 190% to 350%.

Fig. 1 Moisture measurement setup using the microwave free-space technique

In general, the attenuation $\Delta A_1$ and the phase shift $\Delta \Phi_1$ at frequency $f_1$ can be expressed in following functional forms [1][6],

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where $\Psi(M, \rho)$ is a function of the density and the moisture content, $R_{\rho}(\rho)$ is a function of the density, $R_{\phi}(M)$ is a function of the moisture content. It is reasonable to assume that $R_{\rho}(\rho)$ is a constant for small density variation of the same material [4], [5]. Thus the moisture content can be density-independent determined in the form,

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$$M = g_2(\frac{\Delta \Phi_2}{\Delta \Phi_1})$$

The two methods have been used for density-independent moisture measurement in the range from about 4% to about 40% [1], [5], [6]. In this paper, the methods will be tested for the high moisture range from 190% to 350%.
3 Experimental Method

The measurement system as shown in Fig. 1 was set up. Two rectangular horn antennas with 10.9 cm × 7.9 cm apertures and 20±2 dB gain were used as transmitting and receiving elements. The distance between the antennas was 26 cm. A plastic case was used to contain the material under test. The attenuation and phase shift used in the first method were measured at 9 GHz. The phase shifts used in the second method were measured at 9 GHz and 12 GHz, which are in the single frequency range of waveguide WR90. The attenuation and phase shifts were measured by a vector network analyzer (HP8720ES).

The sawdust named MOMIZAI was used in the measurement. At first, the sample was oven dried to 0% moisture content to obtain the mass of dry material $M_d$. Then the tap water was added into the sample until the moisture content was approximately 380%. The sample was stored in a sealed plastic-case for 3 days to obtain a uniform moisture distribution, and the first measurement was performed. After the first measurement, the sample was gradually dried at room temperature to be next moisture content, for example, 350% moisture content. Then the sample was again stored in the plastic-case. After 24 hours to obtain a uniform moisture distribution, the measurement at next moisture content was performed. The processes of store, measurement, dry were repeated till the moisture content to be approximately 180%, which was the lowest moisture content we investigated. The attenuation and phase shift at every moisture content level were measured twice to obtain an average value.

4 Experimental Results

Microwave attenuation and phase shift for two sawdust samples with different density were measured. The dry material density of one sample was 0.066g/cm$^3$, and that of another sample was 0.044g/cm$^3$. The attenuation normalized by the sample thickness with respect to the moisture content is shown in Fig.2. The phase shift normalized by the sample thickness with respect to the moisture content is shown in Fig.3. Both of the attenuation and the phase shift increase with the moisture content increasing, and they are affected by the density of sample. The samples at the same moisture content have different attenuation and phase shift because of different density.
The values of $\Delta A_1/\Delta \Phi_1$ and $\Delta \Phi_2/\Delta \Phi_1$ were calculated according to Eq.6 and Eq.7 for the two samples, and the results with respect to the moisture content are shown in Fig.4 and Fig.5. $\Delta A_1$ and $\Delta \Phi_1$ in Fig.4 are the attenuation and phase shift at 9GHz, and $\Delta \Phi_1$ and $\Delta \Phi_2$ in Fig.5 are the phase shifts at 9GHz and 12GHz respectively. Both $\Delta A_1/\Delta \Phi_1$ and $\Delta \Phi_2/\Delta \Phi_1$ of the samples at the same moisture content have similar value although the sample densities are different. The results show the proposed two methods are able to measure the high moisture content independent of sample density.
The calibration curves in Fig.4 and Fig.5 were used to predicate the moisture content of the two samples. The moisture content calculated from the $\Delta A_1/\Delta \Phi_1$ value versus the oven moisture content is shown in Fig.6, in which the straight line corresponds to the ideal relation. The moisture content calculated from the $\Delta \Phi_2/\Delta \Phi_1$ value versus the oven moisture content is shown in Fig.7.

![Fig. 6 Moisture content predicted by $\Delta A_1/\Delta \Phi_1$ with respect to oven moisture content, SEC=12.9%](image1)

![Fig. 7 Moisture content predicted by $\Delta \Phi_2/\Delta \Phi_1$ with respect to oven moisture content, SEC=8.9%](image2)

The standard error of calibration (SEC) is calculated to evaluate how well the moisture content is predicated [3]. The SEC is defined as

$$SEC = \sqrt{\frac{1}{n-p-1} \sum (\Delta e_i)^2}$$  \hspace{1cm} (3)

where $n$ is the number of measurement, $p$ is the number of variables in the regression equation with which the calibration is performed, and $\Delta e_i$ is the difference between the predicated value and that determined by a standard method for the sample. The value of SEC for the first method using $\Delta A_1/\Delta \Phi_1$ value as Eq.6 is 12.9%. The value of SEC for the second method using $\Delta \Phi_2/\Delta \Phi_1$ value as Eq.7 is 8.9%.

5 Conclusions

In this paper, two methods use the microwave free-space technique are proposed for density-independent high moisture measurement. The first method uses the ratio of attenuation to phase shift at 9 GHz to determine the moisture content, and the second method uses the ratio of phase shift at 12 GHz to that at 9 GHz. The experiments were performed using the samples of sawdust. The results show that the proposed methods are suitable for density-independent high moisture measurement. The standard error of calibration for the moisture determination for the first method is 12.9% in the moisture content range from 190% to 350%, and that for the second method is 8.9% in the same moisture range.
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Comparison of Free Space Reflection and Transmission Time-Domain Measurements for the Determination of the Moisture Content of Bulk Materials

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ABSTRACT. In this paper, ultra-wideband signals are utilised for moisture measurement of layers of solid bulk materials. The signals are radiated and received over free space via broadband antennas and the time domain shapes of the waveforms are directly evaluated in a multivariate calibration. The approach is based on the idea, that broadband time domain signals not only receive a time delay and attenuation by the moist material, but also a change of shape due to the dielectric dispersion, which depends on the composition. Time domain “transmissometry” has been applied earlier for the determination of the moisture content of bulk materials. In this paper transmission and reflection measurements are compared with each other in order to determine the material properties.

In both cases, the measurement signals are directly analysed in the time domain, using artificial neural networks. The networks are trained with a set of training data and the methods are verified and compared. This approach does not require determination or knowledge of the complex permittivity of the material, because the time domain data is directly correlated with the moisture content of the bulk material.

Keywords: TDR/T, UWB, Non-contacting, multivariate calibration

1 Introduction

Instantaneous determination of the moisture content of bulk materials in online industrial processes is often of great interest. Problems arise from different sources of perturbation, which complicate the determination of the moisture. A major problem is the requirement of independence of the measurement from the thickness of the material layer and its density, which can be achieved by the determination and the further processing of the attenuation and phase delay in the frequency domain over a certain frequency range [1,2].

Fig. 1 Gaussian monocycle measurement signal
A disadvantage of this method is a potentially high hardware cost, because a dedicated automatic network analyser has to be used. Another problem arises from phase ambiguities which may occur, if the thickness of the material layer exceeds one wavelength of the measurement signal [3].

In the approach presented in this paper, the problem is circumvented by using a free space time domain measurement setup. An approximate gaussian monocycle as shown in Fig. 1 is utilised as an ultra-wideband (UWB) measurement signal. The duration of the monocycle is $T_w = 400\text{ps}$. The measurement setup was already applied earlier in a transmission measurement system [4]. The pulse shape can directly be radiated by broadband horn-antennas and travels through the bulk of material between the antennas. During propagation through the material every spectral component of the signal is attenuated and delayed with respect to the material’s dispersion, which depends on its composition and, hence, on the moisture content, leading to a deformation of the shape of the pulse. The monocycles are radiated with a repetition rate of $T_r = 200\text{kHz}$. Their peak-to-peak voltage amplitudes $U_{pp}$ are approximately 5 V, in order to provide a sufficiently high signal to noise ratio. This pulse shape was chosen due to its favourably properties at low frequencies, because the amplitude of the spectrum decreases as it tends to zero.

In many cases, it is not possible to access the material under test from both sides, in order to perform a transmission measurement. Thus this paper discusses also a reflection measurement, which only needs one antenna for radiation and reception of the measurement signal. A reference plane having a high conductivity is an additional requirement in this case. The principle of measurement in both cases relies on exploiting not only the attenuation and the time delay of the travelling pulses, but also the changes of the pulse shape. Thus information is obtained about the dispersion properties of the moist material. An a priori knowledge about the permittivity is not required, because an artificial neural network is trained to interpret the measured waveforms and to determine the moisture content of the material.

### 2 Measurement Setups

#### 2.1 Transmission System

![Fig. 2 Transmission measurement system](image)

The transmission measurement setup is shown in Fig. 2. A step signal is generated by the TDR module 80E04 which is hosted in the Tektronix sampling oscilloscope mainframe TDS8000. The module generates a step with a 10-90% rise-time of approximately 35 ps. The
step is pre-filtered with a 100ps absorptive rise-time filter, in order to increase the rise-time, as shown in Fig. 3. Subsequently the signal is differentiated with a pulse former, yielding a pulse. Then it is split into two paths with a 6dB power splitter. One path is delayed and the other inverted, in order to provide two complementary time shifted pulses. The pulses are recombined in a 6dB power combiner. The recombination forms the monocycle, which has already been shown in Figure 1. For avoiding multiple reflections between the pulse forming network and the antenna, isolation is provided using a broadband amplifier and an attenuator. These components can also be used for the adjustment of the amplitude of the pulse signal.

![Fig. 3 Pulse forming network](image)

The pulse signal is radiated via a double ridged horn antenna and travels through the material under test, which is situated in a Rohacell container. Rohacell is a foam material with a relative permittivity of approximately 1. It is thus invisible for the electromagnetic wave. During the measurements the material’s layer thickness $w$ and its moisture content $\Psi$ are varied. The oppositely located receiving horn antenna is aligned in a distance of approximately 50cm to the radiating antenna. The double-ridged horn antennas operate in a frequency range between 1-18GHz. As test material, terracotta particles have been chosen, because of their high water holding capacity and their property of providing a fairly homogenous moisture distribution within the material layer.

After passing the receiving horn antenna the signal is pre-filtered in order to reduce random noise. The received pulses are amplified and then acquired in a sampling head, also hosted in the Tektronix mainframe, having an analogue input bandwidth of 20GHz and a time-base resolution of 500fs. The sampled time window is 2ns, leading to total number of 4000 sampled points in time. The received signal is smoothed by averaging with an averaging factor of 10. During the transmission measurements, the moisture content was varied over a range of approximately 6%-34% (wet basis) and the layer width was changed between 2-9cm. A total number of $M=30$ measurements on different conditions were carried out.

### 2.2 Reflection System

For the reflection measurement the pulse is radiated and received via an antenna of the same kind as in the transmission measurement after travelling twice through the moist material and being reflected at the aluminium back plate below.

In the experimental system a so called pickup-tee is used having the main signal path (see Fig. 4) between the first amplifier and the horn antenna. The receive path is connected to the loosely coupled port of that tee, accepting a loss in sensitivity and dynamic range for the experimental system. The pulse generation circuit is the same as for the previously described transmission measurement setup. In a fully engineered reflection measurement system, the signal would be sampled directly after the pulse forming network in order to circumvent the attenuation in the pickup-tee.
The monitored signal is pre-filtered, amplified and finally acquired in the sampling head, which is the same as previously used. The sampling resolution equals that of the transmission measurement.

Due to non-existent isolation between the transmit- and receive-circuits, the sampled output signal also contains spill-over components of the transmitted pulse (e.g. residuals of multiple reflections between antenna and amplifier) corrupting the pure reflection from the material. The situation is even worsened by the fact, that the desired reflection signal is attenuated on its journey through the material under test, while the spill-over of the transmitted signal directly monitored at the output of the pick-up tee may be quite large. Thus the received waveform is a superposition of fractions which are due to reflections from the material and the spill-over, which probably has a higher magnitude than the latter.

![Fig. 4 Setup of the reflection measurement System](image)

During the reflection measurements, the moisture content was varied in the range between 6%-34% whereas the layer thickness was varied between 2-9 cm, as in the case of transmission measurements. In total \( M = 50 \) measurements under different conditions were carried out, in order to have sufficient data for an extensive calibration.

### 3 Data Analysis

#### 3.1 Transmission System

The time-domain data are directly analysed. The information about the moisture content of the bulk material is derived from both the shape variation of the pulses and the time delay in the material. These data are considered to be specific for every combination of moisture content and layer width. Analysis of the pulse shapes delivers information about the material’s dispersion, which in turn is exploited for the determination of the moisture content. Before the analysis is started the signal is digitally low-pass filtered with a FIR-filter\(^1\) in order to eliminate noise. A typical reception signal of the transmission system is shown in Fig. 5. As can be seen, the samples of the measurement curve deliver a subset of discrete points within a time interval around the pulse maximum, which form a vector \( \vec{v} \) describing the condition of the material. The shape data in the vector \( \vec{v} \) is supplemented by digital representations of the delays of the pulse centroids, finally yielding a vector of data with \( N \)-entries.

---

\(^1\) Finite-Impulse-Response
When travelling through empty space, which is used as a reference, the shape of the received signal should resemble the first derivative of the transmitted gaussian monocycle. In practice it is deformed due to imperfections of the antennas and the surrounding of the measurement setup (e.g. antenna fixtures, etc.). Furthermore, the far field condition is not fulfilled for all frequency components. However, since, after introduction of the sample material, only the changes of the waveform are processed, these distortions can be tolerated.

$M$ different input vectors, each representing a particular measurement, are combined in a dataset and are expressed as a $N \times M$-Matrix. The principal components of that dataset are calculated [5] in order to de-correlate the data and to reduce the number of dimensions. A new truncated dataset, consisting of the most representative principal components, is finally used to train a radial basis artificial neural network [6], which is capable of estimating the moisture content of the material. The artificial neural network is further explained in Section 0.

Fig. 5 Typical transmission measurement curve

3.2 Reflection System

As already discussed in Section 0, reflections from the sample material are causing received waveforms with relatively small amplitudes, in comparison to the spill-over of the transmitted pulses and other distortions. In order to eliminate undesired signal fractions, the system has to be calibrated against free space in advance of the measurement.

Fig. 6 Typical reflection measurement curve
That calibration trace is subsequently subtracted from the measurements of the materials under test. Additionally the signal is digitally low-pass filtered with a FIR-filter. An example of the resulting waveform is shown in Fig. 6. The remaining procedure is the same as in the transmission measurement system. Again only changes in the waveforms caused by sample materials are processed. Principal components are again deployed for training an artificial neural network, which finally yields the moisture values.

3.3 Architecture of the Radial Basis Artificial Neural Network

The architecture of the applied radial basis artificial neural network (RBF-ANN) [6] is shown in Fig. 7. The network consists of one input layer and one output layer. The input layer is formed by radial basis function neurons (RBF’s), after which that type of network is named. In each of the \( R \) neurons of the first layer, the distance between the input vector and the weight factor \( IW_r \) is calculated. The result is multiplied with the bias vector \( b_r \), thus providing the input \( n \) for the RBF, defined by

\[
a_r = \exp(-n_r^2).
\]

Hence, values close to the centre of the RBF produce an output in the vicinity of one, while values with a great distance produce an output tending to zero. This means, that the neurons in the input layer produce an output signal corresponding the distance of the measured input vector and the weight of the particular input neuron. The sensitivity of the neurons is further influenced by the spread factor, which is varied during the training of the network, in order to determine the optimal network architecture. The output layer consist of \( R \) weight factors \( LW_r \) and a bias \( b_o \), which are summed up. Finally this summation is the input value to a purely linear output function, describing the moisture content of the bulk material.

Fig. 7 Architecture of an RBF-artificial neural network
3.4 Validation

The root mean square errors of calibration $\text{RMSE}_c$ and validation $\text{RMSE}_v$ are calculated in order to judge the quality of the estimation value of the moisture content [5]. Furthermore, the coefficient of determination $R^2$ is calculated, because both measurement setups under consideration have been developed successively and the conditions of the bulk material are not exactly the same in both trials. For this reason, the $R^2$ is a proper measure for comparison of both methods. The training of the network is monitored with the $\text{RMSE}_c$ and the $\text{RMSE}_v$. The latter is determined, using the method of full internal cross-validation. This means, that one particular measurement (represented by the vector $v_m$) is removed from the dataset, while the calibration is performed with the remaining dataset. After the calibration procedures, (also called “training” with respect to ANN’s) the system is tested with the previously removed vector $v_m$. This procedure is repeated for all measurements and the $\text{RMSE}_v$ is calculated. The $\text{RMSE}_c$ is calculated with all measurements included in the dataset, thus all measured curves are known to the network. The number of input neurons is increased during the training of the ANN, whereby the $\text{RMSE}_c$ decreases continuously while the $\text{RMSE}_v$ runs through a minimum before increasing again. This occurs due to an over-fitting of the network and the training is stopped at this point, what is called “early stopping”. After the determination of the optimum number of neurons, the spread factor is varied and the training is restarted, in order to find the optimal RBF-ANN.

4 Comparison of the Results

![Comparison of the Results](image)

Fig. 8 Results of the transmission (left) and reflection (right) measurement system

The results, calculated according to Section 0, are presented in Fig. 8. At the left, the results obtained by the transmission measurement are shown. The pentagrams represent the estimated versus the actual moisture content, derived from the calibration of the system including all measurements.
Table 1 Comparison of the results

<table>
<thead>
<tr>
<th>(a) Method</th>
<th>RMSE_c</th>
<th>RMSE_v</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>(i) Transmission system</td>
<td>1.6 %</td>
<td>2.1 %</td>
<td>95 %</td>
</tr>
<tr>
<td>(ii) Reflection system</td>
<td>1.1 %</td>
<td>3.0 %</td>
<td>90 %</td>
</tr>
</tbody>
</table>

From these values the $RMSE_c$ is calculated, which amounts to 1.6% of moisture content for the transmission setup. The triangles represent predicted moisture values which are calculated using the method of full internal cross validation. These values are the basis for the calculation of the $RMSE_v$. The $RMSE_v$ amounts to 2.1% of moisture content, yielding a coefficient of determination $R^2$ of 95% in this experiment. In the reflection measurement an $RMSE_c$ of 1.1% of moisture content can be obtained with an $RMSE_v$ of 3.0%. Based on the error of validation, the result is an $R^2$ of 90%. The results are displayed in Table 1. As mentioned in Section 0, the number of measurements $M$ for the reflection setup is somewhat higher as compared to the transmission system. That fact generally leads to an increase of accuracy because of the additional information in the calibration / training datasets.

In spite of the increased effort of calibration, the accuracy of the reflection measurement system is worse compared to the transmission system. One reason may be the lower amplitude of the measurement response. One has to take into account that the signal has to travel through the material twice, causing a higher attenuation in the material under test. Another reason is the decrease of the amplitude by a factor 10 caused by the applied kind of monitoring through a pickup-tee. Finally an increased number of multiple reflections take place between the antenna, the interface of the material under test and the ground plane. Thus the performance of the reflection setup deteriorates. Hence a transmission measurement has to be preferred in comparison to a reflection measurement, if the environmental conditions allow the realisation.

5 Conclusions

A comparison is presented for two approaches of measurement of the moisture content of bulk materials, which are present in layers with unknown layer width and density. The approaches are capable of delivering the moisture content independently from the layer width of the material under test. Ultra-wideband pulses are used for the measurements, in order to exploit the permittivity of the material over a wide range of frequencies. Artificial neural network are applied for the estimation of the moisture content. Input data are the pulse delays and sampled shapes of the transmission and the reflection pulses. The dimensionality of data of the input vectors representing the condition of the material are decreased in by calculating their most representative principal components. These are used as input for an artificial neural network.

Hardware setups for both systems are described, which are very similar in design. Although requiring a higher effort of calibration, the reflection system yields a lower accuracy compared to the transmission system. A further disadvantage is the requirement for the subtraction of a reference measurement. This may cause problems because of the potential drift over time of the acquisition system. Further modifications are necessary for the reflection setup, with respect to the monitoring of the reflected signals, in order to supersede the application of the pickup-tee. Furthermore the drift of the system has to be compensated, possibly through the digital data-processing.
References


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Hydrous Profile Modeling in Porous Materials
From Reflection Coefficient Measurements at 2.45 GHz

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ABSTRACT. Usual methods used to determine a hydrous profile are based on TDR or UWB techniques. In this paper we present a study devoted to the modeling of a hydrous profile in porous materials from the measurement of the reflection coefficient at a single frequency. In particular, in case of a multi-layer approach the reflection coefficient is shown to be not only a function of the characteristics of the individual layers but also of the arrangement considered.

Keywords: porous material, multi-layer material, hydrous profile, reflection coefficient

1 Introduction

In this paper we present a study devoted to the modeling of a hydrous profile in porous materials from the measurement of the reflection coefficient. Generally, the methods used for such a determination are based on TDR or UWB techniques [1, 2] (for instance moisture measurement of soils). In this study, we propose an alternative approach that makes use of a contact-less reflection coefficient measurement only at a single frequency (2.45 GHz). The concern of this paper is not that of the reconstruction of the profile but that of demonstrating the sensitivity of the proposed method to the water content distribution. To that end we first consider for the porous material a homogenization technique based on a mixing approach that gives an effective moisture content from the measurement of an effective permittivity. Then a method that consists of approximating the moist porous material to a multi-layer structure where each layer is homogenized is presented.

In addition to this investigation we want to see in which extent the loss of the spectral richness could be compensated by the determination of the reflection coefficient for different distances between the antenna and the material under test.

2 Homogenization Method of Porous Material

Generally, moist porous materials, which are complex structures, are modeled in a first approach as media with different phases as described in figure 1.

![Fig. 1 Structural approach for a moist porous material](image)

- air and moisture
- solid particles
- water
For this kind of materials we find typically three different areas: air and moisture, solid particles and water. So, to treat the problem of interaction between electromagnetic waves and such complex structures a homogenization approach is used and an effective permittivity of the system is defined. In fact, it has already been shown [3] that the permittivity of a moist material ($\varepsilon_{\text{moist}}^*$) can be written as a function (equation 1) of its dry permittivity ($\varepsilon_{\text{dry}}^*$) and density ($\rho_{\text{dry}}$), the water permittivity ($\varepsilon_{\text{water}}^*$) and its moisture content ($MC$):

$$\sqrt{\varepsilon_{\text{moist}}^*} = \sqrt{\varepsilon_{\text{dry}}^*} + MC \rho_{\text{dry}} (\sqrt{\varepsilon_{\text{water}}^*} - 1)$$  (1)

Note that $\rho_{\text{dry}}$ and $MC$ are respectively linked to the porosity of the material and to the degree of pore saturation. The measurement of the different permittivities leads to the determination of the effective moisture content according to the relation:

$$MC = \frac{\sqrt{\varepsilon_{\text{moist}}^*} - \sqrt{\varepsilon_{\text{dry}}^*}}{\rho_{\text{dry}} (\sqrt{\varepsilon_{\text{water}}^*} - 1)}$$  (2)

In order to have an idea of the water distribution in the material it is interesting to combine a multi-layer approach and a mixing law based model for each layer to predict the permittivity of the material under test against its moisture profile.

### 3 Measurement Technique

The technique proposed to determine the permittivity of a moist material is based on the measurement of the reflection coefficient at the frequency of 2.45 GHz. In the present approach the material is laid on a metal plate. The measurement procedure includes a calibration step.

#### 3.1 Calibration Procedure

To conduct the experiments a calibration step is needed. The measurement technique and the modeling approach associated to this procedure are depicted in figure 2.

![Fig. 2 Schematic diagram of the calibration measurement configuration](image)
The establishment of the flow graph of the structure where:

- $\Gamma_1, \Gamma_2,$ and $T_1, T_2$ are respectively the reflection and transmission coefficients that take into account the antenna and the space contained between the antenna and the plane located at a distance $L_{ref}$ chosen so that the far-field conditions are fulfilled,

- $t_0$ represents the transmission coefficient in the medium 0 (air),

permits to derive the reflection coefficient.

One can note that in the expression of $t_0$ appears the term $e^{-\gamma_0(L-L_{ref})}$ but also a term $\sqrt{\frac{L_{ref}}{L}}$ that is due to the free space losses.

The derivation results in the following expression of the reflection coefficient:

$$S_{11} = \Gamma_1 - \frac{T_1 T_2}{L L_{ref}} e^{2\gamma_0(L-L_{ref})} + \Gamma_2$$

(3)

An equivalent form of this formula, written slightly differently, can be found in the literature [4]. The findings of the present modeling approach (eq. 3) are given in figure 3 where we have also reported the results obtained by using the commercial software High Frequency Structure Simulator (HFSS) from Ansoft® to simulate the proposed structure.

![Graph](image)

**Fig. 3** Comparisons between the reflection coefficients collected against the distance $L$ when no material is laid on the plate (■ : HFSS simulations, —— : modeling)

This figure demonstrates the good behavior of the model.
3.2 Simulation of a Homogenized Material

If we consider that the moist material has been homogenized according to the method described in the section 2, the determination of the reflection coefficient can be derived from the flow graph given in figure 4.

Fig. 4 Schematic diagram of the homogenized material configuration measurement

- $r_{ij}$ and $t_{ij}$ define respectively the reflection and transmission coefficients at the interface air - material,
- $t_1$ represents the transmission coefficient in the medium 1 (material).

As one can see the distance $L$, through the term $\frac{L}{L + d_1}$, influences the transmission ($t_1$) in the material.

In these conditions the reflection coefficient is written as:

$$S_{11} = \Gamma_1 + \frac{T_1T_2S_{11\text{material}}}{T_0} - \Gamma_2S_{11\text{material}}$$

with

$$S_{11\text{material}} = r_{01} = \frac{t_0t_{10}}{L + d_1} e^{2\gamma_{d1}} + r_{10}$$

(4)

The material chosen to validate the model (eq. 4) is cellular concrete. The characteristics of the sample tested are: a thickness $d_1 = 50$ mm, a dry permittivity $\varepsilon_{\text{dry}}^* = 2.05 - 0.01j$ and density $\rho_{\text{dry}} = 0.53$. The sample is considered as uniformly moistened ($MC = 10\%$) with water at $20$ °C ($\varepsilon_{\text{water}}^* = 77 - 12j$). Therefore, the effective permittivity estimated from equation 1 is $\varepsilon_{\text{moist}}^* = 3.40 - 0.15j$.

The reflection coefficient calculated from equation 4 for different distances between the antenna and the material under test is collected to plot the figure 5. On the same graph, for comparison purposes, the results obtained by means of HFSS for the same configuration are reported.
Fig. 5 Comparisons between the reflection coefficients collected against the distance $L$ when a sample of cellular concrete ($d_1 = 50$ mm, $MC = 10\%$) is laid on the plate ( ■ : HFSS simulations, —— : modeling)

These graphs exhibit that the modeling approach gives a good fit to the HFSS data, especially for distances greater than 100 mm. This is due to the fact that for this kind of distances the far field conditions are fulfilled. This study demonstrates the possibility of predicting the reflection coefficient of a moist porous material through a relatively simple model that consists in approximating this medium by a homogenized material. The retrieval of the homogenized material permittivity is then achieved by inverting the equation 4.

3.3 Modeling of a Hydrous Profile

When the reconstruction of a hydrous profile is aimed the homogenization approach that leads to an effective value of the moisture content is no more suitable. So, to simulate a moisture profile a method that considers a stratified medium is used [5, 6]. In this approach each layer is then homogenized and the corresponding moisture content $MC_i$ ($i$ is the layer number, $i = 1$ for the upper layer) is determined according to the method described in the previous section.

In figure 6 is given a schematic diagram representing the multi-layer approximation and the corresponding flow graph.

In the flow graph description, the following parameters are mentioned:

- $\Gamma_1, \Gamma_2, T_1, T_2$, which are obtained by the calibration procedure (section 3.1),
- $r_{ij}$ and $t_{ij}$ which define respectively the reflection and transmission coefficients at the interface between media $i$ and $j$,
- $t_i$ which represents the transmission coefficient in the medium $i$ (0 for air, 1 for the upper layer, $n$ for the lower one). One can note that this parameter depends on the permittivity and the thickness of the layer but as it is shown in equation 4 it is also a function of its depth.
4 Results

In this part of the paper we show that, contrary to the homogenization approach that leads to an averaged moisture content, the multi-layer model permits to show the sensitivity of the reflection coefficient to the material moisture profile. The demonstration is made on simulation basis by considering a three layers material. Once again the material chosen for the tests is cellular concrete. In this three layers structure the blocks have the same physical characteristics (thickness, density and dry permittivity). We consider a constant amount of water distributed in a different manner to simulate several moisture profiles. Two situations are presented to illustrate the method: in the first one a porous material with an effective moisture content of 1\% is considered (mat 1) whereas a moisture content of 5\% is selected in the second example (mat 2).

For the measurement conditions chosen the effective permittivity of the material under test is given by the relation found in the mixing dielectric theory (complex refractive index) [7, 8]:

$$\sqrt{\varepsilon_{\text{homogenized}}} = \frac{1}{3} \sqrt{\varepsilon_{\text{moist} \ MC_1}} + \frac{1}{3} \sqrt{\varepsilon_{\text{moist} \ MC_2}} + \frac{1}{3} \sqrt{\varepsilon_{\text{moist} \ MC_3}}$$

Table 1 Examples of profiles for the material presenting an effective moisture content of 1\%
Table 2  Examples of profiles for the material presenting an effective moisture content of 5 %

<table>
<thead>
<tr>
<th>Moisture Content (%)</th>
<th>case 1</th>
<th>case 2</th>
<th>case 3</th>
<th>case 4</th>
<th>case 5</th>
<th>case 6</th>
<th>case 7</th>
</tr>
</thead>
<tbody>
<tr>
<td>MC₁</td>
<td>15</td>
<td>10</td>
<td>5</td>
<td>5</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>MC₂</td>
<td>0</td>
<td>5</td>
<td>10</td>
<td>5</td>
<td>10</td>
<td>5</td>
<td>0</td>
</tr>
<tr>
<td>MC₃</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>5</td>
<td>5</td>
<td>10</td>
<td>15</td>
</tr>
</tbody>
</table>

When the homogenization approach is considered, the effective permittivity found for the two materials are: \( \varepsilon^\text{homogenized (mat 1)} = 2.17 - 0.02j \) and \( \varepsilon^\text{homogenized (mat 2)} = 2.68 - 0.07j \).

To appreciate the sensitivity of the reflection coefficient to the moisture profile (case i) we have drawn respectively in figures 7 and 8 the difference observed, in term of reflection coefficient, between an homogenous distribution (case 4) and the other configurations for the materials noted mat 1 and mat 2. The simulations are made as a function of the distance between the antenna and the material under test.

Fig. 7 Difference between the homogenization approach and the multi-layer model in term of reflection coefficient determination for the mat 1

Fig. 8 Difference between the homogenization approach and the multi-layer model in term of reflection coefficient determination for the mat 2
These graphs demonstrate that the moisture profile has a relatively strong effect on the reflection coefficient of the material. The simulations conducted on the two sets of profiles displayed in tables 1 and 2 are illustrative of the two situations encountered when measuring a material with a particular moisture profile. Actually, one can not predict, a priori, which parameter (magnitude or phase of the reflection coefficient) will be the more sensitive, so a vector measurement is required. In addition, as depicted on figures 7 and 8 the results are not linearly ranged for a given distance $L$. Therefore, to solve the inverse problem the knowledge of both magnitude and phase is needed. These simulations also confirmed that the distance between the material under test and the antenna has a small but measurable effect on the reflection coefficient. Although not very sensitive, this parameter could be advantageously used in some cases to partially compensate the lack of information due to the monochromatic character of the method.

5 Conclusion

This study shows, through comparisons to the calculations made by means of HFSS, that the model proposed for the characterization of a homogenized material described correctly the evolution of the reflection coefficient against the distance antenna-material under test. In case of a moist porous material we have demonstrated that the water distribution in the material affects the reflection coefficient measurement. The subtraction of the results obtained by a homogenization approach to those derived from a multi-layer model have permitted to predict the effect caused by a given profile on the reflection coefficient. This simulation study will be supported, in future works, by an experimental investigation where the set-up will consist of a low-cost system, the SPMS – 2450 [3], associated to a patch antenna. The last step of the study is the resolution of the inverse problem to retrieve the moisture profile from the reflection coefficient measurement. This can be achieved by taking advantage of techniques based on artificial neural networks treatments.

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ABSTRACT. Current activities to regulate the emission of electromagnetic waves in the spectral band up to 10 GHz for sensor applications opens new perspectives for microwave moisture sensing. Therefore, improved and cost effective ultra wideband measurement principles become more and more of interest. The article deals with two variants of an M-Sequence approach which permit a bandwidth beyond 10 GHz. Different versions of data representation in the time and frequency domain will be shown by means of simple measurement examples.

Keywords: M-Sequence, ultra wideband, quadrature modulation, time domain reflectometry

1 Introduction

Moisture sensing by electromagnetic waves is undisputed an interesting measurement approach due to its non-destructive nature, easy handling, continuous and fast operation and further more. But it is also an indirect measurement method and by that it suffers from multifaceted cross sensibilities to unwanted physical parameters and phenomena. There is no way to completely prevent from these ambiguities. However as a general rule of thumb, one can reduce the ambiguities by collecting as much as versatile data from the material under test (MUT) and to gain the wanted information by applying these data to an appropriate model of the MUT. If one is restricted to exclusively electromagnetic sounding signals, the data versatility can only be achieved by measuring over a large frequency band.

Different substances or substance components are usually connected to a specific frequency behaviour. Thus, one can expect with some probability to be able to separate the water effects from superposed perturbation effects as long as the measurements were performed over a sufficient wide bandwidth. Furthermore, inhomogeneous material distributions and an irregular moisture profile provoke variations within the propagation path of the sounding wave which also strongly affect the frequency behaviour of the measurement data. Also here, wideband measurements can improve the measurement performance due to their ability of a high range resolution.

For practical applications, wideband measurements were often out of scope up to now because of the quite restrictive rules for radiation of electromagnetic waves and the need of sophisticated equipment. This situation is changing now:

- Currently the radiation rules are revised and adapted to today’s demand. The FCC, the US radio regulation authority, has been relaxed the radiation restrictions for a couple of years. It permits radiations to about 10 GHz with an EIRP\(^1\)-level of -41,3 dBm/MHz, excepted for the band from 1 to 3 GHz \[1\]. This frequency band is practically closed for sensor applications. In Europe and Asia, also some effort is made to find appropriate rules. The current situation in Europe is still confusing and difficult to predict. At the one hand, the
European regulation authorities insist on quite restricted power levels (perhaps 10 or 20 dB below the US limit) for large scale applications as the ultra-wideband communication (wireless USB etc.) in order to protect the existing communication systems against ultra-wideband interference. On the other hand, they appear to be more “friendly” concerning applications which involve operations under controlled conditions and a low activity factor. The activity factor indicates an average value of simultaneously active devices referred to a specified area (e.g. per km²). So it seems for example, that the ETSI\(^2\) will open the whole frequency band up to 12.6 GHz (without any frequency gap as in the US) for Ground Penetrating Radar (GPR) in the frame of trained professional use (controlled condition!) \[^2\]. Comparable circumstances as for GPR can be observed also for other sensor applications e.g. microwave moisture sensing. But up to now beside the GPR-rules, there was no effort undertake to prepare appropriate radio regulation rules. Recently however, the ETSI has been established a new task group TG31C, which is charged with corresponding topics. The new task group covers notable European enterprises.

- The classical wideband measurement device is the network analyser. It offers extremely flexible measurement conditions and provides a high resolution and precision. Thus, it is widely used for wideband moisture sensing. However its cost, weight, size and handling banish it to the laboratory. Referred to certain aspects, the classical time domain reflectometer (TDR) represents an interesting alternative. But often, the cost effective devices only have a low bandwidth and suffer from drift phenomena which unfavourably affect the data processing. The current developments in the ultra-wideband technique and circuit technology open however new perspectives of devices concepts, which can overcome the indicated inadequacies.

The goal of the article is to familiarise the reader with a new device approach, which offers the opportunity to perform the measurements over a large frequency band respectively to adapt the spectral band to the actual need. As mentioned above, the frequency band covered by the sounding signal is important for the information which can be extracted from the measurements. It should be noted, that due to the (in general) linear\(^3\) and time invariant nature of the considered MUTs the actual time shape of the sounding signals has no influence on the achievable information but rather on the technical implementation of the measurement device. This opens some flexibility in the device conception. The article will deal with ultra-wideband pseudo random codes, i.e. M-Sequences, as sounding signals. Their use enables a technical solution which is characterised by a highly integrated RF-part, a large bandwidth, a stable and drift free operation and a flexible operation due to the largely digital operation.

The basic idea of a M-Sequence device initially be intended for base band operations at e.g. 0 … 5 GHz is known from a couple of former publications, see [3]. In what follows, the current state of its development will be summarised and a measurement example will be demonstrated. Furthermore it will be shown how the operational spectrum of this device can be shifted to an arbitrary frequency band. The device concept involves an ultra-wideband IQ (inphase-quadrature phase)-down conversion, which results in the representation of complex valued time signals. A simple measurement example will illustrate what this means. The

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\(^2\) ETSI – European Telecommunications Standards Institute

\(^3\) Here, the term “linear” is considered concerning the behaviour of the MUT with respect to different sounding signals. In that sense, a system is linear if the law of superposition holds. That is, if the reaction of the MUT to a stimulus \(x(t)\) is given by the function \(y(x(t))\), than superposition implies \(y(x_1(t)) + y(x_2(t)) = y(x_1(t) + x_2(t))\). The consequence is, that linear transformations as the Fourier transform, convolution etc. are permitted to apply.
example refers to the frequency band from 3 to 10 GHz which is conform to the FCC radio regulation rules in the US.

2 The ultra wideband M-Sequence Baseband Module

For completeness and as starting point for the following consideration the basic approach of a M-Sequence (also called maximum length binary sequence) device will be shortly revised. The reader is addressed to [3] and [4] for details.

![Diagram of M-Sequence measurement head and photo of a device equipped with two receive channels and one stimulation port.](image)

Fig. 1 Basic structure of the M-Sequence measurement head and photo of a device equipped with two receive channels and one stimulation port. The impedance of all ports is either 50 Ω for grounded operation (coax cable) or 100 Ω for symmetric applicators. The data exchange and the operational control act via an Ethernet interface.

Fig. 1 shows the elementary structure of a wideband M-Sequence device and a practical implementation. The M-Sequence – the stimulus signal of the MUT – is generated by a digital shift register, which is pushed by a stable RF-clock $f_c$. The shift register, binary divider and the T&H-circuit are manufactured in a low cost SiGe-semiconductor technology. Current designs permit a clock rate up to 15 GHz. This results in a maximum usable bandwidth of about 7.5 GHz. One of the most important features of the M-Sequence approach is, that the actual sampling rate $f_s$ can be derived by a simple and stable way (i.e. by a binary divider) from the RF-master clock $f_c = 2^n f_s$ (see [4] for details). The digital signal processing can be freely adapted to the actual need, so that the provided data can be given in the frequency domain or the time domain (i.e. impulse or step response) or in some other form.

The baseband module provides signal energy from dc to $f_c/2$. Thus it can be profitably applied for TDR-moisture sensing since the probing cables do operate down to dc. Fig.2 summarises a TDR-experiment which involves symmetrically fed flat band cables. Two types were used:

- cable 1: conductor spacing 2,3 cm, flat conductors of 5 mm width, impedance in air 290 Ω;
- cable 2: conductor spacing 4,6 mm, round conductors of 0,8 mm diameter, impedance in air 240 Ω.

The length of both cables was 66 cm. However, only the central part of about 21 cm extension was surrounded by the MUT. The cables were left open at their end. The signal processing covers three parts: the correlation of the gathered data with a reference waveform in order to gain the impulse response function of the measurement arrangement; removal of systematic errors from the impulse response function and finally the temporal integration in order to gain
the desired step response function. The results clearly show the water effect on the cable impedance as well as on the propagation time. Furthermore a detailed analysis (not shown here) of the open end cable reflections also indicates the growing suppression of high frequency signal components by increasing the moisture content.

Fig. 2 Example of a TDR-measurement with cable 2 for moist sand.

3 The Ultra Wideband IQ-M-Sequence Approach

By adding an up-down-converter (see Fig. 3), the operational band of the M-Sequence head can be increased without turning to an improved (and thus more expensive) semi-conductor technology for the RF-components.

Fig. 3 Basic concept of a direct up- and down conversion via IQ-demodulation.

The key-components of the RF-front-end are commercially available double-balanced wideband mixers or Gilbert-cells. On the transmitter side, the spectrum of the original M-Sequence is shifted to a higher spectral band by mixing it with the carrier signal of frequency $f_{RF}$. This implicates that about 80 % of the stimulation energy is concentrated now in the frequency band $f_{RF} - f_c/2$ to $f_{RF} + f_c/2$. Thus, not only the spectral power is shifted to an arbitrary frequency band but also the usable bandwidth has been doubled compared to the baseband device.

On the receiver side, the captured signal is directly down-converted back to the base band by the carrier signal and then converted into the digital domain by the same approach as in the base band module. It should be remarked, that a simple direct down conversion causes some ambiguities due to the overlapping of spectral parts with positive and negative frequency.
This ambiguity can be overcome by introducing a second orthogonal channel. It is gained from a local signal which is delayed by a quarter period i.e. 90°. By doing that, one channel captures the parts of the measurement signal which are in phase with the carrier signal and the other one captures those which are in quadrature phase. This is a widely used principle, that is called IQ-demodulation.

The digital signal processing applied to the I- and Q-channel is quite the same as for the base band concept. That is, by subjecting the data to the Fast Hadamard Transform for example, one gets two impulse response functions $I(t)$ and $Q(t)$ characterising the behaviour of the test object. The usual approach is to joint both functions to a single, complex valued time domain function $Y(t)$

$$Y(t) = I(t) + jQ(t).$$

The function $Y(t)$ represents a kind of a complex envelope of the actual (real) system response $y(t)$, which is given by

$$y(t) = \Re\{Y(t) \exp(-j2\pi f_{RF} t)\}.$$  \hspace{1cm} (2)

A simple example shall illustrate the utility of a complex response function in the time domain. Let’s consider for simplicity a wideband delay line of variable delay as measurement object. The shape of its complex response $Y(t)$ will have a pulse form. The angle between the real and imaginary part is the same for the whole time. Thus $Y(t)$ lies in a flat plane as demonstrated in Fig.4. The inclination of the “pulse plane” depends from the actual delay time of the test object. That is, if the delay time will be continuously increased, the tip of the pulse will move along a screw thread track having a “flank lead” corresponding the wavelength of the carrier signal $f_{RF}$. This leads to the opportunity of precise delay time measurements. If additionally the transmission channel is subjected to a frequency depended behaviour, the pulse will get an arbitrary shape in the t-I-Q coordinates (see next paragraph).

4 Modified IQ-M-Sequence Measurement Head

The basic concept of an ultra wideband IQ-system as demonstrated in Fig. 3 is not yet very practical. First, it still demands too much components and second it is quite difficult (if not impossible) to build a precise IQ-demodulator which is working over a bandwidth of several GHz. The key-point of an IQ-demodulator is the identical behaviour of both sub-channels I and Q. This cannot be guaranteed if the bandwidth exceeds the GHz-range.

Therefore it is proposed to deal with a single down-conversion channel and to remove the ambiguity by capturing the I- and Q-data one after another. This can be done by introducing a switchable phase shifter (see Fig. 5). In an experimental sample, the clock rate $f_c$ of the shift register and the carrier frequency $f_{RF}$ were chosen identically and fixed to 7 GHz. This implies an operational band which well coincides with the FCC rules for UWB radiation.

Due to the large bandwidth, it is impossible to build the sub-components with nearly perfect behaviour i.e. flat frequency response, constant group delay, matched ports etc. This will provoke systematic measurement errors even if all system components are properly designed. The only way to reduce these errors is to perform a system calibration as it is common in the work with network analysers. The prerequisite for a successful calibration is a time stable measurement device and the availability of sufficient reference objects with know behaviour. As shown in [5], the M-Sequence approach is time stable by principle. Thus for many applications, an initial device calibration should be sufficient over a long running time. The number and type of calibration standards strongly depends on the error types which should be
eliminated and the required quality of correction. The wideband calibration of the IQ-down-converter is a challenging task since both signal paths are working in parallel and they don’t have identical parameters. In contrast to that, the calibration of the modified IQ-down-converter is a straight-forward procedure since the I- and Q-channel are identical by principle.

Fig. 4 The complex impulse response of a variable delay line

Fig. 5 Block schematics of the modified IQ-M-Sequence head respecting the FCC frequency mask. The order of the shift register and the binary divider was 9.

Fig. 6 demonstrates the results of a simple response calibration. For that purpose, the length of a mechanically variable delay line was increased by 400 µm increments. The bandwidth of the delay line was beyond that of the test signal. Thus one should expect the following behaviour if the measurement device would be perfect:

- Within the t-I-Q-coordinates, the complex impulse response function should lie in a flat plane which includes the time axis (compare Fig. 4).
- The shape of the function should be a short single impulse with FWHM ≈ 300 ps.
- The tip of the pulse should move along a circle in the complex plane by elongating the delay line. The angular spacing between two adjacent points should be 3.3°.

Fig. 6 shows that this is not the case in reality. The frequency response of the measurement chain is not flat and its group delay is not exactly constant which cause a looping waveform instead of an impulse waveform (Fig. 6 right). Furthermore, the phase switch does not work exactly between 0° and 90° thus the impulse tips does not form a circle (Fig. 6 left) but an ellipse. By the calibration, these effects are removed and the expected behaviour was gained.
Finally, a simple transmission experiment shall show the moisture effect onto the shape of the complex impulse response function (see Fig. 7). Sand of different moisture was filled in a plastic box and exposed by an electromagnetic field. Transmit and receive antennas were common double rigid horns. The sounding signal occupied the spectrum from 3.5 GHz to 10.5 GHz, its power was below 0 dBm. Both, the frequency response function and the complex impulse response function were gained from the captured data. The frequency response function has been shown the expected behaviour i.e. a growing attenuation by increasing the moisture content and the enhancement of the water influence at higher frequencies. The frequency response function was gained from the complex impulse response function by gating out signal components which were not caused from the test object e.g. multiple reflections.

Fig. 7 faces different impulse response functions. In order to enhance the dispersion effect, they are represented in the complex IQ-plane rather in t-I-Q-coordinates. That is, the time axis is standing perpendicular to the paper. Though the fundamental delay due to the moisture can not be seen by that representation, this effect is always known from common data representations (see Fig. 2). The antennas as well as the dry sand only show little dispersions, since their impulse response function are close to a straight line in the IQ-plane. The fundamental propagation delay between the antennas has been changed by adding the sand, indicated by the variation of the inclination angle and an overall time shift of the complete function (not to be seen in this representation). The remaining graphs refer to moist sand. The dispersion due to the water is in evidence. Increasing water content splits up the impulse response, provokes more and more loops and it reduces the magnitude of the response function.

5 Summary

Current radio regulation activities and new cost effective measurement principles will promote the application of ultra wideband devices for moisture sensing operating in the GHz range. The article presents two variants of an ultra wideband measurement device dealing with M-Sequence stimulation signals. A baseband principle was presented, which can operate as a TDR or a network analyser, so that the behaviour of the MUT can be described in the time as well as the frequency domain. Its test signal also contains spectral power at frequencies quite below 1 GHz, so it can be applied for moisture profile measurements over a long distance. A second principle – gained by adding an up-down converter - operates within a very wide spectral band centred at a carrier frequency. Here, the measurement results also
can be represented in the time and frequency domain. It was shown, that the use of a complex
time domain representation may be advantageous. Due to the high operational frequency
band, the device is mainly thought for investigation of small volume MUTs respectively
surfaces. The unambiguous derivation of a moisture value from wideband measurement data
under different experimental conditions still requires some investigations.

Fig. 7 Transmission experiment with moist sand.

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A Compact Network Analyzer for Resonant Microwave Sensors

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ABSTRACT. A measuring system for determining the moisture content in concrete buildings and masonry is presented. The system consists of a small and robust resonant microwave sensor and a compact low-cost network analyzer (NWA). First, the sensitivity and frequency range requirements for the analyzer are derived from the sensor design. Then, a suitable realization of the analyzer is discussed in detail. Finally, obtained measurement results of the system are presented.

Keywords: moisture, network analyzer, measuring system

1 Introduction

Within the collaborative research centre SFB 477 [1] of the German Science Foundation (DFG) microwave sensors for innovative online monitoring of concrete structures are being developed, tested and optimized. Within this frame, several sensors have been presented which allow determining the moisture content of buildings from the complex permittivity $\varepsilon_r$ in the microwave frequency range. For reliable statements about the moisture, a continuous readout of the sensor is indispensable. Due to high costs and vulnerability against harsh weather conditions common high precision laboratory equipment is generally not suitable for this task. Therefore a compact low-cost measurement unit is needed.

This paper briefly illustrates the characteristic properties of the utilized sensor and describes the resulting requirements on the measuring unit. These can be met by a scalar network analyzer, which allows the measurement of the reflection coefficient, i.e. the sensor response. The measurement data of several sensors is transmitted via a wireless link to a central web server from which it can be accessed and where parameters like the measurement rate can be controlled via the internet.

2 The Sensor Design

The required performance of the measuring unit regarding the frequency range and the sensitivity is defined by the properties of the sensor shown in a cross sectional view in Fig. 1 [2,3].

Fig. 1 Sensor design
The resonator consists of a circular waveguide containing a copper beryllium helix soldered to the prolonged centre conductor of a 0.250 inch semi rigid cable, which provides the interface to the measuring unit via a SMA connector. The utilisation of a helix as the main resonance frequency determining element allows the small overall size: the sensor exhibits a diameter of 10 mm and a length of 20 mm. The resonator is loaded by a sensitive material located in an open-ended circular waveguide. The suitable design of this section ensures below cut-off operation and thus prevents the electromagnetic wave from radiating through the aperture. The sensing mechanism is as follows: changing humidity in the measurement environment results in altered water content within the highly homogeneous sensitive material. This causes characteristic changes of the resonant response as can be seen in Figure 2. Here, the reflection coefficient \( r \) of the sensor is shown for a wet and a dry sensitive material. These measurements were performed with a high precision laboratory network analyzer (HP 8753D). From this graph, the required characteristics of the measuring unit can be derived: for a detection of the entire resonance shift, the frequency range from 2.3 GHz to 2.65 GHz has to be covered, whereas the dynamic range should reach from 0 dB down to the minimum reflection coefficient \( r_{min,sens} = -15 \) dB.

![Figure 2: Sensor reflection coefficient for a wet and a dry sensitive material.](image)

3 The Measuring Unit

3.1 General Aspects

Commonly networks are characterized by means of NWA at microwave frequencies. Generally, these allow the detection of scattering parameters for active, passive, linear and nonlinear networks with one, two or even more ports. If the measured network parameters are complex, the network analyzer is called vectorial in contrast to a scalar one, which only allows the detection of the magnitude. Since the magnitude of the reflection coefficient \( r \) is the relevant quantity of the resonant sensor, a linear and scalar network analysis for passive single port measurements is sufficient, here.
A basic narrowband network analyzer (see Fig. 3) without the need for internal frequency conversion consists of a signal source, a coupler for separating forward (a) and backward travelling (b) electromagnetic waves, a detector and a central control unit which drives the frequency sweep of the signal source, manages the analogue-to-digital conversion of the detected signal and organizes the data transmission to the central web server. General design parameters are a small overall size and low power consumption at a supply voltage of 5V combined with low-cost electronic devices and mounting techniques.

Fig. 3 Schematic of a network analyzer

3.2 The Control Unit

The complex task to control the high frequency source, the detector, and the data transmission can be done with a standard microcontroller. The used model is an AVR MEGA 8 from AT-MEL clocked with the same external 10 MHz signal used to reference the phased locked loop (PLL) (see Section 3.2). The controller is equipped with an In System Programmer (ISP), which allows easily updating the source code and controlling the high frequency source via a standardized data transmission protocol. Furthermore, implemented analogue-to-digital converters (ADC) allow 8-bit or 10-bit conversion of the detected electromagnetic signals and the environmental temperature. The chosen supply voltage of 5 V results in a current consumption of approximately 12 mA during operation and less than 50 µA in sleep mode.

3.3 The Signal Source

Stable frequency synthesizer with low noise performance usually includes a phase locked loop. A PLL is a feedback system combining a voltage controlled oscillator (VCO) driven by a phase comparator (PD) in such a manner that the VCO maintains a constant phase angle compared to an external oscillator. A simplified schematic of a PLL is shown in Figure 4.
In this design a high precision temperature compensated oscillator (TCXO) with a frequency of $f_{ref} = 10\text{ MHz}$ and an uncertainty of less than 3 ppm is compared to the output frequency of the VCO. The frequency $f_{\text{out}}$ is controlled by the reference register $R$ and the counter register $N$ of the PLL according to the following formula:

$$f_{\text{out}} = \frac{N}{R} f_{ref}.$$  \hspace{1cm} (1)

In the following, the reference register $R$ is set to a value of 40, resulting in a frequency increment of 250 kHz, determining the maximum frequency resolution. To cover the required frequency range from 2.3 GHz up to 2.65 GHz the $N$ register has to be set to values between 9200 and 10600, which is well below the maximum register value of $N_{\text{max}} = 2^{17}$. After the output of the VCO is locked to the desired frequency, this information is transmitted to the microcontroller which starts the conversion of the detected signals. The output power of the stabilized source is 4 dBm over the entire frequency range of interest.

To enable linear frequency sweeps as they are required for the evaluation of the sensor (see Fig. 2), the $N$ register needs to be updated permanently. Therefore, a PLL was used, which allows the setting of these registers via the standardized ISP protocol. The power consumption of this chip ($ADF\ 4112$ from ANALOG DEVICES) is 6.5 mA under operation and only 1 µA in idle mode.

The chosen VCO (JTOS-2700 from MINICIRCUITS) is capable of providing output frequencies from 2.3 GHz up to 2.7 GHz with a control voltage ranging from 1 V to 5V. Even at the highest frequency the VCO exhibits a current consumption of less than 20 mA. During sleep mode, the VCO is switched off by the microcontroller.

### 3.4 The Directional Coupler

The reflection coefficient is obtained from the ratio of the forward and the backward traveling wave powers $P_a$ and $P_b$, respectively (see Fig. 3 and Fig. 5):

$$r = 10 \cdot \log \left( \frac{P_b}{P_a} \right) \text{dB}.$$  \hspace{1cm} (2)

The separation of waves $a$ and $b$ is accomplished by directional couplers [4]. The common structure of such a device in microstrip technology is depicted in Fig. 5.
Fig. 5 The structure of a directional coupler connected to a source and a sensor.

Under matched conditions the performance of a directional coupler is mainly influenced by the length $L$ and the distance $s$ of the coupled lines and is characterized by three parameters: the coupling $C$, the isolation $I$ and the directivity $D$. The coupling is given by the ratio of the incident power at port 1 and the power transmitted to port 4 and is normally expressed in decibels:

$$C = 10 \cdot \log \left( \frac{P_1}{P_4} \right) \text{ dB}. \quad (3)$$

Port 3 is ideally decoupled from the signal source. The rate to which this is accomplished is described by the isolation $I$:

$$I = 10 \cdot \log \left( \frac{P_1}{P_3} \right) \text{ dB}. \quad (4)$$

Both these parameters are used to calculate the directivity $D$, which describes how well the power can be coupled into the desired direction.

$$D = I - C = 10 \cdot \log \left( \frac{P_1}{P_3} \right) \text{ dB}. \quad (5)$$

The overall coupler performance is furthermore influenced by reflections of the detector ($r_{det}$) and at the connector between the coupler and the semi-rigid-cable of the sensor ($r_{con}$) (see Fig. 5). This leads to an effective directivity $D_{eff}$.

The coupler was simulated and optimized using the Method of Moments solver integrated into the *Advanced Design System 2003* from *Agilent Technologies*. The optimization goal for the coupling and the directivity was set to 10 dB and 30 dB, respectively. The optimized structure was realized on high frequency substrate *RO 3210* from *Rogers Corporation*. Due to the high dielectric permittivity of $\varepsilon_r = 10.2$, the structure exhibits a final length of only 8 mm and a height of 2 mm. To minimize errors induced by reflections $r_{det}$ at the detector ports, a dual directional coupler was used, which consists of two of the directional couplers as depicted in Figure 5 arranged back to back with the isolated port terminated. The performance is reported in combination with the measurement results in Section 4.2.
3.5 The Detector

The used detector chip (ADF 8302 from ANALOG DEVICES) is specified for signals ranging from -60 dBm to 0 dBm and from low frequencies up to 2.7 GHz. It has two inputs for waves \( a \) and \( b \), respectively. At the output it provides a stabilized voltage \( V_{\text{ref}} = 1.8 \text{ V} \) and a voltage \( V_{\text{mag}} \) which is a measure of the reflection coefficient according to the formula

\[
\frac{r}{60dB} = \frac{V_{\text{mag}}}{V_{\text{ref}}} - 30dB .
\]

(8)

\( V_{\text{mag}} \) may vary between 0 V and \( V_{\text{ref}} \), allowing the detection of the reflection coefficient in a \( \pm 30 \text{ dB} \) range. These characteristics of the detector are consistent with the output power of the PLL, the minimum reflection coefficient of the sensor and the coupling \( C \).

\( V_{\text{mag}} \) is connected to a 10 bit ADC of the microcontroller, which is referenced to \( V_{\text{ref}} \). The converted result is stored in the EEPROM of the controller. The current consumption of the detector is approximately 20 mA during operation whereas in sleep mode it is switched off by the microcontroller.

3.6 The Wireless Link

The wireless link is implemented in the ISM band around 433 MHz. Both the remote and the base station comprise a complete transceiver including two antennas. The RX antenna is connected to an external interrupt of the microcontroller. At the sensor this enables the transition from sleep to operating mode. The TX antenna is simply connected to an I/O pin. At the base station the web server controls the transceiver via a RS 232 interface. In case of a measurement request from the web server to a specific sensor unit, all units wake up from sleep mode, compare the transmitted device identification number with their own and, if necessary, start the frequency sweep, store the reflection coefficient and transmit the measured data back to the web server.

Both antennas operate at a transistor stabilized voltage of 3.2 V. The current consumption is 10 mA.

3.7 The Design

To ensure a compact assembly, a multilayer design is used. An important design goal is the consequent separation of high and low frequency devices to reduce the usage of the expensive high frequency substrate RO 3210 to a minimum. Figure 6 shows a cross section of the schematic.

![Fig. 6 Schematic of the design with different layers and the corresponding devices.](image-url)
The actual system measures 5 cm x 4 cm x 2 cm and is shown in the following pictures. Since the transceiver partly covers the layout it is not depicted here.

![Fig. 7a Picture of the LF side of the NWA](image1)

![Fig. 7a Picture of the HF side of the NWA](image2)

The entire system exhibits a current consumption of 79 mA in operation and 12 mA in sleep mode.

### 4 Measurements

#### 4.1 Comparison with a Laboratory NWA

In Section 3.4 the directivity of the directional coupler was introduced as the parameter of major importance for accurate measurements. The effective directivity $D_{e\vartheta}$ is easily assessed by replacing the sensor with a termination ($r_{\text{term}} < -40$ dB) and performing a measurement. The resulting curve is shown in Fig. 8a. As can be seen, the directivity remains above 15 dB over the entire frequency range of interest. The difference to the simulated directivity of 30 dB is probably due to uncertainties during the manufacturing process and an unexpected high reflection at the interface between the substrate and the SMA connector. Despite of the reduced directivity the system is capable of reliably evaluating the resonance frequency as it will be shown in the next section.

To test the performance of the compact network analyzer, it is compared to a calibrated high precision laboratory device (HP 8753D). A sensor as depicted in Fig. 1 is used. The sensitive material was replaced by a PTFE cylinder, whose dielectric permittivity does not change with the environmental conditions and thus guarantees a maximum degree of reproducibility. To cover a broad frequency range and because of limited memory resources, only every 8th step of the PLL-$N$-divider was set, leading to a maximum resolution of 2 MHz. Fig. 8b shows the comparison of the measurement results.
As can be seen, the resonance curves differ only marginally. The resonance frequency found by the compact network analyzer is 2.558 GHz, which is 400 kHz below the one found by the HP 8753D. This is within the chosen resolution of 2 MHz of the compact network analyzer.

4.2 Long term measurements

To test the entire system, a long time measurement was performed with the sensor reported above. The sensitive material was an Al₂O₃ ceramic with a porosity of 50%. At time t = 0, the ceramic was completely soaked with water and then brought into an environment kept at 0% relative humidity.

Figure 9a shows the increase of the resonance frequency during the drying process of the sensitive material. The different slopes (44 MHz/h and 7 MHz/h) of the curve are interpreted as the successive drying of pores with different diameters. An increase of accuracy beyond the frequency resolution of 2 MHz can be achieved by fitting the measured resonance curves to a mathematical model of a resonator [5]. This fitting routine is especially useful if the resonance frequency exhibits only small slopes as it is the case in the time interval 12 h < t < 16 h (see Fig. 9a). The improved accuracy allows the determination of the resonance frequency with an optimized resolution as depicted in Figure 9b.

Figure 10 shows the complete system consisting of the sensor and the NWA compared to a 1 € cent coin.
5 Conclusion and Outlook

A measuring system for monitoring moisture of concrete buildings is presented. The system consists of a resonant sensor and a scalar network analyzer. It operates in the frequency range from 2.3 GHz to 2.65 GHz and is capable of detecting resonance frequencies with an uncertainty of 400 kHz. The minimum frequency resolution due to the hardware is 250 kHz and can be improved by the application of suitable fitting routines. The data transmission from the sensor to a central web server is implemented as a wireless link.

Future work will focus on improved directivity by appropriately matching the detector and on upgrading the scalar NWA to a compact vectorial one. This can be achieved by taking advantage of the detector’s ability to measure the phase of the reflection coefficient, and thus allowing the application of powerful calibration routines. Finally, the system is to be tested under realistic conditions.

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Session 6

Electromagnetic Sensors in Time- and Frequency Domain for Moisture Content Determination in Solids and Liquids
Chairmen: R. Knöchel, K. Khalid

1 Universal Microwave Moisture Sensor
   S. Trabelsi and S. O. Nelson; Athens, GA, USA

2 Dual Frequency Moisture Sensor Based on Circular Microstrip Antenna
   M. M. Ghrettli, K. Khalid, M. H. Sahri, I. V. Grozescu, and Z. Abbas; Selangor, Malaysia

3 Characterization and Comparative Evaluation of Novel Planar Electromagnetic Sensors
   C. Gooneratne, S.C.Mukhopadhyay, G. S. Gupta; Turitea; and K.P. Thakur; Auckland, New Zealand

4 Trough Guide Ring Resonator for Precision Microwave Moisture and Density Measurements
   R. Knöchel, W. Taute; Kiel; C. Döscher; Hamburg; Germany

5 Simple Soil Moisture Probe for Low-Cost Measurement Applications
   St. Schlaeger, Ch. Huebner, R. Becker; Karlsruhe, Germany
Universal Microwave Moisture Sensor

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ABSTRACT: A unified method for moisture sensing in grain and seed from measurement of their dielectric properties is presented. The method is based on a complex permittivity calibration function that is independent of both bulk density changes and the kind of material. The performance of the method was tested for soybeans, corn, wheat, sorghum and barley at 7 GHz and 24 °C.

Keywords: Dielectric properties, microwaves, bulk density, moisture content, unified moisture calibration function.

1 Introduction

Water is an important component of many biological and nonbiological materials. Classical methods for moisture content determination can be tedious and often require several hours if not days. In the late sixties, microwave techniques emerged as a promising alternative for rapid and nondestructive sensing of moisture, which made them suitable for on-line use. Microwave techniques rely on wave/material interaction and thus basically, in one way or the other, on correlations between the dielectric properties and moisture content. Because moisture content is not the only entity that affects the dielectric properties, there is a need to compensate or eliminate the effects of other factors [1]. It is obvious that eliminating these effects represents technical and economical advantages in developing microwave moisture sensors.

Granular and particulate materials constitute a large category of organic and inorganic materials that are encountered in diverse industries including food and agriculture, pharmaceutical, mining and construction. Rapid characterization of these materials is important for process and quality control. Moisture sensing in these materials requires compensation for or minimization of bulk density effects. Therefore, calibration functions [2-6] for moisture determination independent of bulk density changes were developed and have been used successfully in many industries. However, there remains the need for distinct calibration for each granular material. This, along with cost concerns, discouraged commercial development of microwave moisture sensors although this technology has many advantages [7]. Small-size and light-weight microwave components are now available at affordable prices, which make microwave sensing technology competitive and attractive, particularly with the growing need for real-time and nondestructive characterization of materials in highly automated and computerized industries. Furthermore, the problem of distinct calibration for each granular material can be solved by defining a single calibration equation that provides moisture content with acceptable accuracy for different materials.

In this paper, a unified microwave calibration method that provides moisture content in cereal grain and oilseed with significant structural and compositional differences is presented. The concept is tested at a single microwave frequency, because this would simplify the design and
reduce the cost of a sensor based on this method. Results are shown at 7 GHz and 24 °C for soybeans, corn, wheat, sorghum, and barley.

2 Permittivity-based Unified Moisture Calibration Function

Both the dielectric constant, \( \varepsilon' \), and loss factor, \( \varepsilon'' \), of grain and seed are dependent on bulk density, moisture content and temperature at a given microwave frequency [8, 9]. Therefore, moisture determination from dielectric properties requires compensation for bulk density and temperature effects. Also, there is a need for establishing an individual moisture calibration for each material. In recent years, a permittivity-based calibration function that is density-and material-independent was defined [10]. It is expressed in terms of \( \varepsilon' \) and \( \varepsilon'' \) as:

\[
\psi = \frac{\varepsilon''}{\varepsilon' \left( a_f \varepsilon' - \varepsilon'' \right)}
\]

where \( a_f \) is the slope in the complex plane of the straight line representing the loss factor divided by bulk density, \( \rho \), as a function of the dielectric constant divided by bulk density. The slope \( a_f \) was found to be dependent on frequency only [6, 11]. The dielectric properties of each material used in this paper were measured in free space with a pair of horn/lens antennas. Special precautions were taken for accurate determination of \( \varepsilon' \) and \( \varepsilon'' \) [12]. Figure 1 shows the complex plane representation for soybeans, corn, wheat, sorghum and barley at 7 GHz and 24 °C. Each material has a distinct straight line with an x-axis intercept, \( k \), corresponding to the material with zero moisture content or any moisture at very low temperature [6]. Data corresponding to each material can be fitted by a linear regression of the form:

\[
\frac{\varepsilon'}{\rho} = a_f \left( \frac{\varepsilon'}{\rho} - k \right)
\]

Table 1 summarizes regression statistics.

![Complex-plane representation of the dielectric constant and loss factor divided by bulk density for soybeans, corn, wheat, sorghum, and barley at 7 GHz and 24 °C.](image)
Table 1 Regression statistics corresponding to equation (2) for soybeans, corn, wheat, sorghum and barley.

<table>
<thead>
<tr>
<th>Material</th>
<th>$\rho$, g/cm$^3$</th>
<th>$M$, %</th>
<th>$a_f$</th>
<th>$k$</th>
<th>$r^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soybeans</td>
<td>0.71-0.82</td>
<td>9.8-20.3</td>
<td>0.623</td>
<td>2.89</td>
<td>0.995</td>
</tr>
<tr>
<td>Corn</td>
<td>0.71-0.87</td>
<td>10.8-20.4</td>
<td>0.606</td>
<td>2.62</td>
<td>0.988</td>
</tr>
<tr>
<td>Wheat</td>
<td>0.75-0.89</td>
<td>9.9-18.0</td>
<td>0.543</td>
<td>2.58</td>
<td>0.957</td>
</tr>
<tr>
<td>Sorghum</td>
<td>0.74-0.89</td>
<td>10.1-21.2</td>
<td>0.532</td>
<td>2.58</td>
<td>0.983</td>
</tr>
<tr>
<td>Barley</td>
<td>0.60-0.71</td>
<td>9.7-24.8</td>
<td>0.459</td>
<td>2.64</td>
<td>0.982</td>
</tr>
</tbody>
</table>

The x-axis intercepts of cereal grains (corn, wheat, sorghum and barley) are nearly the same.

Figure 2 shows variation of the density- and material-independent function $\psi$ with moisture content in soybeans, corn, wheat, sorghum and barley at 7 GHz and 24 °C. The data corresponding to cereal grain and seed are superposed and can be fitted by a single linear regression:

$$\psi = 0.017 \cdot M + 0.104 \quad r^2 = 0.955$$

(3)

where $M$ is the wet-basis moisture content [13]. From equation (3) a single moisture calibration equation can be established for materials presenting significant structural and compositional differences. This equation is:

$$M = \frac{\psi - 0.104}{0.017}$$

(4)

The standard error of calibration for moisture prediction in soybeans, corn, wheat, sorghum and barley with equation (4) is 0.81%.

![Fig.2 Variation of permittivity-based calibration function $\psi$ with moisture content in soybeans, corn, wheat, sorghum, and barley at 7 GHz and 24 °C.](image)
3 Conclusions

A unified method for moisture sensing in cereal grain and seed was proposed. Results shown here for soybeans, corn, wheat, sorghum, and barely at 7 GHz and 24 °C indicate that moisture content can be predicted in all materials from a single calibration equation with a standard error of calibration of about 0.8%. The method shows promise of development of a universal microwave moisture meter operating at a single frequency and suitable for routine, real-time sensing of moisture content in granular and particulate materials.

References


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Dual Frequency Moisture Sensor Based on Circular Microstrip Antenna

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ABSTRACT. A dual frequency sensor was developed to measure moisture content, MC of rubber latex. The experiment is based on measurements of near field reflection at two frequencies in the X-band, 8.48 and 10.69 GHz. The replacement of the conventional open horn antenna with microstrip radiating patches will make the sensor more versatile and compact. A calibration equation was found that instantly gives moisture content of the samples under consideration. The sensor interfaces with a laptop PC using a graphical language, LabView® which helped analyzes the data. Error correction and three calibration techniques were implemented and compared. The system is tested using rubber latex and has predicted MC with standard error less than ± 0.4% compared to standard oven drying techniques.

Keywords: Microstrip antenna, rubber latex, moisture content, dual microwave frequency.

1 Introduction

In the last few decades, microwave dielectric-based sensors were developed for sensing moisture in different materials (Kraszewski, 1996). The fact that these sensors rely on measurement of dielectric properties, which are also dependent on other physical properties of the material i.e. bulk density, moisture content, temperature etc, makes them potential multi-parameter sensors provided that appropriate correlations are established between measured dielectric and physical properties (Nykors and Vainikainen, 1989). In this paper, we deviate from the open-ended coaxial line as a common sensor for permittivity measurements. An alternative approach is introduced here based on disk patch microstrip antenna operating at two different operating frequencies in the X-Band 8.48 and 10.69 GHz. It is based on measuring reflection of the mature fields in near-field region from the sample interface. This new sensor is developed for special application of determining water content of liquids and food products easily and efficiently through the use of pre-determined calibration equation without the need for expensive vector network analyzers. The sensor was tested with hevea rubber latex.

2 Design of the Sensor

The first step was the design of each single frequency circular patch antenna and trying to optimize its parameters. Other than using full wave analysis, the circular patch antenna can be analyzed conveniently using the cavity model (Balanis, 1971). By using cylindrical coordinates, the cavity can be considered as two perfect electric conductors at the top and bottom to represent the patch and the ground plane. The dielectric material substrate with height $h$ and dielectric constant $\varepsilon_r$, is truncated beyond the extent of the patch. The radius $a$ does not take into account fringing fields. Fringing makes the patch look electrically larger (Bahl and Bhartia, 1980) and for circular patch a correction introduced by using an effective radius $a_e$ to replace the actual radius $a$: 

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\[ a_e = a \left\{ 1 + \frac{2h}{\pi \varepsilon_r} \left[ \ln \left( \frac{\pi a_e}{2h} \right) + 1.7726 \right] \right\}^{1/2} \]  

(1)

Where \( h \) and \( \varepsilon_r \) are the thickness and the dielectric constant of the patch substrate.

A numerical technique, modified regula falsi method was used to solve for the actual radius in equation (1). Two of the patches were connected to Dielectric Resonator Oscillators; DRO’s to make the transmitting antennas. And the other two were connected to broadband coaxial detectors with sensitivity of 500mV/1mW as shown in the schematic figure 1.

Fig. 1 The Schematic Diagram for the Sensor Patch with Connectors.

After careful consideration of separation and orientation of the disks to minimize the mutual coupling between the patches, E-plane orientation was chosen to minimize surface-wave coupling while keeping the edge separation to minimum.

Figure 2 shows a digital photo of the top and bottom view of the double transceiver sensor patch.

Fig. 2 A photo of Top and Bottom View of the Patch Sensor.

The whole sensor system is interfaced to a laptop PC through the use of data acquisition card, DAQ 6024E. Two electronic boards integrated to the system were designed. One served some functions like supplying a stable 7.0 volts to the oscillators and protecting them from a
voltage over surge. The second used for amplifying and conditioning the output signal in order to be fed to data acquisition card.

3 Sample Preparation and Procedure

The sensor was tested on samples of diluted concentrated rubber latex. The samples were divided into two groups. The first group was used to calibrate the sensor system. This group consists of 12 diluted solution samples with moisture content ranging from 38.7 % to 95.0 %. The steps of calibration are as follows:

- Turning on the oscillators by relays for 2 minutes warm up time and calibrating the sensor with two standards open air, and distilled water.
- Measuring the reflected power from the sample interface for each diluted solution sample.
- Normalization of the reflected signals to that of distilled water $\Gamma_i / \Gamma_w$ for each frequency and fitting the results into a 3-th order polynomial, equation (2) and calculating the correlation factor. This is done for each operating frequency.

$$MC_i = \alpha_0 + \alpha_1 \Gamma_i^1 + \alpha_2 \Gamma_i^2 + \alpha_3 \Gamma_i^3$$  \hspace{1cm} (2)

The second group is used to test the sensor and the regression. This group consists of twelve sample solutions with random moisture content percentage. After warming up the oscillators and measuring the two standards as instructed by the software, air and distilled water, the holder was filled to the specified level with the sample solution. The reflected signal was sampled at a rate of 1000 s$^{-1}$ for a total of $N = 10,000$ points. Leveling the liquid sample in the holder is crucial as demonstrated in (Khalid, 1988) and (Daschner and Knochel, 2003). The software will instantly display the moisture content based on the two standards measurements and reflection from the sample.

4 Results and Discussion

4.1 Distance Optimization and Warm-Up

The distance from the sample interface i.e. the bottom of the sample holder to the radiating antennas should be optimized for maximum reflected power as shown in figure 3.

![Optimizing Distance between Sample Interface and Antenna](image)

Fig. 3 Optimizing Distance between Sample Interface and Antenna.
The position of first maxima, corresponding to constructive interference of the standing waves, was chosen as the optimum distance between the sample interface and the antennas. For good reproducible measurements, enough time, should be allowed for warming up of the oscillators and the supplying circuitry as shown in figure 4. Failing to do so will result erroneous readings and the accuracy is significantly reduced.

![Graph](image)

Fig. 4 Detected Voltages at Both Receiving Patches as The Oscillators Warm-up.

4.2 Independent Calibration for Each Frequency

The true values of $MC\%$ for the 12 calibration sample solutions were obtained using the formula:

$$MC\% = \frac{m_w - m_d}{m_w} \times 100\%$$  \hspace{1cm} (3)

where $m_w$ is the mass of wet material and $m_d$ is the mass of dry material.

Standard moisture content values were measured and listed in the first and fourth rows of table 1, by weighing the samples before and after 24 hours drying using standard oven technique.

Table 1 True Moisture Content with Normalized Reflected Signals.

<table>
<thead>
<tr>
<th>MC $\pm 0.05$</th>
<th>38.71</th>
<th>43.99</th>
<th>48.98</th>
<th>54.10</th>
<th>59.21</th>
<th>64.37</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\Gamma 1$</td>
<td>0.46651</td>
<td>0.52172</td>
<td>0.56537</td>
<td>0.61859</td>
<td>0.69170</td>
<td>0.73208</td>
</tr>
<tr>
<td>$\Gamma 2$</td>
<td>0.78046</td>
<td>0.80474</td>
<td>0.82924</td>
<td>0.85363</td>
<td>0.87327</td>
<td>0.90231</td>
</tr>
<tr>
<td>MC $\pm 0.05$</td>
<td>69.46</td>
<td>74.52</td>
<td>79.58</td>
<td>84.73</td>
<td>90.02</td>
<td>95.02</td>
</tr>
<tr>
<td>$\Gamma 1$</td>
<td>0.78049</td>
<td>0.82390</td>
<td>0.86908</td>
<td>0.90349</td>
<td>0.91459</td>
<td>0.95681</td>
</tr>
<tr>
<td>$\Gamma 2$</td>
<td>0.91858</td>
<td>0.92688</td>
<td>0.95117</td>
<td>0.96178</td>
<td>0.97112</td>
<td>0.99702</td>
</tr>
</tbody>
</table>

Reflections from each frequency are calibrated with true $MC\%$ values and used to evaluate the four unknown constants of equation (2) using third order polynomial regression analysis. We get two independent calibration equations (4) and (5):

$$MC_l \% = 344.5061 \Gamma^3 - 656.0488 \Gamma^2 + 505.0197 \Gamma - 89.29337 \text{ at } f_1$$  \hspace{1cm} (4)
\[ MC_2 \% = 513.5249 \Gamma^3 - 964.2386 \Gamma^2 + 751.8770 \Gamma - 204.5638 \text{ at } f_2 \] (5)

With determination coefficients, \( R^2 \) of 99.896 and 99.750, and standard errors of calibration, SEC 0.624 \% and 0.967 \% moisture content respectively. Figure 5 shows the calibration points used to derive calibration polynomial, equations (4) and (5).

![Calibration Curve](image)

Fig. 5 Calibration Curve for MC \% of Diluted Hevea Rubber Latex at 8.48 and 10.69 GHz.

Validity of the calibration equations (4 and 5) was checked with another twelve sample solutions. Using the normalized detected signals in calibration equations, the moisture content were evaluated and listed in table 2. These values were compared with the values obtained from oven drying technique; equation (3).

![Verification](image)

The results are summarized in table 2. The quality of the verification can be evaluated by the standard error of performance, which is a standard deviation of differences between predicted and standard values. The standard errors of performance, SEP were 0.47\% and 0.40\% moisture content at \( f_1 \) and \( f_2 \) respectively.

![Regression](image)

If we plot the \( MC \) contents at each frequency against true \( MC \) in the same graph, figure 6, we see that each has a straight line with tangent equals to unity as expected from the high \( R^2 \) value for each regression.

![Plot](image)

Fig. 6 A Plot of Predicted MC from Calibration Equations at Two Frequencies and Weighted Method Against True MC Obtained Using Oven Method.
4.3 The Average Method Prediction

The predicted moisture content calculated can be improved slightly using the average of \( MC_1 \) and \( MC_2 \) values predicted from calibration equations (4) and (5) derived earlier.

\[
MC_{\text{ave}} \% = \frac{(MC_1 \% + MC_2 \%)}{2.0}
\]  

(6)

There is a slight improvement in prediction of \( MC \) using eq. (6) in the range from 40% to 95. This improvement can be seen by the decrease in SEP to \( \pm 0.47\% \) in table 2. The high variance of calibration data at \( f_2 \) limits the accuracy of the simple averaging technique.

4.4 Weighted Average Method Prediction

The accuracy is further improved by finding appropriate weighing functions \( \varphi_1 \) and \( \varphi_2 \) to incorporate signals from both frequencies such that the average moisture content \( MC \% \) is given by

\[
MC_{\text{weighted}} \% = \varphi_1 MC\%_{f_1} + \varphi_2 MC\%_{f_2} ; \quad \varphi_1 + \varphi_2 = 1
\]  

(7)

The weight functions \( \varphi_1 \) and \( \varphi_2 \) are calculated from the standard deviation of population of reflection measurement \( \Gamma \), which is the mean of \( N = 10,000 \) sample points collected at the rate of 1000 samples per second.

\[
\varphi_1 = \frac{\sigma_{f_2}}{\sigma_{f_1} + \sigma_{f_2}} \quad \text{and} \quad \varphi_2 = \frac{\sigma_{f_1}}{\sigma_{f_1} + \sigma_{f_2}} ; \quad \sigma_1 = \sqrt{\frac{\sum_{i=1}^{N} (\Gamma_{ij} - \overline{\Gamma})^2}{N-1}}
\]  

(8)

Using equation (7) instead of the simple average equation (6) ensures that the higher spread of the standard error, the less weight it is given in the calculation of the final \( MC\% \) value. Table 2 shows that the predicted \( MC\% \) values were compared well with the corresponding standard oven method values. The straight line with a slope of unity in Figure 6 clearly shows closeness of prediction with true \( MC\% \).

**Table 2 Predicted Moisture Content for Validation Group.**

<table>
<thead>
<tr>
<th>TRUE MC% ± 0.05%</th>
<th>Normalized ( \Gamma_1 )</th>
<th>Normalized ( \Gamma_2 )</th>
<th>Predicted ( MC_1 )</th>
<th>Predicted ( MC_2 )</th>
<th>Predicted ( MC_{\text{average}} )</th>
<th>Predicted ( MC_{\text{weighted}} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>38.90</td>
<td>0.47838</td>
<td>0.78196</td>
<td>38.60</td>
<td>39.28</td>
<td>38.94</td>
<td>38.72</td>
</tr>
<tr>
<td>44.16</td>
<td>0.54315</td>
<td>0.81158</td>
<td>44.73</td>
<td>43.99</td>
<td>44.36</td>
<td>44.42</td>
</tr>
<tr>
<td>49.09</td>
<td>0.58889</td>
<td>0.83323</td>
<td>49.25</td>
<td>48.27</td>
<td>48.76</td>
<td>49.14</td>
</tr>
<tr>
<td>54.21</td>
<td>0.64010</td>
<td>0.85859</td>
<td>54.50</td>
<td>54.07</td>
<td>54.28</td>
<td>54.29</td>
</tr>
<tr>
<td>59.25</td>
<td>0.67210</td>
<td>0.87888</td>
<td>57.89</td>
<td>59.26</td>
<td>58.57</td>
<td>58.93</td>
</tr>
<tr>
<td>64.36</td>
<td>0.73377</td>
<td>0.90142</td>
<td>64.65</td>
<td>65.54</td>
<td>65.09</td>
<td>65.16</td>
</tr>
<tr>
<td>69.52</td>
<td>0.78449</td>
<td>0.91678</td>
<td>70.46</td>
<td>70.09</td>
<td>70.27</td>
<td>70.37</td>
</tr>
<tr>
<td>74.64</td>
<td>0.81748</td>
<td>0.93201</td>
<td>74.35</td>
<td>74.79</td>
<td>74.57</td>
<td>74.43</td>
</tr>
<tr>
<td>79.71</td>
<td>0.85635</td>
<td>0.94862</td>
<td>79.07</td>
<td>80.11</td>
<td>79.59</td>
<td>79.40</td>
</tr>
<tr>
<td>84.95</td>
<td>0.90433</td>
<td>0.95833</td>
<td>85.08</td>
<td>83.31</td>
<td>84.20</td>
<td>84.76</td>
</tr>
<tr>
<td>89.94</td>
<td>0.94689</td>
<td>0.97517</td>
<td>90.60</td>
<td>88.97</td>
<td>89.79</td>
<td>89.78</td>
</tr>
<tr>
<td>94.95</td>
<td>0.97616</td>
<td>0.99564</td>
<td>94.50</td>
<td>96.02</td>
<td>95.26</td>
<td>94.97</td>
</tr>
</tbody>
</table>

SEP = 0.64, 0.83, 0.47, 0.40
Improvements in the predicted values were accomplished, by adjusting the weights of equation (8). In other words, recalculating the predicted values using adaptive averaging is done by adjusting the weights depending on the spread of the $N$ points collected for each reflection measurement at $f_1$ and $f_2$. Using this adaptive predication, $MC_{\text{weighted}}$ of rubber solution is obtained with standard errors less than $\pm 0.40\%$ of true $MC\%$.

5 Conclusion

Reflection measurements at frequencies 8.48 and 10.69 GHz, using diluted Hevea rubber latex placed at optimized distance from radiating microstrip antennas were used to determine the moisture content of the samples rapidly and nondestructively. A predication of $MC$s for the samples tested were obtained with standard errors less than $\pm 0.40\%$ of true $MC\%$ in the range from 38.7\% to 95.0\% using weighted average technique.

With the standard error of the mean $MC$ is less than $\pm 0.40\%$, the sensor is very versatile and can be made conformal in production processes due to the flat shape of Microstrip antenna. Using this method, it might be possible to construct accurate and rapid nondestructive moisture sensors of other similar products i.e. fresh milk, natural latex, palm oil… etc.

References


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Characterization and Comparative Evaluation of Novel Planar Electromagnetic Sensors

C.P. Gooneratne, S.C.Mukhopadhyay, G. Sen Gupta and K. Thakur

Abstract. The characterization of three types of novel planar electromagnetic sensors, (i) meander, (ii) mesh and (iii) interdigital configuration, have been studied and their comparative performance have been evaluated based on their areas of applications. All of them are suitable for inspection and evaluation of system properties without destroying them. The experiments on fabricated sensors have been conducted and results are presented here.

Keywords: Planar electromagnetic sensors, Meander, Mesh and Interdigital type, NonDestructive evaluation, pork meat.

1 Introduction

Nondestructive evaluation techniques are able to detect the presence of cracks, discontinuities, mechanical fatigue and so many other imperfections without material damage results in application of this techniques has increased considerably in recent times. It has been demonstrated that the mechanical stress has the ability to change the electrical properties [1]. So the change of electrical property such as electrical conductivity can be used as an index of mechanical fatigue. The planar meander type magnetic sensor has been used for the inspection of defects in the printed circuit [2]. The characterization of planar type mesh sensor has been reported [3, 4] which can be used in many areas and can overcome the directional problem of meander type. The sensitivity of meander and mesh type sensors to dielectric materials can be overcome by another planar sensor of interdigital configuration. The characterization and comparative performance evaluation of all sensors has been carried out.

2 Research Works Employing Planar Type Sensors

Research works employing planar meander type sensors as shown in figure 1, started quite a few years back with the target of development a complete inspection system of the printed circuit board (pcb) of pentium processor [2]. The exciting coil is of meander configuration whereas the sensing coil is either of mesh type or figure-of-eight type configuration. The pcb of pentium processor having many long conductors, the meander configured exciting coil is a suitable choice.

The planar meander type sensor has also been used for the inspection of material defects such as the existence of inner layer cracks and for the estimation of fatigue of metal products [5]. A crack with alignment in parallel with the exciting meander coil is difficult to be detected by meander configuration. The alternative is to employ mesh type sensors as shown in figure 2 [3, 4]. Both the meander and mesh type planar electromagnetic sensors have been employed for the quality inspection of food products such as milk, butter, cheese etc. and response is moderate. In order to increase the sensitivity of the sensor system another type of sensor, the interdigital one as shown in figure 3 has been fabricated and developed. Experiments have been conducted on all sensors and results are reported.
Fig. 1 Fabricated meander type planar electromagnetic sensors (4 types)

Fig. 2 Fabricated mesh type planar electromagnetic sensors (4 types)

Fig. 3 Fabricated planar interdigital type electromagnetic sensor

3 Characterization of Planar Sensors

Both the meander and mesh type sensors consist of two coils: one used for excitation and another for sensing. The high frequency alternating supply is provided to the exciting coil and the voltage across the sensing coil is measured. The transfer impedance (the ratio of the sensing voltage to the exciting current) is used as the characterization parameter for the meander and mesh type sensors. In case of interdigital sensor the exciting voltage and the displacement current through the sensor is measured. The impedance (the ratio of the applied voltage to the current) is the characterization parameter used for the interdigital sensor. The sensors are characterized with the help of an experimental set-up as shown in figure 4.
Different sizes of sensors of each meander, mesh and interdigital types have been experimented using the experimental set-up. The frequency of excitation has been varied between 100 kHz to 100 MHz. The impedance characteristics of the sensors are shown in figures 5, 6 and 7 respectively. It is seen that the transfer impedance for both meander and mesh type sensors increases with the increase in frequency whereas the impedance of interdigital type sensor decreases with frequency. With the same effective area the response of mesh type sensors is better than meander type. Basically meander and mesh types sensors are inductive type whereas the interdigital one is capacitive type. It is also seen that both meander and mesh type sensors response well at high frequency whereas the response of the interdigital sensor is very good at low frequency and doesn’t response well at high frequency. The selection of operating frequency is to be carefully selected.
4 Experimental Results and Comparative Evaluation

Table 1 shows the experimental results for all the sensors at an operating frequency of 84 MHz. Only the change of magnitude of impedance is shown here (the impedance with air is considered as unity), in practice the phase information is also used. It is seen from Table 1 that both meander and mesh type sensors respond very well to conducting and magnetic material but not so well to dielectric materials. On the other hand the interdigital sensor responds very well to dielectric materials. The experiment has been conducted by mixing cream with water to change the percentage of fat content and the actual experimental values obtained for all three types of sensors are shown in figure 8. It is seen the response of the interdigital sensor is very distinct. The change of transfer impedances for meander and mesh type sensor is not significant. The operating frequency is kept at 500 kHz. One of our application is to make a sensing system to estimate the fat content in the meat of pork.

Table 1 Comparison of results

<table>
<thead>
<tr>
<th>System Under Inspection</th>
<th>Meander</th>
<th>Mesh</th>
<th>Interdigital</th>
</tr>
</thead>
<tbody>
<tr>
<td>Air</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Copper</td>
<td>0.9127349</td>
<td>0.8764259</td>
<td>1.0151976</td>
</tr>
<tr>
<td>Aluminium</td>
<td>0.9118998</td>
<td>0.8669202</td>
<td>1.0151976</td>
</tr>
<tr>
<td>Iron</td>
<td>0.9077244</td>
<td>0.7742395</td>
<td>0.9984802</td>
</tr>
<tr>
<td>Milk</td>
<td>1.0205952</td>
<td>0.9697908</td>
<td>0.9022801</td>
</tr>
<tr>
<td>Water</td>
<td>1.0188946</td>
<td>0.9733264</td>
<td>0.9251887</td>
</tr>
</tbody>
</table>

Fig. 8 Change of impedance of different sensors with fat content in milk
There were mainly four main varieties

- Fat
- Mixed
- Muscle
- Skin

The sensor was tested on 4 pieces of fat, 2 pieces of mixed meat, 4 pieces of muscle and 1 piece of skin. One piece of each type is shown in Fig. 9. All the pieces were of different shape and size. The experiment involved analyzing the sensor results, to see if the different amount of fat content affected the original signal.

![Fig. 9 Pork samples for test](image)

Each piece was tested separately, and the sensor was placed as close as possible to the meat for all experiments except expt#1. In experiment#1 glad-wrap was placed on top of the meat, and the sensor lightly touched the top of the meat. During the last experiment (expt#4) the skin was put on top of a piece of fat, mixed and muscle. With the exception of expt#1, the sensor had no direct contact with the meat, during all other experiments.

The inter-digital sensor was connected with a 4.7 KΩ series resistor. The operating frequency was set at 500 KHz. The impedance was then calculated as shown below.

\[ \text{Impedance} = \frac{V_1}{i} \]

Voltage across the exciting coil = \( V_1 \)

Current (i) across sensing coil, \( i = \frac{V_2}{R} \), (\( R = 4.7 \text{ KΩ} \))

So the Impedance is given by \( Z = \frac{V_1}{i} \). The phase is calculated from the two waveforms.

Operating conditions: Temperature = 16.1°C, Frequency = 500 KHz

Some experimental results are shown in figures 10 to 12. The signal#1 (top) corresponds to the applied excitation and signal#2 corresponds to the current through the sensor.

The variation of impedance of the sensor with different conditions are shown in figures 13 to 16. An initial measurement is obtained for the sensor with no materials under it, before each of the four experiments. In the graphs above this measurement is shown as “Air”. The first experiment had low values of impedance compared to the other three experiments, for all types of meat. The direct contact the sensor had with the meat in expt#1 gave a higher output signal which resulted in lower impedance. Looking closely at expt#1 it can be seen that fat has the lowest impedance followed by muscle and finally mixed. This shows that the sensor can detect different types of pork based on the different permittivity values. Skin, which has the lowest thickness, fits in the middle. A clear cut analysis of skin is hard to obtain since it is very thin comparatively. In expt# 4 the skin is placed on top and experiments are carried out.

Expt#2 shows that fat has the highest impedance, followed by mixed and muscle. Once again skin fits in the middle. These results are backed up in expt#3. Expt#4 shows fat once again...
having the lowest impedance followed by mixed and finally muscle. In all but one (expt#1) of the cases fat and muscle are the two extremes and mixed is in the middle.

Fig. 10 Signals corresponding to sensor in air

Fig. 11 Sensors placed on fat

Fig. 12 Sensor placed on muscle
Fig. 13 Impedance of the sensor obtained from Expt#1

Fig. 14 Impedance of the sensor obtained from Expt#2

Fig. 15 Impedance of the sensor obtained from Expt#3
Fig. 16 Impedance of the sensor obtained from Expt#4

5 Conclusions

Novel planar electromagnetic sensors of meander, mesh and interdigital type have been fabricated. The sensors have been characterized and their comparative performance have been evaluated. The sensors are used for the nondestructive evaluation of system properties. The interdigital sensor has the great potential to be used to determine the fat content in pork. Some other novel applications are presently under consideration.

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Trough Guide Ring Resonator for Precision Microwave Moisture and Density Measurements

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ABSTRACT. The paper describes a novel trough guide stray field ring resonator for moisture and density measurements, which is entirely manufactured from alumina oxide. The ring is metallized at its surface except at the front face, which permits a stray field to come into contact with the material under test. The changes in resonant frequency and resonator bandwidth are utilized for the determination of the moisture content or the material’s density. The described resonator is capable to withstand considerable abrasive forces without having a protective cover. At the same time it shows excellent linear and predictive temperature behaviour, which allows precise compensation of the inherent temperature dependence of the resonator.

Keywords: Moisture measurement, ring resonator, stray field

1 Introduction

Microwave resonators can be used for highly accurate monitoring of the dielectric properties of materials. They are thus frequently employed for the purpose of moisture or density measurements. By determining the decrease of the resonant frequency and the increase of the bandwidth of a resonator loaded with a material in comparison to the empty resonator, and by properly processing these data [1], a material’s moisture content and its density can be determined precisely and independently. Such a two parameter measurement is only possible within a certain range of moistures and densities. Some materials exist, which can not at all be tested in that way. However, the approach works excellent and is highly accurate at moderate moisture levels for a wide range of natural materials like wool, tobacco, or wood chips, just to mention some examples. Such measurements are often made at bulk solids in industrial processes. If it is inconvenient to convey the material through the resonator, open resonant structures can be employed. The stream of material under test is then located in the stray field of the resonator.

2 Ring Resonators

A review of possible stray field resonators shows, that open ring resonators are very well suited for the desired purpose. Ring resonators have been widely used for the determination of effective permittivities in the microwave frequency range. Normally the determination of the dielectric properties of the substrate is intended, which is employed for the realization of the transmission line, forming the resonator. In the case under consideration, the material under test, covering the transmission line and thus reducing the effective permittivity, is of interest. A microstrip ring resonator, for example, can provide a suitable stray field, where the material under test can be positioned. Avoidance of extensive radiation is important, because radiation losses could not be distinguished from dissipation in the material. This is achieved by choosing the permittivity of the microstrip substrate much higher than the effective permittivity of the moist material. Various other shapes of resonators have been proposed, which serve the same purpose as the microstrip ring. An example for the described class of...
resonators is an $E_{mpo}$-resonator as proposed in [2], which is manufactured from a flat plate of high permittivity material, which has a central hole in its top plate. The stray field is admitted to expand into the outer space at the edges of that hole. The material under test is located on top. Such resonators are typically manufactured from cheap ceramic filled plastic substrates with copper cladding. Their resonant structures are defined by etching. For protection of the etched copper cladding against abrasive forces caused by the material under test, a hard ceramic layer is glued onto the etched pattern, preventing the immediate destruction of the metallization. Typically the cover is a thin plate of Al$_2$O$_3$.

A microstrip resonator of this type, suitable for moisture or density measurements, is shown in fig. 1. A typical substrate which is used for the microstrip resonator is TMM10 [3], having a permittivity value of about 10, which is approximately the same as that of Al$_2$O$_3$. The glue, however, typically shows a lower permittivity of approximately 4 (epoxy). The stray field, which is utilized for the moisture measurement, is forced to travel through the interface between the various dielectric materials approximately at right angles (Fig. 2, not to scale). Thus a high field strength results in the gap which is filled by the glue. The glue contact is not very well defined and is strongly temperature dependent as are the dielectric properties of the glue itself. Hence the glue in the gap has a strong effect on the resonant frequency and bandwidth of the resonator, resulting in a nonlinear temperature dependence. Even hysteresis may occur in the temperature characteristics of the resonant frequency and bandwidth.

This can be derived from fig. 3. In that picture, resonant frequency and bandwidth are shown.
versus temperature, which was decreased and then increased in a climatic exposure cabinet in steps of approximately 5°C between 18°C and 50°C. After applying a step, temperature was kept approximately constant for a while. After cycling the temperature down and up again, the climatic exposure cabinet was switched off at approximately 52°C and the resonator was allowed to cool down slowly. Fig. 3 shows an undesired poor reproducibility of the resonant frequencies and bandwidths versus temperature, and hysteretic deviations at the final cooling. Part of the deviations can be explained by an inhomogeneous temperature distribution in the whole assembly, but unsatisfactory reproducibility is obvious. In the past this problem, which is not a special feature of the resonator shown but occurs for virtually every resonator built in a similar manner, has largely complicated the measurement of materials at varying temperatures. This is particularly true if low density materials are to be measured with high accuracy.

3 Novel Trough Guide Ring Resonator

In the present paper a solution to the described temperature problem is reported. The Al₂O₃– protection plate on top of an etched pattern and hence the glue-layer is eliminated. This is only possible, if there is no etched copper layer on the upper face of the resonator. The resonator itself is made from Al₂O₃ in order to provide the required abrasion protection. The ring resonator is implemented as a dielectrically filled trough guide, and consists of a cylindrical ring of ceramic material metallized at its surface except at the top face of the ring cylinder [4]. Its construction is depicted in Fig. 4. A resonance is excited in the ring with the electric field being polarized tangentially to the upper open surface. A stray field extends into the outer space (Fig. 5). The electrical field distribution is virtually constant in radial direction across the upper face of the ring. The variation in axial direction is sinusoidal. The height of the ring corresponds to a little bit more than a quarter wavelength in the dielectric. The electrical field variation in
circumferential direction is shown in Fig. 6. As can be seen, there are six full wavelengths around the circumference in a sinusoidal distribution. Regions of high field intensity are represented in Fig. 6 by dark shades. The ring resonator is soldered into a metallic plate. Together with that plate it forms a flush surface, where the material to be measured can be placed on. A photograph of the new resonator is shown in Fig. 7. In that picture, the resonator is mounted together with the required microwave and microprocessor electronics to form a full moisture and density meter, suitable for the operation in a harsh environment.

Since no surface metallization of the sensitive area of the resonator is present and the resonator dielectric is highly resistant against abrasive forces, no further protection cover is needed. The temperature characteristics are shown in Fig. 8. As for the microstrip resonator of Fig. 3, temperature is cycled between 20°C and 50°C in steps of 5°C in the climatic exposure cabinet, with a final cooling after the exposure cabinet is switched off. Temperature cycling shows that the resonant behaviour of such a resonator is depending on temperature in a highly linear fashion. Hysteresis effects do not occur. The temperature coefficient of the resonant frequency is approximately that of the permittivity of Al₂O₃-ceramic. In comparison, the resonant frequency variation over the temperature change of 30°C has increased from approximately 600 kHz (TMM10, Fig. 3) to 4.5 MHz (Al₂O₃, Fig.8). This is due to the fact, that TMM10 is a temperature compensated dielectric composite. Since the change at Al₂O₃ is exactly linear with temperature, it is easy to apply temperature compensation numerically after the temperature has been determined with an independent probe.
On the other hand, when comparing the temperature changes of Fig. 3 and Fig. 8 in resonant bandwidth, there is registered approximately 1 MHz for the microstrip resonator, but only about 400 kHz for Al₂O₃. The bandwidth increase over temperature has several reasons. For the TMM10-resonator, it is caused by direct losses in the dielectric composite, by losses in the glue and by a decrease of the conductivity of the metal, defining the resonator (copper cladding). As pointed out earlier, a detrimental hysteresis effect is also noticed. A possible temperature change of the coupling to the connecting coaxial lines can be neglected, as this coupling is quite weak. For the Al₂O₃ trough guide resonator, there is no glue, which may produce losses, and dielectric dissipation in Al₂O₃ can virtually be neglected. Only the conductivity in the resonator walls decreases with temperature, thus creating a bandwidth increase, but again in a highly linear manner and highly predictable. The origin of that change is easily taken into account after measuring temperature.

In Fig. 5 also a reference resonator can be seen. That reference resonator does not see the material under test, but is exposed to the same temperature as the ring resonator used for the moisture or density determination. It is also manufactured from the same ceramic as the latter. By measuring its resonant frequency and resonant bandwidth, it can be utilized as a kind of thermometer for determining the average thermal conditions of the measurement resonator. Thus the whole assembly acts as highly precise moisture or density meter, which is suitable for measurements over wide temperature ranges of the materials under test. This is also true for low density materials at quite low moisture levels. Due to their low effective permittivity values (near 1) such materials cause only small detuning and attenuation of the resonance. The determination of their properties is thus highly susceptible to inherent and systematic measurement errors of the measurement resonator. Using the novel trough guide resonator assembly, such errors are effectively eliminated.

4 Measurements

Fig. 9 shows a measurement with the new resonator. The change of resonator bandwidth is plotted versus the change of resonant frequency for the moisture values of 5.1% (lower bold curve) and 10.2% (upper bold curve). The moisture values are on the wet basis. The density (mass) in the measurement area was varied as denoted by the dotted lines. From step to step the mass was doubled. As can be easily seen, the slope of the bold lines represents the moisture value. The ratio of change of bandwidth to change of resonant frequency is taken
and calibrated to denote the moisture content. In a similar way, a calibration for the density (mass) is possible by analysis of the dotted lines.

A microwave stray field resonator in an industrial environment for the measurement of wood chips is shown in Fig. 10. An example for a microwave measurement in comparison to measurement with a rapid dryer (infrared) is displayed in Fig. 11. As the reference, a moisture value from oven drying is given on the abscissa. The ordinate shows the respective values for a microwave measurement (Bold squares) and the rapid dryer (Circles). As can be seen, microwaves deliver much more accurate measurements, as compared to the rapid dryer. Further on, one has to take into account, that oven drying, although it is taken as the reference, inherently shows quite large measurement errors, which are attributed to the microwave measurement in the given presentation.

5 Conclusions

Microwave stray field resonators for moisture content or density measurements can be manufactured from ceramic-filled plastic substrates with copper cladding, where the resonator is defined by an etching process. In practise such resonators require a cover for protection against abrasive forces, which is realized by gluing a thin plate of alumina oxide onto the etched circuit. Such a multilayer circuit is prone to increased temperature sensitivity of resonant frequency and resonator bandwidth including hysteretic behaviour, which can make the resonant properties unpredictable after temperature changes. Moisture content and density measurements are deduced from changes of the resonance with respect to the empty resonator. If the reference measurement on the empty resonator is made at an early stage, and the material measurement is carried out later after some cycling of the temperature, a precise measurement becomes impossible. In practise, the empty measurement often cannot be repeated at a later time, because in a production process the resonator may be permanently loaded by the material under test.
By taking advantage of a trough guide ring resonator, a resonator can be realized which is by far superior to the cited multilayer construction. It is completely made out of alumina oxide and metallized at its surface except its upper face, which is in contact to the material under test. The described protection plate and an associated glue layer are not required, because the ceramic resonator structure itself already provides the ruggedness against abrasive forces. The resonators described in this paper, built in that way, show excellent performance in practise and are used in commercial moisture and density meters [5].

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Simple Soil Moisture Probe for Low-Cost Measurement Applications

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ABSTRACT. A simple and cost-effective soil moisture probe is described. It consists of a transmission line in the feedback loop of a ring oscillator, which can be embedded in soil or other material of interest. The water content of the surrounding material influences the frequency of the oscillator counted by a microcontroller circuit and transformed into water content by a calibration function. An integrated temperature measurement circuit may compensate for thermal effects and detect frozen material. The main challenge is a robust and low-cost probe design, especially suited for building sensor arrays to determine water content distribution in heterogeneous media.

Keywords: simple soil moisture probe, ring oscillator, calibration procedure

1 Introduction

For many applications soil moisture measurement devices like time domain reflectometers are too expensive and offer more features than really needed. Therefore a prototype for a simple and economical soil moisture probe has been developed, which allows measuring the mean moisture. Another important state variable besides moisture is temperature. Temperature measurements are needed for many purposes: To detect if the soil is frozen, which would change its hydraulic properties drastically. To gain information important for energy budget investigations (soil heat flux). A coupled moisture and temperature sensor could help to get more insight into the problem of temperature as tracer. Therefore a moisture sensor and a temperature sensor have been combined in one device. The probe is named Simple Soil Moisture Probe (SISOMOP). Its temperature sensor is a common semiconductor bandgap temperature sensor. The soil moisture measuring part is based on a ring oscillator whose frequency is changing with water content. This principle has been known for many years and varied in several ways [1, 2]. Recent patents focus on the ability to compensate for some level of variable conductivity, e.g. by analyzing the waveform on the transmission line in detail [3, 4]. Though the measurement principle has already been investigated, there is still room to improve electronic design and develop application specific transmission lines.

2 Sensor Design

2.1 Ring Oscillator and Transmission Line

The core of the probe's moisture sensor is a ring oscillator based on a digital inverter, which acts like a line driver (Fig. 1). This inverter drives a transmission line the end of which is fed into its input again. This makes the line driver oscillate. If a logic 1 state travels along the transmission line it will reach the input where it is inverted. The output of the line driver becomes logic 0 and this distortion propagates along the transmission line, until it reaches the
input of the inverting line driver. It is transformed into logic 1 and sent out again. The line
driver toggles with a frequency determined by the propagation velocity of the positive and
negative voltage pulses along the transmission line.

Fig. 1 Block diagram of the Simple Soil Moisture Probe and Temperature Sensor

If the transmission line is buried in soil, the pulse will interact with the surrounding medium,
especially the stored water. The higher the moisture the higher the effective dielectric
permittivity, leading to a lower wave propagation velocity $v_p$ and a thus lower frequency of
the ring oscillator. The inverting line driver is realised in ECL logic. The transmission line
consisting of two copper strips is etched on one side of a two-sided epoxy printed circuit
board (PCB), which also carries the necessary electronic parts (Fig. 2 and Fig. 3). This leads
to a simple, robust, and cost-effective design. To seal the strip line and to protect it from
scratches when pushed into soil, the transmission line is covered by a protective lacquer.

Fig. 2 Printed circuit board of the Simple Soil Moisture Probe, size: 185 mm × 30 mm

Fig. 3 Close-up picture of the Simple Soil Moisture Probe
The electrical field in the cross section of the PCB is shown in Fig. 4. It concentrates around the copper conductors only slightly shifted towards the upper side of the PCB where the conductors are positioned.

![Copper conductors](image)

Fig. 4 Qualitative view of the electrical field distribution in the cross section of the PCB. The surrounding material is assumed to be air. Dark areas correspond to low electrical field strength and bright areas (between two copper conductors on the left and on the right hand side) to high electrical field strength.

The lancet formed by the PCB is rigid and flexible at the same time due to the glass fiber substrate. The width of the PCB is 3 cm and the length of the part forming the ring line can vary between 10 and 20 cm depending on the user's need. The electronics occupy additional 5 cm. The typical frequency range determined by the current ring line layout is around several hundred megahertz. This ring oscillator frequency is too high to be countable by a standard microcontroller. A frequency divider brings it down to a range which can be conveniently counted. To determine the frequency the microcontroller counts the signal transitions from logic 0 to 1 during a fixed predefined timing window. The resulting moisture counts $N_c$ are proportional to the oscillator frequency and thus for the propagation velocity of the electromagnetic wave along the transmission line.

### 2.2 Integrated Temperature Sensor

The chosen temperature sensor DS18B20 [5] is a digital thermometer with a so-called *1-Wire bus* interface. A microcontroller can communicate with the chip over this interface, which only needs one date line and ground to transfer the digital data. The DS18B20 measures temperatures between -55°C and 125°C with an accuracy of ±0.5°C from -10°C to 85°C without calibration or error correction. The maximum resolution of 12 bits corresponds to temperature steps of 0.0625°C. A very useful feature of this integrated circuit is a unique 48 bit identifier stored in its ROM. Thus the combined moisture and temperature sensor using this chip can be identified uniquely. This information can be used to refer to sensor specific external calibration data if needed.
3 Calibration and Experimental Results

3.1 Preliminary Investigations

The sensitive area of the SISOMOP which is described by the extension of the electrical field is very small (cf. Fig. 4). This means in return that the influence of different soil densities or probe insertion events is very high. To receive adequate counts \(N_c\) at different moisture states for comparable soil situations and insertion events the calibration procedure was carried out during a dry-out experiment. The dimensions of the soil sample and the position of the moisture measurement device compared to the soil-air boundary have an influence how much the local moisture measurements represent the mean moisture of the probe. Fig. 5 describes the experimental setup for the determination of a minimal soil sample probe which is not influenced by any surrounded material. Using several different soil samples the minimum sensitive area was set to approximately 8 cm in diameter.

![Fig. 5 Determination of minimum sensitive area around the SISOMOP using air- and water-surrounded soil samples](image)

To get more independent information about the electrical properties (dielectric permittivity \(\varepsilon\)) of the soil a conventional time domain reflectometry (TDR) rod-probe has been inserted in the same soil sample. The two probes will be very close to each other so the interaction of the probes has also to be taken into account. Fig. 6 shows three different combinations of SISOMOP and/or TDR-probe inside of one soil sample. Similar to the procedure from Fig. 5 the minimum distance between these two measurement devices has been determined for several soil samples by at least 10 cm.

![Fig. 6 Investigation of minimum distances between SISOMOP, TDR-rod-probe and soil-boundary](image)
As a result of these preliminary investigations the optimal size of the soil sample has a diameter between 18 and 20 cm. The height of the sample depends on the length of the probes plus an additional overhead of 3 to 4 cm on the top and bottom layer.

The cylindrical-shaped soil sample is surrounded by a thin plastic meshwork to allow evaporation and prevent damaging. The whole sample is also placed on a grid so that free water can leak out of the soil.

3.2 Calibration

The electrical measurement devices described in the previous section lead to an indirect determination of the soil moisture content. These measurements require a calibration function between the electrical and the soil-physical parameters. To get permanent measurements of the gravimetric water content of the soil sample the total test equipment has been placed on an electronic weighing machine (Fig. 7). The volume of the soil sample can easily be calculated by measuring the soil-cylinder and probe dimensions. With this additional information the volumetric water content can also be calculated.

![Schematic view of the calibration-equipment using SISOMOP, TDR-rod-probe and an electronic weighing machine](image)

To start one calibration experiment the soil is saturated between 70 and 80% of its pore volume. Then the material is manually filled into a rigid pipe segment which is covered inside with a plastic meshwork. During the filling procedure the two moisture probes are inserted into the material. After the filling procedure the stabilising pipe segment is removed.

When the soil sample is placed on the weighing machine a computer collects $N_c(t)$ from the SISOMOP, dielectric permittivity $\varepsilon(t)$ from the TDR-probe and the gravimetric water content $\theta_g(t)$ from the electronic weighing machine.

One calibration experiment takes about one to three weeks to reach a steady state of dry material depending on the soil type (sandy soil dries out much faster than clayey material). To compare field taken moisture counts $N_c$ with its corresponding water content three different calibration experiments with silty soil were carried out (sample-1: silty material from Untergrombach, Germany, dry density $\rho = 1.3 \text{ g/cm}^3$, sample-2: silty material from Vicunia, Chile, $\rho = 1.6 \text{ g/cm}^3$, sample-3: same location than sample-2 but $\rho = 1.5 \text{ g/cm}^3$).
Fig. 8 shows the relation between the moisture count $N_c$ and the dielectric permittivity $\varepsilon$. As one can see the moisture count and the dielectric permittivity are correlated and do not depend on the material properties. Nevertheless future calibration experiments with sand or clay have to prove this assumption.

The relation between the moisture count $N_c$ and the dielectric permittivity $\varepsilon$ can be described with a polynomial of third class to fit all three calibration data-sets.

$$\varepsilon = 5.15 \cdot 10^{-12} N_c^3 - 2.684 \cdot 10^{-7} N_c^2 + 1.208 \cdot 10^{-3} N_c + 71.66$$  \hspace{1cm} (1)

When using known material dependent transformation functions between and $\varepsilon$ and $\theta_v$, the water content can easily been calculated. Using the standard transformation developed by Topp et al. [6] the relation between moisture count $N_c$ and water content $\theta_v$ becomes approximately linear (see Fig. 9).

Fig. 8 Relation between SISOMOP moisture-counts and dielectric permittivity measured with TDR-rod-probes

Fig. 9 Relation between SISOMOP moisture-counts and volumetric water content using $\varepsilon$-calibration from Fig. 4 and empirical $\varepsilon$-$\theta_v$-relation according to Topp et al.
This relation can be described by

\[ \Theta_v = -3.531 \cdot 10^{-3} N_c + 114.9 \]  

(2)

3.3 Experimental Results

The volumetric water content \( \theta_v \) from (2) is determined by the dielectric permittivity \( \varepsilon \) of the soil using a standard transformation that has been developed empirically for many soil types. But this water content may vary from the soil-specific water content measured gravimetrically. Therefore the weight measurements were added to receive a better conversion from moisture count \( N_c \) to actual water content.

Fig. 10 shows the relation between moisture counts and gravimetrically determined water content. One can see that different materials (sample-1 from Germany, sample-2 and 3 from Chile) may lead to different calibration functions. The wet materials (15 to 35 \%vol) behave similar whereas the dry materials (4 to 15 \%vol) lead to different moisture counts. There are no important variations between two different installations of the same material (sample-2: \( \rho = 1.6 \text{ g/cm}^3 \), sample-3: \( \rho = 1.5 \text{ g/cm}^3 \)).

![Fig. 10 Relation between SISOMOP moisture-counts and volumetric water content which was determined gravimetrically on a electrical weighing machine (the standard calibration function from Fig. 9 is also included to see the deviation between these two methods)](image)

This calibration procedure leads to material-dependent calibration functions to describe the correlation between moisture counts \( N_c \) and volumetric water content \( \theta_v \). The Untergrombach-material (Germany) can be described with

\[ \Theta_v^1 = 1.246 \cdot 10^{-31} N_c^3 - 7.144 \cdot 10^{-7} N_c^2 + 8.429 \cdot 10^{-3} N_c + 62.99 \]  

(3)

and the Vicunia-material (Chile) with

\[ \Theta_v^{2,3} = 1.369 \cdot 10^{-31} N_c^3 - 8.358 \cdot 10^{-7} N_c^2 + 1.166 \cdot 10^{-2} N_c + 37.52 \]  

(4)

These calibration functions were successfully used in field experiments on the two test sites.
4 Conclusion

A simple soil moisture probe has been developed based on the ring oscillator principle. The transmission line in the feedback loop is etched on a printed circuit board which can be inserted in the material of interest. In combination with an onboard temperature sensor and a material specific calibration reliable long-term measurements of water content are achieved. Cost sensitive applications may profit from the simple and economical design.

To calibrate this new type of moisture sensor a combined electrical and gravimetrical calibration procedure has been tested. It leads to a material-independent relation between the dielectric permittivity and the counts from the moisture probe. This allows on site water content measurements without knowledge of the tested soil. To enhance the quality of the water content determination a soil-specific calibration is required. The presented calibration procedure leads to material-dependent calibration functions.

References


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Session 7

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Non-Contact Moisture Sensor for Fresh Concrete

Ferenc Volgyi
Budapest University of Technology and Economics, Hungary

ABSTRACT. This paper is devoted to microwave moisture-measurement (aquametry) and composition estimation of civil structures. The first part of this paper is an overview of the work done by researchers (all over the World) involved in using microwave (or high frequency electromagnetic-) signals for moisture measurement and non-destructive testing and evaluation (NDT&E) mainly of concrete (in place and fresh). In the second part of this paper will be introduced a Non-Contact Sensor (NCS), designed to use in concrete mixers above the moving mix, and using a cross- polarised, active back- scatter (BSC). The system is based on a microwave free- space/ double transmission/ reflection type-, two-parameter complex vector measurement.

Keywords: sensor, concrete, moisture measurement, non-destructive testing

1 Introduction

Nowadays, microwave and radio frequency signals are employed for a wide variety of applications. It includes wireless communication systems, mobile phone, radar, telemetry, medicine, biology, agriculture, industrial process control, etc. Electromagnetic (EM) energy can be used efficiently in these applications only if its interaction behaviour with associated substance is known. This, in turn, depends on the EM properties of that material. Therefore, the material characteristics can be deduced by analysing the EM signals interacting with it. One of the most important parameter of materials is the water content, or moisture content (MC). Many damages in buildings are caused by moisture. They can be of various forms, 24% because of penetration of rain- water, 18% cracks, 17% combined damages, 16% are due to condensed water, 15% flaking of plaster, 5% moisture rising from ground. Damage caused by moisture required expenses of maintenance (in the year of 1996) of ca. $28 billion [1]. Low intensity microwave radiation is applied for microwave moisture measurement was called "microwave aquametry" by Kraszewski [2], [3].

Concrete is one of the major materials used in construction globally. It is comprised of cement, water, fine aggregate and coarse aggregate. The cement and water combine into a cement paste binder. The compressive strength of concrete is determined mainly by its water-to-cement w/c ratio, which can be measured by near-field microwave techniques [4]. Changes of moisture in aggregates in the range of 2 - 6% MC reduce the compression strength of concrete up to 25% [5]. Reinforcing steel bars are applied in concrete bridge decks and columns. Chloride intrusion into concrete can lead to depassivation of the steel and initiation of corrosion, which can be detected by microwaves [6]. Reinforcing steel corrosion is the cause of damage in the majority of reinforced concrete bridges in the United States.

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It is essential to insert control points into modern automated manufacturing processes to ensure homogeneous product quality. Particleboard PB, chipboard, oriented strand board OSB manufacturing also needs a reliable control method that is able to monitor large size composite boards continuously [7]. The uneven density and MC distribution, voids and fissures, the uneven spreading of the glue on the particles and other less important factors
have great effect on the quality of the boards. The majority of these factors can be related to the change of the properties of microwaves as they penetrate through the board [8].

Earliest reports on the concrete moisture measurement appeared in the second half of 1980's [9-12], with some patents [13-15]. Intensive R&D programs have started in 1990's at the Colorado State University, Fort Collins [16], continued in 2003 at the University of Missouri, Rolla [6,17]. There were solved some NDT&E problems of cement based materials by this research group, namely, evaluation of water-to-cement ratio and compressive strength of hardened cement paste [18,19]; evaluation of fresh concrete w/c [20]; evaluation of porosity and sand-to-cement ratio (s/c) in mortar [21]; evaluating mortar permittivity using a combined microwave near-field and modulated scattering technique [22]; evaluation of coarse aggregate-to-cement ratio (ca/c), cure-state and material properties of concrete [4, 23, 24, 25]; detection of grout in masonry bricks [26]. Near-field microwave NDT techniques have also been extensively used in evaluating chloride contamination in cement based materials [27], [28].

After studying more than 150- papers, we could mention some authors and research groups who are active on this field: Bosisio [29] in Canada; King [56], Kraszewski [2, 3], Nelson [30], Trabelsi [31], Zoughi [16, 18, etc.] and his research group in the United States; Stelzer [32] in Austria; Nyfors [33] in Finland; Bolomey [57] and Lasri [34] in France; Brandelik [35], Kupfer [1, 5, 15], Leschnik [37] in Germany, Volgyi [7,8,50-55,58-63] in Hungary; Gentili [38], Paletta [9] in Italy, Kalinski [10] in Poland, Berentsev [14] in Russia, Lenngren [39] in Sweden, Akay [25] in Turkey, Kent [40] and Wang [41] in the UK; Hayashi [42] and Okamura [43] in Japan; Khalid [44] in Malaysia, Van der Berg [45] in South Africa; Bialkowski [46] and Cutmore [47] in Australia; Holdem [48] and Lovell-Smith [49] in New Zealand.

Some measurement methods-, microwave circuit elements are given in the next references. Calibration (density independent): [31]; detection (of rebars in concrete): [16]; free space technique: [9,25,50,59,60]; GPR: [39]; homodyne system: [56]; instruments: [9,13,14,15,33, 40,47,51,52,58-62]; MSA: [36,51,55,59,60,61]; MST (modulated scattering technique): [7,8, 21,51,52,57,58,59,61,62]; multi- (and swept-) frequency technique: [2,47,48,63] references (overview): [3,6,12,43,63]; reflection measurement: [11,32,50,53,58, etc.]; six-ports: [29,32, 46]; spread spectrum technique: [52,63]; transmission measurement: [11,13,15,31,40, 46,47, 50,58,60]; transmission line technique (S-parameters): [41]. Materials; sand: [1,10,12,23,53]; water: [2,9,20,53]; wood: [2,7,8,43,44,51,59].

2 NCS above the Moving Mix

The goals for this research were: (1) sensor will mount inside mixer and measure reflections from material to measure moisture independent of density. (2) It should be self-learning by presenting samples of different moisture and density. (3) It should be independent of distance between sensor and material, which will be in motion. (4) It should be unaffected by motions of arms and blades. (5) Distance between sensor and material is 1 m nominal. (6) Temperature range for this sensor will be 0-40 °C. (7) Accuracy objective will be ± 0.2 % moisture over a range of 0-10 % if possible. (8) The reading will be further averaged in an external computer in order to obtain average- moisture for the mix. This averaging should
take 20 seconds or less, after which the water will be added to the mix. (9) The sensor will be splashed with liquid concrete. Therefore, use of a PTFE from shield is envisaged, with an automatic water spray to clean the surface. (10) Mechanical design should include isolation between sensor electronics and body of several thousand Volts for lightning protection.

2.1 Model of the Non-contact Sensor

The simplified model of the non-contact sensor for use in concrete mixers above the moving mix is shown in Figure 1. A microwave source is generating the signal for transmission on a level nearly 20 dBm, according to the control of rotation. Co- and cross-polarised COP/CRP microstrip antennas are used, and they are protected by a PTFE-radem. The sensitivity of the receiver must be better than -80 dBm. Polarisation changer passive detector back-scatter, PDB or detector active back-scatter, DAB is used for returning of the microwave energy, depending on the size of mixer used. In the DAB circuit is applied an amplifier, with low power consumption, and may be amplitude- or phase-modulated by special codes. Multi-frequency operation is suggested, according to the motion of fresh concrete, so according to the variation of distances R and d. The back-scatter is protected by a very hard SiN-cap. This integrated unit is built into the bottom of mixer for some years without any cable-connection, so its integrated circuits are sometimes in sleeping-mode.

2.2 Preliminary Calculations

A simple analysis of the double transmission-attenuation for NCS is written next. In Figure 2, a representation of the attenuation process is shown, neglecting the multiple-reflections.

The energy reflected at the boundaries of different materials and with it the energy due to unwanted reflections of the environment is definitely lost. That is due to the fact that those
waves are not modulated by the back-scatter (BSC) and then not detected by the receiver. Neglecting the air gaps, setting off the radar equation (1), the free space attenuation is:

\[ A_0 = (4\cdot\pi)^3 \cdot (R + D)^{4} / (\lambda^2 \cdot \sigma \cdot G^2 \cdot \Gamma^2 \cdot M) \]  

This has to be added to attenuation due to the energy reflected inside the round path. In (1) \( \lambda \) is the free space wavelength, \( \sigma \) is the radar cross-section of the back-scatter antenna, \( G \) is the gain of the Tx and Rx antennas when equal, \( \Gamma \) is the reflection coefficient of the BSC (passive or active, using amplifier), and \( M \) is a modulation efficiency at BSC. The loss of the boundary between air gap and the ceramic cap is:

\[ A_{iac} = \left\{ 1 - \left[ (1 - \sqrt{\varepsilon_{rc}}) / (1 + \sqrt{\varepsilon_{rc}}) \right]^2 \right\}^{-1} \]  

and

\[ A_{ica} = \left\{ 1 - \left[ (\sqrt{\varepsilon_{rc}} - 1) / (\sqrt{\varepsilon_{rc}} + 1) \right]^2 \right\}^{-1} = A_{iac} \]  

The attenuation inside the material due to \( \varepsilon_{rs}'' \) and the total attenuation of the double transmission path are then:

\[ A_s = \exp \left( \pi \cdot \varepsilon_{rs}'' \cdot 2 \cdot D / \lambda \cdot \sqrt{\varepsilon_{rs}'} \right) \quad (4), \quad \text{and} \quad A_T = A_0 \cdot A_s \cdot \prod_{i=1}^{4} A_i^2 \quad (5) \]

Figures 3 and 4 show the calculated attenuation and phase for the double path set-up.

Fig. 3 Transmission loss for the double path set-up at 0.9 GHz

Fig. 4 Phase shift for the double path set-up at 0.9 GHz
The calculations were done for planetary counter-current mixers (Fig. 5) types HPGM-375, 1500, 3750, Fejmert S-150, 6000, Sun-750, 1500, and for twin shaft mixers (Fig. 6) with types given in Table 1.

Fig. 5 Planetary counter-current mixer.

Fig. 6 Twin shaft mixer.

Basic geometry for calculations of microwave properties

Table 1 Manufacturers of twin-shaft mixers: AVANTI, BHS, TEKA, SIMEM, KABAG

<table>
<thead>
<tr>
<th>Type</th>
<th>Manufacturer</th>
<th>H [m]</th>
<th>D [m]</th>
<th>Yield uncompacted concrete output [m$^3$]</th>
<th>Filling factor</th>
<th>d [m]</th>
<th>R [m]</th>
<th>d + R [m]</th>
<th>Calculated $A_T$ [dB] @ M=1%</th>
<th>M=6%</th>
</tr>
</thead>
<tbody>
<tr>
<td>AV-4500</td>
<td>Avanti</td>
<td>1.72</td>
<td>2.6</td>
<td>3.6</td>
<td>0.47</td>
<td>0.64</td>
<td>0.92</td>
<td>1.56</td>
<td>67.9</td>
<td>85.0</td>
</tr>
<tr>
<td>AV-6000</td>
<td></td>
<td>1.72</td>
<td>2.6</td>
<td>4.8</td>
<td>0.62</td>
<td>0.90</td>
<td>0.66</td>
<td>1.56</td>
<td>69.0</td>
<td>89.6</td>
</tr>
<tr>
<td>DKX-2.0</td>
<td>BHS</td>
<td>1.4</td>
<td>1.48</td>
<td>1.6</td>
<td>0.39</td>
<td>0.43</td>
<td>0.83</td>
<td>1.26</td>
<td>63.3</td>
<td>77.6</td>
</tr>
<tr>
<td>DKX-6.0</td>
<td>Sont-hofen</td>
<td>1.93</td>
<td>3.22</td>
<td>4.8</td>
<td>0.40</td>
<td>0.62</td>
<td>1.12</td>
<td>1.74</td>
<td>69.7</td>
<td>86.5</td>
</tr>
<tr>
<td>DKX-9</td>
<td></td>
<td>2.40</td>
<td>3.20</td>
<td>7.2</td>
<td>0.28</td>
<td>0.48</td>
<td>1.68</td>
<td>2.16</td>
<td>72.8</td>
<td>87.8</td>
</tr>
<tr>
<td>TDZ-2250</td>
<td>TEKA</td>
<td>1.2</td>
<td>1.5</td>
<td>1.8</td>
<td>0.83</td>
<td>0.86</td>
<td>0.22</td>
<td>1.08</td>
<td>62.5</td>
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<tr>
<td>TDZ-3750</td>
<td></td>
<td>1.4</td>
<td>2.04</td>
<td>3.0</td>
<td>0.53</td>
<td>0.58</td>
<td>0.68</td>
<td>1.26</td>
<td>63.9</td>
<td>80.2</td>
</tr>
<tr>
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<td>1.4</td>
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<td>3.6</td>
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<td>0.76</td>
<td>0.50</td>
<td>1.26</td>
<td>64.7</td>
<td>83.5</td>
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<tr>
<td>TDZ-5250</td>
<td></td>
<td>1.65</td>
<td>2.04</td>
<td>4.2</td>
<td>0.53</td>
<td>0.68</td>
<td>0.81</td>
<td>1.49</td>
<td>67.3</td>
<td>85.0</td>
</tr>
<tr>
<td>MSO-2500</td>
<td>SIMEM</td>
<td>1.15</td>
<td>2.12</td>
<td>0.40</td>
<td>0.37</td>
<td>0.67</td>
<td>1.04</td>
<td>59.7</td>
<td>73.2</td>
<td>62.3</td>
</tr>
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<td></td>
<td>1.50</td>
<td>3.75</td>
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<td>0.87</td>
<td>1.35</td>
<td>64.7</td>
<td>79.7</td>
<td>69.0</td>
</tr>
<tr>
<td>MSO-9000</td>
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<td>1.95</td>
<td>7.5</td>
<td>0.40</td>
<td>0.62</td>
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<td>1.75</td>
<td>69.8</td>
<td>86.7</td>
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<td>DWM-3375</td>
<td>KABAG</td>
<td>1.41</td>
<td>1.73</td>
<td>2.7</td>
<td>0.55</td>
<td>0.63</td>
<td>0.64</td>
<td>1.27</td>
<td>64.3</td>
<td>81.3</td>
</tr>
<tr>
<td>DWM-4500</td>
<td>Wiggert + Co.</td>
<td>1.41</td>
<td>2.30</td>
<td>3.6</td>
<td>0.56</td>
<td>0.65</td>
<td>0.62</td>
<td>1.27</td>
<td>64.4</td>
<td>81.6</td>
</tr>
</tbody>
</table>

We made calculations for concrete with water to cement ratio w/c= 0.45, sand to cement ratio s/c= 1.5, and course aggregate to cement ratio ca/c= 1.5. A large variation of the calculated attenuation, and difference between attenuation measured at MC= 1 % and 6 % is observable, depending on the type of concrete mixer. If we should make a calibration starting with empty mixer, then the dynamic range is an important factor, which is between 48.7 dB and 133.8 dB. From point of view of MC measurement, the variation versus MC is important, which is between 2.5 dB and 58.5 dB. By all means, the appropriate frequency may be selected.
2.3 Concept of the Measurement System

Simulation results showed that the lowest frequency (2.45 GHz) of our previous measurement system (reflection sensor, [54]) is applicable for the new sensor working in double transmission mode, using passive detector back-scatter, PDB. For twin-shaft mixers where the thickness of the concrete is higher, an even lower frequency (0.915 GHz) is suggested with detector active back-scatter, DAB. The 915 MHz ISM-band is frequently called the "garbage band" since it has become filled with a diverse set of emitters ranging from spread spectrum data links to amateur radio operators, each with a different level of protection. It is largely unregulated since operation there is on an unprotected base, except for government services, and type acceptance of equipment is not necessary for the majority of users.

Synchronised measurements are designed, because the result of moisture measurement must be unaffected by motions of arms and blades of the rotating mixer, and only part of a round is actively used. An other solution is also possible: making a statistic of received signal and the self-learning system has a decision, which is the appropriate time-window for measurement. The measured attenuation and phase shift depends on the moisture content (MC) and the distance (R) between sensor and material. This distance is changing at moving mixer, and phase determination is doubtful, because we have not data enough. The suggested solution is the Multi-frequency operation, e.g. to use two additional frequencies around the center frequency. Our inverse software, function (R, MC) can calculate the exact value of the distance using frequencies of 2.41-2.45-2.49 GHz.

Back-scatter modulation technology, BMT is the basic idea came from the radio frequency identification, RF/ID market [64], where the systems are using radio signals to communicate. Line of sight is not needed and the system can operate in hostile environments characterised by water, oil, paint, and dirt. It can even be used for communication through cement, glass, wood, or other non-metallic materials. The signal scattered by a probe is generally quite small, and it may get lost in the background noise. To overcome this drawback, which results from wireless transmission, this signal can be modulated, and then retrieved by a receiver making use of coherent detection [65].

Passive BMT TAGs (transponders) not need a separate external power source, or no microwave power is generated inside it [66]. They derive their operating power from the energy sent by the interrogator (transmitter). Some passive tags contain a battery to maintain internal memory information in read/write applications. The main problem is battery life. Since the lifetime of the TAG is 5 to 10 years on the self contained battery supply, the quiescent battery drain must be in the order of 1 or 2 microamperes [67]. Operation of the TAG at a toll interchange requires several milli-amperes to operate the internal digital and analogue intelligent devices. Consequently, it is necessary for a TAG to exist in a "sleep mode" until called upon to exchange information. In our system a passive detector back-scatter, PDB will be applied [68]. The activation ("wakeup mode") is made, by sending an on-off keying (OOK) code by the transmitter. The matched detector of PDB demodulates the signal and transfers the data to the digital circuits of PDB. The activation is made with a binary phase shift keying (BPSK) encoder, which modulates the continuous wave (CW) of the transmitter. The receiver will sense this reflected wave, back-radiated by the MSA of the PDB [52].

Active BMT is realised, if microwave power is generated or amplified in an active TAG. In our system a detector active back-scatter, DAB unit will be designed, where an integrated amplifier is taken between the two inputs of a cross-polarised microstrip antenna. This unit was used in our first laboratory measurements [62]. The basic problem in such systems is, that the receiver front-end may be receiving a fixed carrier directly from the transmitter at a level of 0 dBm at the same time that the modulated reflected signals from the PDB/DAB may be on
the order of - 80 dBm. While the -80 dBm level may be easily detected, the high level of fixed carrier into the mixer may overload the system, supressing the signals in a highly non-linear manner. To combat this, the following methods are used: A/ Sufficient isolation between transmitter output and receiver input must be maintained, using cross polarized Tx/Rx antennas. B/ The reflections from the environment must be minimised, using circular polarisation. C/ The front-end of the receiver must support a high dynamic range. D/ The transmitter power must be the minimum necessary for the system performance, using, e.g., a spread- spectrum system. Spreading is used not only reducing average radiated power and against jammers, but passive clutters, too, therefore modulation takes part at PDB/ DAB. There will be a radar-like operation: measure signal's relative amplitude and phase, not transferring information.

Having neglected the attenuation inside the PTFE- and ceramic cap. Calculated results [62] for industrial planetary counter-current mixers with yield concrete of 0.3 - 4.8 m$^3$, with MC = 6% are: at the frequency of 0.915 GHz, $A_T = 54 - 67$ dB, at the frequency of 2.45 GHz, $A_T = 71 - 93$ dB. For twin shaft mixers (2.1 - 7.5 m$^3$), the calculated total attenuation is $A_T (0.915) = 62 - 75$ dB, $A_T (2.45) = 92 - 117$ dB. To handle this large attenuation in industrial environment, a sophisticated code- modulation will be applied at BSC.

3 Measurement Results

The preliminary measurement set-up [62] is shown in Fig. 7. The dual- polarised, single MSA-patch receives the co- polarised signal, which is amplified (14 dB) by a low noise amplifier, modulated and re-radiated in cross polarisation. This double-transmission signal is received by the Rx-MSA. The experiment with fresh concrete show linear function of the attenuation versus moisture content, namely: $A [\text{dB}] = 24.1 + 1.2 \text{MC} [%]$, at the frequency of $F_0 = 2.457$ GHz. The measurement is independent of the density and the distance- variation between transmit / receive antennas and material, because the information from two additional frequencies around $F_0$ (e.g. ± 40 MHz) are used. The new instrument, developed, will also apply the 0.9 GHz frequency- band.

![Measurement set-up for NCS using active-BSC](image)

Fig. 7 Measurement set-up for NCS using active-BSC

4 Conclusions

In recent years, microwave NDT&E techniques, mostly in the near field using open- ended rectangular waveguide probes, coaxial monopole antennas, microstrip antennas, have been extensively used to interrogate a wide variety of cement based materials for their important
physical and structural properties [63]. Some examples of these R&D projects were mentioned in the Introduction of this paper. The double frequency (0.9 and 2.45 GHz) NCS is designed for use in concrete mixers above the moving mix, or for inspection of civil structures with large wall-thickness, because of the high dynamic range in attenuation measurement. The active BSC is independent of the Tx/Rx unit, so the arrangement is practically single-sided. The density-independent operation will be provided by an ANN with self-learning by presenting samples of different moisture and density.

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Application of Microwave Impulse Method for Measuring Moisture Profiles in Building Materials

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ABSTRACT. A microwave impulse method is employed for measuring moisture profiles in building materials. The experimental setup is relatively compact and consists of three basic components, the impulse generator, the applicator and the sampling oscilloscope. The experiment is arranged in the form of a horizontal capillary suction of water. Samples of three typical porous building materials, cement paste, ceramic brick and autoclaved aerated concrete, are water and water vapor insulated with Epoxy resin on four lateral sides to ensure the one-dimensional transport. Moisture contents are measured at different positions and time intervals during the experiment producing moisture profiles for the particular times. The moisture diffusivity is then calculated from the moisture profiles using the inverse analysis involving Boltzmann transformation.

Keywords: microwave impulse method, moisture profile, moisture diffusivity, building materials

1 Introduction

Measuring transient moisture profiles is considered as a common and effective tool for determination of liquid moisture diffusivity of porous building materials. In classical experimental setups rod specimens water and vapor proof insulated on all lateral sides (parallel to the direction of moisture transport) are placed in either horizontal or vertical position. Then, they are brought in contact with water by one of their face sides. Beginning from this time, moisture contents are measured at different positions and time intervals during the experiment producing moisture content profiles versus time. Finally, measured moisture profiles are analyzed using the methods of inverse analysis and moisture diffusivity vs. moisture content function is determined.

There are a variety of methods for determination of moisture content. The most frequently used in building physics are the \( \gamma \)-ray attenuation technique [1-3] and the NMR technique [4-5]. Other commonly used techniques are for instance the capacitance method [6], positron emission tomography [7], neutron radiography [8] and microwave method [9]. The TDR technique [10] originally used in soil science or the X-ray radiography evolved from the well-known medical technique [11-12] can also be applied to building materials.

In this paper, we use for the determination of moisture profiles in porous building materials the microwave impulse method. The results obtained by this method are then compared with results obtained by the capacitance method and the standard gravimetric method.

2 Materials and Samples

Three typical building materials, namely cement paste, ceramic brick and autoclaved aerated concrete, were tested. The cement paste was prepared using Portland cement CEM I 32.5 R (ENV 197-1) (Horní Smí, CZ) and water. The water to cement ratio \( w=0.3 \) was chosen in our experiments. The bulk density of the cement paste was 1910 kg-m\(^{-3}\). The ceramic brick was
produced by the brick kiln Nebužely, CZ. The bulk density of ceramic brick was 1720 kg⋅m\(^{-3}\). Autoclaved aerated concrete (AAC) was produced by Ytong (Laussig, Germany). Its bulk density was 650 kg⋅m\(^{-3}\).

For the measurements of moisture profiles we used the following samples: 6 specimens 20 x 40 x 280-300 mm for every measured material and every method. The samples were insulated on all lateral sides by water- and vapor-proof plastic foil. They were stored in the laboratory condition with the temperature of 25 °C and the relative humidity of 50%.

3 Experimental Methods

3.1 Microwave Impulse Method

The measuring system designed in [13] was used for the experiments. It is relatively compact and consists of three basic components (see Fig. 1), namely the impulse generator, the applicator and the sampling oscilloscope.

The generator GPSI-1a (Radan, Ltd.) produces triangular impulses of a width of 250 ps and amplitude 2 V. It consists of the impulse generator itself, its feed circuits, controlling, auxiliary and protecting circuits. The energy output is realized by three SMA coaxial connectors. These signals make it possible to determine the reference and measured position of the impulse and to synchronize the sampling oscilloscope.

The applicator connected to the generator output ensures the necessary exposure of both measured and reference specimens. It consists of two pairs of transmitting and receiving antennas formed by coaxial/waveguide reducers and horns. The pairs of antennas are fixed parallel in separate holders ensuring a defined position, and therefore also stability and reproducibility of measurements. The specimens of the tested materials are put into the applicator between the measuring antennas. The sample thickness is limited mechanically to about 100 mm, from the electric point of view it is limited by the attenuation in the measured material and sensitivity of the oscilloscope. The dynamics of the signal is over 20 dB.

The sampling oscilloscope Tektronix 7603 analyses the impulse signals. It has a 7T11A sampling sweep unit and two 7S11 sampling units with a S-4 sampling head. The time resolution of the oscilloscope is about 10 ps and the sensitivity 2 mV. The frequency range is up to 14 GHz. The signal from the oscilloscope display is recorded by a digital camera and analyzed by a PC.

The experimental setup is shown in Fig. 2. The specimen is fixed in horizontal position in order to eliminate the effect of gravity on the moisture transport. The lateral sides of the specimen are water and vapor-proof insulated in order to simulate 1-D water transport. A viscous sponge arranging for a good contact of the specimen surface with water is put into a Perspex water-filling chamber and applied to one face side of the specimen. The sponge sucks water from a free surface being about 10 mm below the lower side of the specimen. Water in the chamber is maintained on constant level using a float. If water level in the filling chamber decreases due to the water suction by the specimen, the water level in the float chamber decreases in the same way. The needle of the float opens the hole in the cover of the float chamber and water from a burette flows through the hole into the float chamber until the needle closes the hole again due to the water level increase. In this way, a continuous water
supply to the measured specimen is achieved. Scanning by the microwave impulse moisture meter along the specimen was done every 10 mm.

![Block diagram of the microwave impulse method](image)

**Fig. 1** Block diagram of the microwave impulse method

Calibration curve of the microwave impulse moisture meter is for a particular material usually determined in advance using the gravimetric method. In this case, we have chosen another method. The calibration was done after the last moisture meter scan when the moisture penetration front was at about one half of the length of the specimen, using this last scan and the results of the standard gravimetric method measurements after cutting the specimen into 10 mm wide pieces. The calibration curve was constructed as the dependence of the moisture content on the permittivity of the measured material. The permittivity of the material was calculated on the basis of measuring the time difference \( \Delta t_{21} = \Delta t_2 - \Delta t_1 \), where \( \Delta t_2 \) is the travel time of the impulse to pass the thickness of a measured specimen and \( \Delta t_1 \) the respective travel time in the air (see [13] for details of the calculation procedure). The final calibration curve for every material was constructed using the data for 3-6 samples. In the regression analysis, a logarithmic function was found as the best approximation to the measured data. Example of the calibration curve is presented in Fig. 3.

![Experimental setup of the horizontal capillary suction of water](image)

**Fig. 2** Experimental setup of the horizontal capillary suction of water
3.2 Capacitance Method

The capacitance device designed in [6] was used for the measurements. The low-voltage supply drives an oscillator of 400 kHz working frequency which has constant output voltage feeding a circuit where the measuring capacitor (with the analyzed sample as a dielectric) is connected in series with a resistance. On this resistance, the voltage after rectifying is determined which depends on the moisture content of the dielectric in a significant way. A relation between the moisture content and the voltage measured on the resistance can be determined by a calibration. The measured voltage increases with increasing capacity. By a proper choice of the resistance it is possible to achieve that the dependence of the measured voltage on the capacity is linear in the range of approximately one or two orders of magnitude of the capacity. Voltage is recorded in specified time intervals by data logger.

The described capacitance moisture meter was equipped with electrodes in the form of parallel plates with dimensions 20 x 40 mm. The moisture meter readings along the rod specimen were done every 5 mm in order to achieve certain space averaging of results and reduce the effect of inhomogeneities of the material.

The experimental setup was very similar to that for the microwave impulse method.

Similarly as with the microwave impulse method, the calibration curve was determined using the results of the last reading and the data obtained by the standard gravimetric method after cutting the specimen into 10 mm wide pieces. As in the case of the microwave impulse method, also here a logarithmic function was found to be the best approximation of measured data. Example of the calibration curve is shown in Fig. 4.

Fig. 3 Calibration curve of the microwave impulse method for cement paste

![Calibration curve of the microwave impulse method for cement paste](image)

![Calibration curve of the capacitance method for cement paste](image)

Fig. 4 Calibration curve of the capacitance method for cement paste
4 Experimental Results and Discussion

Figure 5 and Fig. 6 present typical moisture profiles in cement paste specimens measured by the microwave impulse method and the capacitance method. A comparison of both sets of curves shows three basic features. First, the agreement of results obtained by both methods is reasonably good for longer times but for short times from the beginning of the experiment it is worse. Second, the agreement is better for high moisture content than in the low moisture range. Third, the data scattering seems to be higher for the microwave impulse method than for the capacitance method.

However, an exact direct comparison of the measured moisture profiles is difficult in general. Due to the characteristic features of each technique the experimental data may be obtained for different positions and different time steps. Therefore, comparing a profile at a certain time step quite exactly would only be possible by an interpolation in the time domain. To overcome this problem the Boltzmann transformation was applied to the experimental data. For each technique the obtained moisture content versus distance profiles were replotted as a moisture content versus $\lambda$ profile, with

$$\lambda = xt^{-1/2}$$

If the Boltzmann conditions are fulfilled (a constant boundary condition applied to a semi-infinite homogeneous medium that is initially at uniform moisture content), all the measured moisture profiles should fall on a single $\lambda$-profile.

Fig. 5 Typical moisture profiles in cement paste specimens measured by the capacitance method

Fig. 6 Typical moisture profiles in cement paste specimens measured by the microwave impulse method
Figures 7-9 compare the Boltzmann transformed experimental data of the microwave impulse method, the capacitance method and the gravimetric method for all three materials. In all cases, the Boltzmann transformation seems to hold very well. However, a systematic difference between the microwave impulse method and the capacitance method in a comparison to the gravimetric technique can be observed. The microwave impulse method gives results closer to the gravimetric measurements in the range of highest moisture content. On the other hand, the capacitance method shows a better agreement with the gravimetric method for lower values of moisture content where samples are almost dry (naturally wet).

Fig. 7 Boltzmann transformed experimental data of the microwave impulse method, the capacitance method and the gravimetric method for cement paste

Fig. 8 Boltzmann transformed experimental data of the microwave impulse method, the capacitance method and the gravimetric method for ceramic brick
Fig. 9 Boltzmann transformed experimental data of the microwave impulse method, the capacitance method and the gravimetric method for autoclaved aerated concrete.

The reasons for these differences lie in the physical background of both methods. The microwave impulse method in the presented setup is a transmission-based technique. Its accuracy depends mainly on the precision of measuring the time difference

$$\Delta t_{21} = \Delta t_2 - \Delta t_1$$

(see above). The permittivity of bound water that prevails in the material in the range of low moisture content is about 20 times lower than the permittivity of free water. Therefore, for low moisture content the mentioned time difference is much lower than for high moisture content and the sensitivity and accuracy of the method is also substantially lower.

The accuracy of the capacitance method depends mainly on the achievement of an ideal contact of the sample surface with the probe because any appearance of air bubbles on the surface can damage the accuracy substantially (two capacitors in series, one of them with a very low permittivity of the dielectric). Once a good contact is achieved, the accuracy of the method depends just on the accuracy of voltage measurement that is only seldom a limiting factor. Therefore, the accuracy should be comparable for both high and low moisture content.

5 Conclusions

Both the microwave impulse method and the capacitance method were shown to be well applicable for the regular determination of moisture profiles in porous building materials. However, taking into account the particular advantages of each method, for the achievement of the highest possible accuracy the application of the microwave impulse technique can be recommended for the measurements where higher moisture content is expected and the capacitance method for lower moisture content measurements.

6 Acknowledgements

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Microwave Scanning Technology for Dielectric Material Testing

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ABSTRACT. Microwaves compared to other technologies are a nearly ideal tool for getting information about the permittivity distribution inside materials. They can penetrate all non–metallic materials up to some decimeters depth. Coupling into a material via air can be accomplished easily. Microwave materials testing systems can be designed to have very low electromagnetic power avoiding all dangers and hazards due to ionizing radiation. These and other positive features of microwaves are used in microwave scanners for materials testing. Such scanners are described in configurations for process and for laboratory applications. A very important part in microwave image processing of materials properties take filter algorithms. Microwave scanners have the potential for use in many applications.

Keywords: Microwave Scanners, Microwave Imaging

1 Introduction

For a broad and increasing variety of quality assurance applications better knowledge of materials inner structure is needed. Science and technology have identified a large number of non–destructive physical principles throughout the last 150 years, that seem to be suited for this. Many of them were transferred to commercially available devices and systems and can be operated as imaging or tomographic methods too.

2 Microwaves Versus other Imaging Technologies

All of these principles have their own fields of application but also have a number of disadvantages especially for testing volume materials. Optical imaging has found broad acceptance, but interacts only in a layer of a few microns at the surface of a material. Ultrasonic testing is a basic technology since decades and allows true volume information and spatial resolutions down to submicron range, but causes a lot of problems with coupling when non–contacting operation is required. X–rays can overcome this easily; their problem is the danger coming from the ionizing radiation leading to high and expensive requirements for safety measures. The same is valid for computer tomography based on radioactive materials. NMR doesn’t show all these problems, but is restricted either to closed magnetic systems like in medicine or, for the case of open systems, to a very limited depth of operation. Radar systems show a real volumetric interaction and therefore have been in use for the detection of inner properties of materials for about 15 years. Their problems are the large wavelengths used in common radar applications, which do not allow the identification of small cracks, and the very time consuming process of processing radar images after the measurement.

Microwave methods have the potential to overcome all these disadvantages. Microwaves show a real volumetric interaction with the dielectric material properties for all non–metallic materials up to some decimeters depth. They can work easily both in reflection and in transmission arrangements and therefore allow flexible configurations of test systems. For electromagnetic waves there is no big problem with coupling into a material through air. Microwave materials testing systems use very low electromagnetic power avoiding all dangers and hazards due to ionizing radiation. Microwave applicators can be designed for a
lot of special applications and penetration depths. All these positive features of microwaves lead to the development of microwave scanners for materials testing.

3 Microwave Basics

Microwave methods for the detection of spatial distribution of materials parameters in most cases are based on measurements of the complex dielectric permittivity of a test object.

$$\varepsilon_r = \varepsilon'_r + j \varepsilon''_r = \varepsilon'_r \left( 1 + j \tan \delta_e \right)$$  \hspace{1cm} (1)

Those dielectric parameters in principle can be measured in all microwave arrangements having stray fields or radiating electromagnetic energy by antennas into the material. In all cases an interaction between dielectric material and electromagnetic field or wave will happen. This interaction can be registrated by microwave applicators in reflection and transmission methods.

Using reflection methods electromagnetic energy is transmitted by a microwave applicator to the material. Both transmitted wave and reflected wave are separated and measured. The reflection coefficient is the ratio of reflected and transmitted wave:

$$\rho = \frac{u_{\text{reflected}}}{u_{\text{transmitted}}}.$$  \hspace{1cm} (2)

Reflection methods only need access to one side of the material. They are put onto the material or mounted in a small distance with fixed size. They allow integral measurement of permittivity across the depth of the material but also measurements with some depth resolution.

![Microwave reflection measurement](image)

Fig. 1 Microwave reflection measurement

When using transmission methods with transmitter and receiver sitting on opposite sites of the test objects, electromagnetic energy completely penetrates the material. Simple transmission arrangements allow integral measurement of the dielectric properties of the test object.

Laying a fictive grid over the surface of the test object and measuring in the grid points leads to at least two–dimensional images of the spatial distribution of permittivity and other physical properties derived from it. The measurement with reflection type applicator of fixed penetration depth gives the dielectric properties in one layer of the material.
A big advantage of microwave technology is that it can use a broad variety of electromagnetic principles. This way a large number of microwave applicator types can be designed. For the commonly used ISM–frequencies 2.45 GHz and 5.8 GHz the table below gives typical orders of field geometries, ranges and spatial resolutions.

Fig. 2  Lateral distribution of measuring volumina in one layer – Reflective Measurement

Table 1 Microwave applicator types and orders of their interaction geometry

<table>
<thead>
<tr>
<th>Applicator type</th>
<th>spat. Resolution</th>
<th>field range</th>
<th>measuring vol.</th>
</tr>
</thead>
<tbody>
<tr>
<td>strayfield linear (lines)</td>
<td>mm</td>
<td>mm</td>
<td>some mm³</td>
</tr>
<tr>
<td>strayfield linear (resonators)</td>
<td>mm</td>
<td>cm</td>
<td>up to some cm³</td>
</tr>
<tr>
<td>strayfield round, symmetrical</td>
<td>cm</td>
<td>cm</td>
<td>up to 100 cm³</td>
</tr>
<tr>
<td>radiation field, planar, not directed</td>
<td>cm</td>
<td>dm</td>
<td>up to 10 l</td>
</tr>
<tr>
<td>radiation field, planar, directed</td>
<td>cm</td>
<td>dm to m</td>
<td>up to 200 l</td>
</tr>
</tbody>
</table>

This shows that microwaves can generate interactions in various geometries even with applicators small in comparison to the test object dimensions.

Not only the spatial distribution of permittivity can be measured, but also a broad spectrum of material and object properties derived from them, like

– material moisture and leakages
– material density
– material inhomogeneities (cracks)
– geometry of layers and layered media
– presence and geometry of construction elements
– arrangements and filling levels in closed non–metallic packages
– properties of tissue
– leakages in packages.
4 Multidimensional Material Parameter Distributions

For getting information concerning the dielectric parameter distribution in the z-axis microwave applicators with different penetration depths can be used (see table 1). Such probes already exist in the MOIST moisture measurement system which microwave scanners are based on. MOIST contains probes with field ranges between centimeters and decimeters. For many applications such a gradation is sufficient for getting informations regarding inner structures.

![Distribution of measuring volumina with two different microwave applicators](image)

Fig. 3 Distribution of measuring volumina with two different microwave applicators

For a large part of measuring problems the number of layers to be investigated reduces to the surface layer and a volume layer in the core of the test object. Fig. 3 shows schematically how two different microwave applicators measure in the same grid points.

The strayfield applicators only interact with the surface layer. Following table 1 strayfield applicators can be open coax lines, striplines open to the sample or open resonators. The can also be designed as special applicators with higher field penetration of the sample. For these applicators it is possible by changes of geometry to change the volume of the electric and magnetic field and therefore the penetration depth.

The most open line structures are TEM or quasi–TEM lines. In many cases they are not symmetrical, but the decrease of the electrical field is similar to

\[ E, H \sim \frac{1}{r^2} \]  \hspace{1cm} (3)

with \( r \) being the distance from the line. Some times the exponent of \( r \) is even higher than 2. Other structures like open resonators show an exponential decrease of electric and magnetic field.

The radiation field applicators interact with the surface layer too but most part of the microwave radiation passes through this layer and therefore it causes only small interaction. This can be demonstrated easily using a simple Hertzian dipole as example: For it’s transversal component of the electric field \( E \) is derived from Maxwells equations:
\[ E_{\varphi} = j Z_0 \frac{i \Delta l \sin \vartheta}{2 \lambda} r e^{-\frac{j 2 \pi}{\lambda}} \left( 1 + \frac{j}{\frac{2 \pi}{\lambda}} + \frac{1}{(j \frac{2 \pi}{\lambda})^2} \right) \] (4)

The brackets on the right side contain three terms. The first one stands for the far field of the Hertzian dipole. The other two terms describe the near field.

From (4) can be seen, that the electrical far field is decreased with \(1/r\)

\[ E_{\varphi, \text{farfield}} \sim \frac{1}{r}, \] (5)

while the near field follows \(1/r^2\) and very close to the radiator \(1/r^3\). Similar equations can be given for the other components of electrical and magnetic fields.

For other types of antennas the variation of the components of electrical and magnetic fields are described by radiation patterns. In case the radiation is concentrated into one direction – like for a directional antenna – this leads to a total decrease of the E–field which is different from (5).

On the other hand applicators can be designed which are somewhat mixed from strayfield applicators and radiation field applicators. Therefore a more general description of the decrease of the electromagnetic fields is

\[ E, H \sim \frac{1}{r^\alpha}, \] (6)

where the exponent \(\alpha\) theoretically can range between zero and infinity. In practical applications this range will be much lower, but in principle it allows the design of various applicators with different penetration depths and their combination.

Applying this to the problem in fig. 3 it is possible to conclude to the permittivity distribution in the volume, because the permittivity distribution of the surface is known from the strayfield measurement.

As shown above this principle can be transferred to more than two layers. It allows to realize arrangements with a minimum depth resolution in steps of some mm for the commonly used ISM frequencies and maximum step sizes in z-direction in the decimeter range.

5 Microwave Scanners

Many applications look for structural investigations of test objects under laboratory conditions with stationary test equipment. For these purposes the laboratory scanner EM VISION LAB was developed. In this scanner the microwave probes can be moved in all three axis. Driving elements for the axis are electronically controlled stepper motors.
The laboratory scanner is available in different configurations. The basic configuration was designed for reflective measurements with the necessary microwave probes working parallel. This arrangement actually allows up to 5 measurements per second. A test object with dimensions of 100 cm x 60 cm can be measured within 5 minutes with a lateral resolution of 2.5 cm. For transmission measurements a second y-axis can be added running parallel to the first one.

In process applications with webs moving at higher speed mechanical drives are too slow and too trouble-prone. For such tasks arranging of the microwave probes in an array is recommended.

![Laboratory Scanner EM VISION LAB](image)

Fig. 4  Laboratory Scanner EM VISION LAB

The microwave sensors act somewhat like optical sensors or line cameras as pixels. For a lateral resolution of 10 mm on a moving web of 2000 mm width it is necessary to have at least 64 microwave probes of each type. Mounting this array on both sides of the material leads to a complete observation of the whole cross section of the material, if the web is not too thick. A process scanner fulfilling these requirements is shown in fig. 5. Both the upper and the lower scanner module can be varied in height over the moving web.

![Microwave Process Scanner EM VISION PROCESS](image)

Fig. 5  Microwave Process Scanner EM VISION PROCESS
6 Image Processing with Microwave Scanners

The high amount of data generated with a microwave scanner increases the meaning of data processing for the desired applications. In many cases certain morphological features of the test object like density variations caused by the manufacturing process are superimposed the desired structure information. Therefore filtering of the generated images becomes a very important part of post-processing.

<table>
<thead>
<tr>
<th>Sample 013_3</th>
<th>Volume upper side</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rough data</td>
<td>Moisture disturbances included</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Filter type</th>
<th>Results – Sample 013_3 Volume upper side</th>
</tr>
</thead>
<tbody>
<tr>
<td>3x3default</td>
<td></td>
</tr>
<tr>
<td>Sobel</td>
<td></td>
</tr>
<tr>
<td>5x5_loc.Max</td>
<td></td>
</tr>
<tr>
<td>_normal</td>
<td></td>
</tr>
<tr>
<td>5x5_P-Median_y</td>
<td>(best filtering results)</td>
</tr>
</tbody>
</table>

Fig. 6 Rough data and results of different filter algorithms

The image shows a typical volume scan at a test object with moisture disturbances included. From rough data it is not possible to decide which part of the image changes due to structural changes and what is moisture caused. For being able to make a clear decision it is necessary to have one ore more filtering steps.

Solving such a problem the first step will be to observe the basic structure of the non-disturbed material in order to characterize it’s special morphological features. If it’s possible to find filter algorithms that identify this basic structure cracks and disturbances can be found with high probability. Fig. 6 shows the results of some filter algorithms. Only some of them give a good differentiation between moisture and material structure.
7 Applications

There are manyfold applications for such a technology in production processes, civil engineering, test of packagings and even in automotive applications like scanning of car seats. One main application is concerning structural, moisture or density investigations at moving webs. Such materials (chip board, laminates, composites, mineral wool) show similar problems in most cases. Either the moisture distribution is to detect, or a detection of cracks in the material is sought for, which can be derived from density or moisture distributions.

Another field of application is packaging technology. The questions to be answered there are position or completeness of goods in closed non-metallic packages or the detection of leakages in bags filled with aqueous liquids packed in cartons. In medicine technology such bags are used for the transport of saline water. In some cases small leakages occur at the port system of such bags leading to rejection of the whole carton. Imaging the cartons with a microwave scanner from the bottom side while moving along a conveyour belt finds small leakages.

For structural imaging of moisture and density distribution in ceramics and glasses the lab scanner is a good solution.

Microwave scanners can also be used for tasks which seem to have nothing in common with the measurement of permittivity distributions, like moisture measurements in car seats. A non-destructive moisture measurement is difficult because of the metallic bottom of the seat. Microwave Imaging generates typical reflection patterns for the seat. A change in these patterns can be correlated with moisture content.

8 Conclusion

Microwave scanners are imaging systems which generate two-dimensional and quasi-three-dimensional structure investigations. They can be regarded as microwave cameras with volume interaction and multidimensional spatial resolution. Because of the outstanding features of this technology a very broad variety of applications can be expected. For different kinds of applications a selection of ready-to-run configured scanners is already available.

References


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Measurement of Continuous Drying out of Subterranean Concrete Walls

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ABSTRACT. The present investigation describes the measurement of subterranean concrete walls using TDR-techniques. The probes are specially constructed for the permanent use in the building and were scanned during a period of 3 years. The water content is derived by the application of a 4-phase dielectric mixing approach, where hydration can be taken into account. The metamorphosis of the concrete is integrated by the electrical conductivity as an indicator of the total dissolved components. Exemplarily the single data are presented for wall elements of different level floors of the building. The differences between wall and ceiling of the respective levels and the mean values reflect the drying behavior of the average building.

Keywords: TDR, mixed dielectric approach, concrete research

1 Introduction

The new construction of the Saxony State and University Library in Dresden 1999 is a representative and honored design of the Architects Ortner and Ortner. The library has a build in volume of 180000 m³, where 90% of the building is placed underground. The building construction with its 3 floors is located 12 m below the terrain and is laying 9 m below groundwater. The volume of large and modern libraries consist of acclimatized magazines, where books are stored in open bookshelves. Due to modern transportation techniques the magazines are not necessary places of work and do not require illumination. Day light or continuous artificial light of strong energy is unauthorized.

Especially the storage and the conservation of digital media, photographs and art have high requirements on the climatic conditions. Beside the amplitude of relative humidity and temperature little tolerance exist in respect to these threshold values. The effort of the acclimatization is as smaller as more mass of the construction is coupled to the ground.

A subterranean, window free construction is leading to problem of drying out the concrete building. These drying out has to be economically organized, predicted and controlled [3]. If the magazines are placed in the groundwater, a number of problems due to the packing and their control have to be solved. Control and planning of the drying out of the S.L.U.B. requires the measurement of water content in the concrete. This was realized by embedded TDR-probes and continuous monthly measurements.

2 Measurement Technology and Probes

The TDR-Method for measurement of moisture uses the fact, that water (~80) has a dominant larger relative dielectric permittivity than solid mater (~5) or air (~1). Beside other properties, this result in the velocity of an electro magnetic pulse, propagating through a material. As more moisture a material has, as larger is the apparent dielectric constant and as slower is the travel time of the electromagnetic pulse. During traveling of the pulse in the sensor-cable system reflections appear at the beginning and the end of the sensor. They can be visualized at the monitor of an oszillograph. In the TDR-device the recorded voltage-time pairs are treated
in a special manner. The reflectogram is interpolated by a smoothing function and then interpreted in TDR-manner, where specific probe data are accounted for. More details on the used technology can be found by [6] [7] [8].

For the interpretation of the measurement the porosity of the building material and the volume fractions of the different phases have to be known. In a calibration experiment at samples of the B35 concrete of the S.L.U.B. moisture content, porosity and solid fraction dielectric constant and content was measured by gravimetry and TDR as well. In order to indicate the change in the chemical composition of the concrete during hardening, the specific electrical resistance was measured by TDR too. The method is basing on voltage deviation at two resistances, where one resistance corresponds to the TDR sensor and the other one to a reference resistor. Additionally Ntc sensors are places into a sensor body for temperature measurements. The measurement is executed, controlled and interpreted by an internal computer, where a LCD-display allows the monitoring of results and the reflectogram for visual control. The TDR-sensors are selected manually via BNC-connectors.

**TDR probes**

The TDR-probes are specially designed for the experimental application in the S.L.U.B. The sensitive part consist of two metal rods of 2 mm diameter, 16 mm distance and 100 mm length, soldered on a pc-board and fixed into a plastic tube. The two-wire sensor is connected via coaxial cable to a BNC-connector, which is placed in a build over box, so that the installation is not visible.

![TDR-sensor diagram](image)

**Fig. 1** A schematic draw of an installed TDR-sensor within a concrete wall.

For installation the probe is fixed at the predefined location. This is done using special fixings, where the plastic tube can be strongly bound at the iron steels. By heating the tube above 125°C using hot air, the plastic tube becomes flexible and can be placed at the exact position of installation. After cooling the plastic becomes stabilized.

The TDR-probe is vertically top wards oriented. During construction the concrete rises slowly from the bottom upwards, preventing a sensor deformation. Before installation the sensors were calibrated using defined liquids like water, benzene and different molar salt concentrations of known electrical conductivity. If the TDR-device is supported by these probe information, the apparent dielectric constant and the bulk electrical conductivity of the material can be derived directly [6] [7].
3 Determination of Dielectric Water Content by TDR

To derive the volumetric water content of the concrete directly from the TDR-measurements, the relationship between apparent dielectric constant and water content of the porous material have to be known in advance.

Mixed-dielectric Approaches

In the simplest form this relationship for the respective capillary porous material can be described using a dielectric mixing law of the three phases: liquid, solid, and vapor. Since free water, has a very high dielectric constant of $\varepsilon_w \approx 80$ in comparison to mineral constituents ($\varepsilon_s \approx 4-5$) or air ($\varepsilon_a \approx 1$), it is possible to find a functional relationship. The mixed-dielectric approach for 3 phases can be formulated according to [9]:

$$\varepsilon_a = [\theta \varepsilon_w^\beta + (1-\phi)\varepsilon_s^\beta + (\phi-\theta)\varepsilon_g^\beta]^{1/\beta}$$

where $\varepsilon_w$, $\varepsilon_s$ and $\varepsilon_g$ are the dielectric constants of the water-, solid- and the air phase. The parameters $\theta$ and $\Phi$ correspond to the water content and the porosity. From both, the volume fractions of all constituents can be determined. $\varepsilon_{bw}$ represents a geometry factor of the 3-phase mixture model and the influence of the orientation in the imposed electrical field. The parameter $\beta$ itself cannot be measured and is derived from reference measurements of water content and least squared minimization techniques.

An improvement of the 3-phase mixture model is the 4-phase dielectric mixture approach, where the bound water fraction and the corresponding dielectric constant are accounted for [2]:

$$\varepsilon_a = [(1-\theta_{bw})\varepsilon_{fw}^\beta + \theta_{bw}\varepsilon_{bw}^\beta + (1-\phi)\varepsilon_s^\beta + (\phi-\theta)\varepsilon_g^\beta]^{1/\beta}$$

Here the total water content can be calculated by the fractions of thermo dynamic ,,free“ $\theta$ and ,,bound“ water $\theta_{bw}$ where $\varepsilon_{fw}$ and $\varepsilon_{bw}$ are the corresponding dielectric constants. The amount of the bound water can be approximated by equation [1] using:

$$\theta_{bw} = l_{mw} S_{eff}$$

where $l_{mw}$ is the amount of mono molecular water layers having a thickness of $\delta = 3 \times 10^{-10}$. $S_{eff}$ corresponds to the effective specific surface area and is calculated as a product of specific surface area and bulk density. Due to [1] the dielectric constant decreases exponentially with the increasing thickness of water layers. For the first mono molecular water layer the apparent dielectric constant is given by some authors $\varepsilon_{bw} = 3.2$ [1], $\varepsilon_{bw} = 6$ [5], and $\varepsilon_{bw} = 20-40$ [2]. For the determination of the parameters of the 3- and 4-phase dielectric mixture models, a number of material specimen were prepared using the B35 concrete of the S.L.U.B. At the samples the relationship between the apparent dielectric constant and the volumetric water content was determined using TDR and the gravimetric method as a reference. The model parameters $f_{bw}$, $\theta_{bw}$ and the geometry parameter $\beta$ were simultaneously determined using least squared minimization technique. The found parameter combination is given in table 1.
Table 1  Parameter combination of the 4-phase dielectric mixture model for the B35 concrete of the S.L.U.B.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>dielectric constant of solid phase, $\varepsilon_s$</td>
<td>4.28</td>
<td>-</td>
</tr>
<tr>
<td>dielectric constant of gaseous phase, $\varepsilon_g$</td>
<td>1.005</td>
<td>-</td>
</tr>
<tr>
<td>dielectric constant of bound water phase, $\varepsilon_{bw}$</td>
<td>6.3</td>
<td>-</td>
</tr>
<tr>
<td>dielectric constant of free water phase, $\varepsilon_{fw}$</td>
<td>80.36</td>
<td>-</td>
</tr>
<tr>
<td>porosity</td>
<td>0.166</td>
<td>m$^3$/m$^3$</td>
</tr>
<tr>
<td>bound water fraction</td>
<td>0.048</td>
<td>m$^3$/m$^3$</td>
</tr>
<tr>
<td>geometry parameter</td>
<td>0.875</td>
<td></td>
</tr>
</tbody>
</table>

The results of the TDR-measurements and the bulk electrical conductivity are shown in figure 1 and 2. The TDR-measurements and the electrical conductivity data as well indicate a strong reduction of values during the first days of drying. The water content decreases from 0.45 m$^3$/m$^3$ to 0.18 m$^3$/m$^3$. Since the total porosity of the hardened B35 concrete is 0.166 m$^3$/m$^3$ only, in terms of dielectric permittivity the solid phase is partly liquid.

The dielectric constant of the bound water phase was found to be $\varepsilon_{bw} = 6.3$, supporting the investigations of Phillips 1975. The electrical conductivity as an indicator for the total dissolved components, decreases in the same time interval for about one order of magnitude. The stiffness of the function indicates the velocity of the process and the dimension of the change in the chemical consolidation of the concrete.

The comparison between gravimetric water content and the TDR-measurement of the calibration experiment is presented by figure 3. Here the 3-phase-dielectric mixing model indicate a weak accordance in respect to the gravimetric reference data, given by the 1:1 line in the graph. In the initial phase the hardening of the concrete is not represented and in the dry range the stronger bound crystal water is not accounted for, where the total moisture content is under estimated.

Fig. 2 Calibration results: Measured volumetric water content by TDR during the 450 days drying out of the B35 concrete of the S.L.U.B.
Contrary the 4-phase-dielectric mixing model delivers an adequate description of water content after consolidation of the concrete. A large amount of water is used for hydration in the beginning of the consolidation. This stronger bound water cannot be oriented in a polarizing electromagnetic field and has a significant lower dielectric permittivity and was found to be $\varepsilon_{bw} = 6.3$. Deviations exist in the range of high ion concentrations, where the chemical composition of the concrete is continuously changing. The temporal process of hardening of the concrete is visualized by comparing the figures 1, 2 and 3, where the dielectric consolidation is reached after 50 to 60 days. There the electrical conductivity reaches values below 0.1 ms/cm.

![Fig 3 Calibration results: Measured bulk electrical conductivity by TDR during the 450 days drying out of the B35 concrete of the S.L.U.B.](image1)

![Fig 4 Comparison of the tested dielectric mixing models for the B35 concrete of the S.L.U.B.](image2)

In a first empirical approach, the influence of the chemical metamorphosis of the concrete is accounted by the bulk electrical conductivity. The moisture content is calculated using the 4-phase-dielectric mixing model according to the following equation:

$$\theta = \theta_{(4\text{-Phasenmodell})}(1 - \sigma^2)$$
Where $\sigma$ represents the measured bulk electrical conductivity of the porous concrete. The corresponding calibration function is presented in figure 3 and shows a good accordance between the gravimetric reference measurements.

4 Drying out of Subterranean Concrete Walls

In total, 40 TDR-probes are installed during the construction phase of the subterranean building. After the availability of the probes for measurement the subsequent installed TDR-probes were scanned every month during 3 years. The yield data are recalculated using the 4-phase-dielectric mixing in combination with the bulk electrical conductivity correction.

During the construction of the building, these data were used to control and economically organize the drying out of the subterranean library. If increasing moisture content was measured at a specific location, suitable measures were used for drying out. Applied drying techniques are the usage of dry and warm air with flexible wind channels and the application of 150 condensation dryers.

The investigations were quite successful. From 40 TDR-probes which have been installed, 3 sensors were destroyed during covering with concrete, 3 TDR-probes became defect as a consequence of connector problems and 1 sensor could not be found for measurement. The profit of the installation procedure is 83%. At a price of about 300€ per TDR-probe the fix costs for all sensors are approximately 12,000€ to control the drying out.

![Graph](image)

Fig. 5 Volumetric water content of the B35 concrete of the bottom level of the S.L.U.B. during a drying out of 3 years. Results measured by TDR and using a 4-phase dielectric mixing models with bulk electrical conductivity correction.

Representative for all individual measurements of volumetric water content, bulk electrical conductivity and temperature, the results of the moisture content measurements in the concrete walls of the bottom level (-2) are given in figure 4. The single data show the expected deviation of 0.05 m$^3$/m$^3$. The initial phase is characterized by a fast decrease of moisture during the winter time. In the end of autumn and beginning winter, all openings of the building were closed and the drying out was realized by heated air from outside. The organization of the drying is basing on TDR measurements.

As a consequence of the mild spring and the necessity of saving money, the outside air heating was switched off and the closed ventilations and openings were opened by the building
contractors. Due to the incline position of the building in the landscape, warm and moist outside air entered the construction and left condensate in the subterranean building after the cooling. Thus from March until July 2000 the water content in the construction show an increase in moisture especially at the openings as identified by the figures 4, 5 and 6. TDR-probes located away from the openings indicate no moisture increase (location 1 and 5 in figure 4). After the closing of the ventilation holes and openings and starting the application of the drying procedures, the moisture content is decreasing. Summarizing, the saving of heating in combination with the outside ventilation was leading to an increase of a total moisture of the construction of about 0.005 m³/m³. This is equivalent to a drying time of 8 month.

![Graph](image)

Fig 6 Mean volumetric water content of the B35 concrete of the S.L.U.B. during a drying out of 3 years. Comparison of different levels, walls and ceilings: the bottom level -2 (walls), bottom level -2 (ceiling), level -1 (ceiling), top level -0 (walls), top level -0 (ceiling).

The further courses of the different walls and ceilings are characterized by the yearly fluctuations and the drying measures. While for planning and organization of a controlled drying out the knowledge of the individual measurement positions in the building are required, the mean values for each level deliver a good overview on the moisture situation.

A comparison between walls and ceilings in the respective levels is given by figure 5. The highest moisture content can be found in the bottom level (-2), where the drying is very slow and will take many more years. Walls and ceiling have comparable moisture. Here the effect of condensation of outside air, coming from ventilation during March until July 2000, on the moistening of the concrete can be seen obviously. The lowest temperatures of about 3°C to 5°C could be measured during this time in level (-2) and deliver the explanation for the strong condensation.

The top level (0) is characterized by the lowest moisture content, because the ventilation was best in comparison to the other levels. This elevation illustrates clearly the influence of the close outside climatic conditions by the large variations in water content. The deviation of moisture in a yearly rhythm is the indicator.

The walls in the top level (0) are slightly dryer than in the bottom level (-2). This implies, that the walls of the S.L.U.B. are continuously drying in the same degree. In the middle level (-1) no wall probes have been installed. The comparison of wall and ceiling sensors show, that the ceilings of the top and middle levels (0 and -1) have about 0.01 m³/m³ less moisture than the walls.
An overview of the total moisture conditions is given by the building trend, where all measurement are averaged. For the period of 3 years, the bottom level (-2) dried by 0.04 m³/m³. In the same time the water content in the top and middle levels (0 and -1) decreases by 0.04 m³/m³. The effect of the already installed heating and climatisation system of the building can be seen by the moisture decrease in September 2001. After 3 years of measurement the hole building construction gained a drying level of 0.10 m³/m³.

Fig 7 Mean volumetric water content of the B35 concrete of the S.L.U.B. during a drying out of 3 years. A total moisture in building trend.

References


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TDR Technique for Measuring the Moisture Content in Brick

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ABSTRACT. This paper presents results of the measurements of capillary water uptake of brick using the TDR method, which is based on measuring the dielectric constant of wet brick. TDR test results are compared with weighing of brick samples. Results show a good agreement between both methods. The TDR method is appropriate for in situ measurement of the moisture content in brick walls.

Key words: moisture content, TDR, brick

1 Introduction

Brick masonry walls have been an integral building component in western civilization since many centuries. Most residential and historical buildings in The Netherlands utilize these walls and they are still being used today because of their durability, availability and aesthetic appeal. Moisture poses the biggest threat to the integrity and durability of masonry walls, accounting for up to 90% of damage in building envelopes. Moisture originates either from rain or humid air. Many theoretical models have been proposed to predict the transfer of moisture in building envelopes. Researchers and building partitions have a significant need for in situ moisture content sensors to help them to understand the problems associated with excess moisture accumulation in building envelopes, and accurate sensors are needed as a research tool to verify the results of these models. Review of literature shows that since the low cost, accuracy, remote reading and durability of sensors is to be concerned, then the most promising techniques for moisture detection in building materials is the use of electrical capacitance or dielectric constant. This article examines if Time Domain Reflectometry (TDR) method is suitable for measuring moisture content in bricks. Up to now, TDR has been used in agriculture for measurements of the moisture content in grains and soils, and a few field surveys documenting moisture measurements with TDR technique are reported for determination of moisture content in building materials. For example, Hansen and Hansen (2002) [2] used TDR for aerated concrete. They concluded that TDR can be used to measure the moisture content in aerated concrete with a dry density of 500 kg/m³ when moisture content is higher than 0.12 %, corresponding to 90% RH. It was found that the water absorption of the tested aerated concrete is 0.0055 kg/m³ per square root of time. They also suggested reproducibility of capillary water uptake test should be increased in order to achieve better results.

In the HAMSTAD-project [3], moisture content of three building materials i.e. autoclaved aerated concrete, ceramic brick and calcium silicate plate were examined. Test on different characteristics of building materials were done with different methods in different laboratories. TDR was one of these methods. It showed a good agreement for water uptake of calcium silicate, but for autoclaved aerated concrete and ceramic brick, results deviated from results of other methods. Research suggested that these different results can be attributed to the contact problem between TDR-probes and the material.

Groot [1] used TRIME, TDR sensors for the determination of moisture content in concrete. He concluded that the TDR method performs satisfactorily in fine and low porous materials like concrete, where a uniform distribution of moisture is to be expected. He also suggested
that the application of this method in determination of the water content in monuments, for the purpose of restoration, is very promising. A full-scale research is ongoing at the south-west façade of the Vertigo building at Eindhoven University of Technology (TUE), where the same weather condition is applied on various types of brick walls. In that study, brick wall panels will be equipped with TDR moisture sensors, if they work satisfactorily.

2 Sample Preparation

In this study, three different measurements are conducted; first determination of the region of the measurement of TDR-probe; second capillary water uptake measurements using TDR-probe simultaneously with weighing of samples and third to determine the behaviour of the TDR-probe when there is a moisture gradient. Therefore, different samples are prepared (see figure 1). Samples used for this research are sawn from 210×650×110 mm³ bricks. They are delivered from TERCA, brick factory in Opheusden, The Netherlands. Bricks with bulk density about 1800 kg/m³ are used for this study.

2.1 Samples Preparation for the Region of the TDR-probe Measurement

In order to determine the region of measurement of TDR-probe, four brick samples of 110×40×40 mm³, 110×45×45 mm³, 110×50×50 mm³ and 110×55×55 mm³ are prepared. All the lateral sides and bottom of the samples are sealed with epoxy resin and only tops are open for inserting the TDR-probe. To place the TDR-probe in four samples, one pair of parallel holes each with a 3 mm diameter and separated by 16 mm, is drilled with a depth of 100 mm.

2.2 Samples Preparation for Capillary Water Uptake

Five samples of 110×50×50 mm³ are prepared for capillary water uptake measurements. In each of these five samples, one pair of parallel holes each with a 3 mm of diameter and separated by 16 mm, is drilled with a depth of 100 mm. All the lateral sides of the samples are sealed with epoxy resin and only tops and bottoms are open.

2.3 Samples Preparation for Measurements with a Moisture Gradient

Five samples of 110×50×50 mm³ are sawn into three parts each. These fifteen samples are prepared for measurements where each part can have a different moisture content. In each of these fifteen samples, one pair of parallel holes each with a 3 mm of diameter and separated by 16 mm, is drilled with a depth of 33 mm.
Fig. 1 Samples used for measurements of the capillary water uptake and moisture content using TDR-probe

3 Measurement Equipments

The 2-rod FP/mts/ns mobile probe and FOM/mts TDR portable reading measurement device developed by EASY TEST, Ltd of Lublin, in Poland is used for this research. The equipment is borrowed from the University Technology of Dresden, Germany (Building Physics laboratory, Rudolf Plagge). This TDR device originally is used in agriculture for determination of the moisture content in bulk materials such as grains and soils. Water uptake absorption of brick samples is also measured by an accurate weighing balance with a resolution of 0.001 gram. Labview software is used to read and record data from weighing of samples on a PC.

3.1 Principle of TDR

An apparatus which is based on the TDR principle launches electromagnetic waves and then measures the amplitudes of the reflections of the waves together with the time intervals between the waves are launched and the reflections are detected. Using the length of the probe, the velocity of the wave is computed. The velocity depends on the dielectric constant and the magnetic permeability. Assuming the magnetic permeability to be close to 1, the velocity depends only on the dielectric constant, which varies strongly with the water content of the medium surrounding the probe. Thus, from the transit time the water content can be detained.

4 Measurement Method

Samples for all three experiments are dried at the temperature of 105 °C for one week. The TDR reading for dry air is -5.2 % and for water 97 %. The TDR reading for dry brick is 2 %.
4.1 Method for the Region of Measurement

Four different sizes of brick samples are tested in water in order to determine the region of the measurement of TDR-probe. For this, samples are placed in the water in a way that the water does not reach the surface of sample. Measurements show TDR-probe readings are different if the size of sample is less then 50 mm. It can be concluded that the effective region of TDR-probe is a cylinder having approximate diameter of 50 mm and height of 110 mm, circumferenced around the sensor rods, which beyond of this region, changes in water content do not markedly affect readings of moisture. Therefore, for the measurements of water capillary uptake, only five samples with the sizes of $110 \times 50 \times 50$ mm$^3$ are chosen for further tests. For one dimensional capillary water uptake of five samples the epoxy resin is removed by sawing a layer of 2 mm from the bottom.

4.2 Method for Capillary Water Uptake

Five samples are tested for capillary water uptake. Samples are hung into the water from a weighing balance connected to a pc for the reading of water uptake. Measurements of the TDR-probe are also recorded during the water uptake simultaneously.

4.3 Method for Moisture Content Gradient

A known amount of water is added to fifteen dry samples. They are then placed in closed plastic bags for the period of two weeks to let the moisture, be absorbed and distributed homogeneously. The TDR-probe reading is recorded in the case the probe is put through three brick parts each with a different moisture content.

5 Measurement Results

The results of capillary water measurements uptake with TDR as well as weighing measurements are given in figure 1. The moisture content and TDR measurements is shown against the time.

![Fig. 2 Comparing weighing measurements with TDR](image)
Result shows that TDR-probe is only able to read the moisture content about 2%. This revolves that below this moisture level the dielectric constant of brick is too close to the dielectric constant of dry material, which TDR sensors are unable to distinguish.

For the measurements of the moisture content when a moisture content gradient is present, the results are given in table 1. It appears that the TDR results are smaller than the average moisture content for high moisture contents and larger than the average moisture content for low moisture contents. This agrees with the results of figure 2.

Table 1 Measurements of different combinations of different moisture content with TDR

<table>
<thead>
<tr>
<th>Sample</th>
<th>Moisture content sample (%) no: 1</th>
<th>Moisture content sample (%) no:2</th>
<th>Moisture content sample (%) no: 3</th>
<th>Moisture content sample (%) no: 4</th>
<th>Moisture content sample (%) no: 5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Part 1</td>
<td>15</td>
<td>7.5</td>
<td>22.5</td>
<td>4.5</td>
<td>0</td>
</tr>
<tr>
<td>Part 2</td>
<td>15</td>
<td>7.5</td>
<td>22.5</td>
<td>4.5</td>
<td>1.5</td>
</tr>
<tr>
<td>Part 3</td>
<td>7.5</td>
<td>4.5</td>
<td>15</td>
<td>1.5</td>
<td>0</td>
</tr>
<tr>
<td>Average Moisture content</td>
<td>12.5</td>
<td>6.5</td>
<td>20</td>
<td>3.5</td>
<td>0.5</td>
</tr>
<tr>
<td>TDR result (%)</td>
<td>7.7</td>
<td>5.7</td>
<td>15</td>
<td>4.6</td>
<td>2.9</td>
</tr>
</tbody>
</table>

6 Caution for Further Measurements

For different TDR sensors, which are used for water capillary uptake of brick samples, results show a small difference between measured values. This difference could be from improper contact in drilling holes, between the sensor pins and the material. Some other conditions may also have influenced the measurements such as temperature, shape of the samples and etc. Therefore, reproducibility of tests for accurate moisture content measuring especially for calibration tests should be considered.

7 Conclusion

The water content of bricks can be measured using TDR-probes. Therefore TDR sensors are appropriate sensors for in situ measurements of moisture contents in brick walls.

Acknowledgments

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Measuring Moisture Profiles in FGD Gypsum Using the TDR Method

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ABSTRACT . The application of time domain reflectometry (TDR) technique for measuring moisture profiles in flue gas desulphurization (FGD) gypsum is presented in the paper. The experiment is arranged in the form of vertical suction of water into naturally dried block-shaped samples. Determination of moisture content from measured apparent relative permittivities is done using three different calibration functions. The obtained results are compared, and the differences in moisture profiles determined by different techniques are discussed. Finally, the Matano method is employed for calculation of moisture dependent moisture diffusivity from the measured moisture profiles.

Keywords: TDR method, moisture profiles, FGD gypsum, moisture diffusivity

1 Introduction

Presence of water in all its phases has negative influence on thermal and hygric performance of porous building materials. Therefore, complete understanding to phenomena of moisture transport in porous matrix is very important, especially regarding the durability and functionality of building structures. Neglecting the influence of gravity force on moisture transport (see [1] for details), the liquid moisture transport can be expressed by classical diffusion equation [2]. In this equation, the moisture changes are related to moisture diffusivity coefficient, which is expressed as a function of moisture content.

The liquid water diffusivity is commonly determined from transient moisture profiles, which are measured in a capillary absorption experiment. In this experiment a specimen is at the bottom side brought in contact with a free water level and water is taken up due to capillary suction. Moisture contents are measured at different positions and time intervals during the experiment producing moisture profiles versus time. Gamma-ray attenuation technique, NMR-imaging, capacitance technique and microfocus X-ray radiography have proven to be reliable techniques for the non-destructive quantitative determination of moisture profiles. For calculation of moisture diffusivity, several methods of inverse analysis of moisture transport were recently proposed, calculating the moisture diffusivity either from a single moisture profile in specific time or from a set of moisture profiles. Typical representatives of both types of methods are the Matano method utilizing the Boltzmann transformation [3] and the double integration method [4].

In this paper, the time domain reflectometry (TDR) technique originally designed for application in soil science, is employed for monitoring moisture changes in samples of flue gas desulphurization (FGD) gypsum. The details of the measuring method are described, including the sensors calibration and moisture content evaluation from measured apparent relative permittivities. On the basis of measured results, the calculation of moisture diffusivity coefficient is done using the Matano method, which in previous work has proved to be very reliable for inverse analysis of measured moisture profiles.
2 Experimental

2.1 Time Domain Reflectometry (TDR) Method

TDR method can be generally classified as a dielectric method, based on an analysis of the behavior of dielectrics in a time-varying electric field, and consists in the measurement of permittivity of moist porous media. The determination of moisture content using the permittivity measurements is based on the fact that the static relative permittivity of pure water is equal to approximately 80 at 20°C, while for most dry building materials it ranges from 2 to 6.

The permittivity of materials is strongly affected by the orientation of molecules in the electric field. This characteristic is high for water in gaseous and liquid phase, but is significantly lower for water bound to a material by various sorption forces, which makes the orientation of water molecules more difficult. This feature makes it possible to distinguish between the particular types of bond of water to the material using the permittivity but on the other hand, it results in the dependence of the sensitivity of moisture measurements on the amount of water in the material. The relative permittivity of water bonded in a monomolecular layer is approximately 2.5, but for further layers it increases relatively fast. Therefore, the dependence of relative permittivity on the moisture content is generally characterized by a more or less gradual change at the transition from a monomolecular to a polymolecular layer. Consequently, the methods of moisture measurements based on the determination of changes of relative permittivity have low sensitivity in the range of low moistures where is their application rather limited.

In principle, a device based on the TDR technique (see e.g. [5]) launches electromagnetic waves to the sensors placed into the measured material and then measures the amplitudes of the reflected waves together with the time intervals between launching the waves and detecting the reflections. Because of demand on accurate length of each particular sensor, the sensors have to be calibrated before their placing into the studied material. In this paper, calibration of each sensor was done by measuring time of wave’s propagation in water and benzene. From the measured time intervals $t_s$ and the known relative permittivities of water $\varepsilon_w$ and benzene $\varepsilon_b$ the lengths of the particular sensors were determined according to equations (1)-(4):

\[
\sqrt{\varepsilon_w} = \frac{c}{2l_p} (t_w - t_{\text{ref}}) \tag{1}
\]

\[
\sqrt{\varepsilon_b} = \frac{c}{2l_p} (t_b - t_{\text{ref}}) \tag{2}
\]

\[
t_{\text{ref}} = \frac{\sqrt{\varepsilon_w} t_b - \sqrt{\varepsilon_b} t_w}{\sqrt{\varepsilon_w} - \sqrt{\varepsilon_b}} \tag{3}
\]

\[
l_p = \frac{c}{2} \frac{t_w - t_b}{\sqrt{\varepsilon_w} - \sqrt{\varepsilon_b}} \tag{4}
\]
where \( c \) is the velocity of electromagnetic waves in vacuum, \( t_w \) the time of wave’s propagation in water, \( t_b \) the time of wave’s propagation in benzene. On the basis of basic theory of electromagnetism and neglecting the imaginary part of complex relative permittivity we obtain from TDR measurements so called apparent value of the relative permittivity \( \varepsilon_a \) expressed by relation (5):

\[
\sqrt{\varepsilon_a} = \frac{c}{2l_p} (t_{\text{probe}} - t_{\text{ref}}).
\] (5)

In this paper, we used the TDR device LOM/RS/6/mps of Easy Test, Poland, which is based on the TDR technology with \( \sin^2 \)-like needle pulse having rise-time of about 200 ps, in the experimental work. It is computer aided instrument [6], originally designed for soil moisture measurement. The built-in computer serves for controlling TDR needle-pulse circuitry action, recording TDR voltage versus time traces, and calculating the pulse propagation time along particular TDR probe rods and finally the relative permittivity of measured material.

We used a two-rod miniprobe LP/ms (Easy Test) for the instantaneous moisture profiles determination that was designed by Malicki et al. [7]. This probe (see Figs.1, 2) is designed for monitoring changes in water and salt distribution in the material.

Fig. 1, 2 LP/ms minihygrometry probe

The sensor is made of two 53 mm long parallel stainless steel rods, having 0.8 mm in diameter and separated by 5 mm. The probe cable length from the sensor to the multiplexer is 1 m and cable feeder length from the multiplexer to LOM is 3 m. The sphere of influence was determined with the help of a simple experiment. The probe was fixed in the beaker and during the measurement, there was added water step by step. From the measured data (relative permittivity in dependence on water level) there was found out that the sphere of influence creates the cylinder having diameter about 7 mm and height about 60 mm, circumference around the rods of sensor. The accuracy of moisture content reading is ± 2% of displayed water content, in the range of measured moisture content 0-100%. The accuracy of moisture content measurements guaranteed by producer is restricted to materials having electrical conductivity ≤ 0.3 Sm\(^{-1}\).
2.2 Sample Arrangement, Measuring Technology

The experiment was arranged in the form of vertical suction of water into naturally dried samples of flue gas desulphurization (FGD) gypsum in air-conditioned laboratory at 23 ±1°C and 30 ± 2% relative humidity. The sample size was 70x50x330 mm. At first, samples were water- and water vapor proof insulated with epoxy resin on four lateral sides to ensure the one-dimensional transport. Sixteen TDR probes were installed into each sample during the process of casting the samples into the mould and connected through multiplexers with the TDR device. The moisture transport was then continuously monitored, and the experiment was stopped before the water suction has reached the end of the measured sample. Experimental setup is shown in Fig. 3a, b.

![Experimental setup for measuring moisture profiles using the TDR method](image)

3 Evaluation of Moisture Content from the Measured Apparent Relative Permittivity

There are three basic approaches to the determination of moisture content from measured relative permittivity. The first possibility is application of dielectric mixing models (e.g. [8]), which assumes knowledge of permittivity of the material matrix, permittivity of water, air and other parameters, that can not be measured directly, but have to be determined by empirical calibration of the model. The second possibility is utilization of empirical conversion functions generalized for a certain class of materials (e.g. [9]), which, however, are always limited for specific groups of materials only, for which they were proposed. The third method for evaluation of moisture content from measured relative permittivity consists in empirical calibration for the particular material using a reference method, such as the gravimetric method. This method is the most reliable until now but the most time consuming one.

In this work, we used for the calculation of moisture content from measured relative permittivity two empirically derived conversion functions proposed by Topp et al. [9] and
Malicki et al. [10] that were designed for monitoring moisture changes in soils. The Topp’s third order polynomial relation which is expressed by equation (6)

\[ \theta = -5.3 \times 10^{-2} + 2.92 \times 10^{-2} \varepsilon_a - 5.5 \times 10^{-4} \varepsilon_a^2 + 4.3 \times 10^{-6} \varepsilon_a^3 \] (6)

had for the originally studied materials standard error of estimate 0.0468 and was proposed for materials having the bulk density close to 1500 kg m^{-3}. The normalized conversion function proposed by Malicki et al. [10] is considered presently by many authors as universal for different types of materials. The Malicki’s function (7)

\[ \theta = \frac{\sqrt{\varepsilon_a} - 0.819 + 0.168 \rho - 0.159 \rho^2}{7.17 + 1.18 \rho} \] (7)

where \( \rho \) is bulk density of dry material, had for the originally studied materials standard error of estimate 0.0269.

For a validation of the two mentioned conversion functions for FGD gypsum studied in this paper, the calibration was also done by the gravimetric method. The calibration curve was determined for each sample. The final moisture profile was used for that purpose, which was determined by the gravimetric method as well. The sample was cut to 1 cm wide pieces, and the relative permittivity data obtained by the TDR device were assigned to the moisture content of the particular 1 cm segments determined by the gravimetric method.

### 4 Determination of Moisture Diffusivity on the Basis of Measured Moisture Profiles

For calculation of moisture diffusivity from measured moisture profiles, the transient Matano method [3] was chosen. The method consists in application of Boltzmann transformation, which can be used in sufficiently short time intervals, when the boundary condition on the dry end of the specimen is not yet effective. The advantage of the Boltzmann transformation lies in conversion of a solution of partial differential equation generally applied for description of moisture transport, to a solution of an ordinary differential equation (for details see [4]). If the moisture distribution \( u(x) \) in time \( t = t_0 \) is known, it is possible to find the moisture diffusivity coefficient \( \kappa(u,x) \) according to the relation

\[ \kappa(u,x) = \frac{1}{2t_0 \left( \frac{du}{dx} \right)} \int_{x_0}^{x} x \frac{du}{dx} dx. \] (8)

### 5 Experimental Results and Discussion

Fig. 5 presents the moisture profiles in Boltzmann form calculated by the three conversion functions described before. The results show that the moisture content determined by Malicki [10] and Topp [9] relations are in a relatively good agreement. The differences between these two functions vary in the range of 2 – 3 volumetric moisture per cent, what seems to be
sufficient regarding the general accuracy of the probe. However, moisture content determined using the empirical calibration by the gravimetric method is systematically higher in comparison with these two methods.

These differences can be interpreted as follows. As it was mentioned before, the relative permittivity of water bonded in a monomolecular layer is approximately 2.5, but for further layers it increases relatively fast. Therefore, moisture in the hygroscopic range which is bonded mainly by sorption forces to the material matrix can not be detected by the TDR method. This phenomenon is generally more remarkable for low moisture content measurements. However, for highly hygroscopic materials it can be significant in wider moisture ranges.

In Fig. 6, the sorption and desorption isotherms of the studied FGD gypsum are presented. Clearly, the studied material is highly hygroscopic. The desorption curve exhibits very high moisture content of about 17% by volume even for the relative humidity as low as 11%. Therefore, our guess concerning the significant effect of bound water on the TDR measured data in the particular case of FGD gypsum studied in this paper can be considered as correct, and only the data given by the empirical calibration can be employed for the calculation of moisture diffusivity.

The moisture dependent moisture diffusivity calculated from the moisture profile given by the empirical calibration by the gravimetric method is presented in Fig. 7. The course of the $\kappa(w)$ curve is within the expected range, taking into account the fast water transport in gypsum materials.

![Fig. 5 Moisture profiles of FGD gypsum determined by three different methods](image-url)
Fig. 6 Sorption and desorption isotherms of FGD gypsum

Fig. 7 Moisture diffusivity of FGD gypsum in dependence on moisture content
6 Conclusions

The experiment performed in this paper can be considered as a first step towards the application of TDR technique for monitoring moisture content in hygroscopic building materials. It was found that methods for calculation of moisture content from measured apparent relative permittivity used in soil science are not applicable for building materials in general, and verification of conversion functions by a reference method is necessary case by case. The problem of calibration of TDR data and development of new conversion functions will be the subject of our future work in the field of application of TDR methodology for measuring moisture content in building materials.

7 Acknowledgements

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References


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Advanced Monitoring of Wetness in Pharmaceutical Powder Processes Using In-Situ Dielectric Probe Measurements

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ABSTRACT. We applied spectroscopic analysis of microwave measurements of the complex dielectric constant of different mixtures of pharmaceutical materials using a coaxial dielectric probe. The dominating material in the investigated mixtures was microcrystalline cellulose. We conducted laboratory and in-situ measurements in a small-scale high-shear mixer in the frequency range of 1-19 GHz using a network analyzer. By using the suggested microwave technique we demonstrate the possibility to measure the initial wetness of the mixture in-situ. Density independent estimation having an accuracy of the order of 0.3-0.5 % wetness is demonstrated. The results envision the possibility to perform an adaptive control of the evolution of the mixing process by utilizing the microwave sensor information.

Keywords: in-situ measurements, dielectric probe, pharmaceutical process, dry mixing

1 Introduction

The manufacturing of pharmaceutical oral solid dosage forms (tablets) involves several unit operations. In this stages the material content is treated to change some of its physical properties in order to improve a certain material characteristics e.g. flow capability. Measurements of these changes are a key factor for the correct understanding of the pharmaceutical process. Performing such measurements in-situ and in real-time could further provide a possibility for the process monitoring and enable for an adaptive process control.

We present a study on the possibility to apply microwave (MW) techniques in the pharmaceutical industry. The advantage of the MW approach is in its non-invasive and non-destructive nature. The technique’s fast acquisition times are also a precondition for its successful implementation for real-time applications. We study in-situ characterization measurements of the process of high-shear mixing of powders and wet masses, a process also referred to as granulation [1,2]. This process is a widely used approach for enhancing the manufacturing properties of the pharmaceutical substances. Solid materials are here mixed with a binding liquid, e.g. water or a mixture of water and a solid binder, providing in this way a homogenisation of the substance distribution, accompanied by growth of the mixture particles. The liquid content, the temperature and the density of the mixture increase in the process. Since the role of the water in the mixing is crucial [1,2], the choice of using MWs for the process characterisation is well suited due to the well known strong interaction between the polar water molecule and the MW electromagnetic field.

We applied a spectroscopic analysis of MW measurements of the complex dielectric constant \( \varepsilon = \varepsilon' - j\varepsilon'' \) of a pharmaceutical mixture. We conducted laboratory and in-situ measurements using a coaxial dielectric probe and a network analyzer. For calibration purposes, laboratory measurements of a preconditioned material were carried out, while the in-situ measurements were performed in a small-scale high-shear mixer.

* other solvents e.g. ethanol could also be used but water is the most common
2 Measurement Setup

2.1 Microwave Measurements

For the purpose of measuring the dielectric properties of the materials we employed an Agilent PNA series network analyser equipped with the Agilent 85070D dielectric probe kit and the 85070E measurement software [3]. The accuracy of the measured dielectric constant according to the manufacturer is 5 % for the permittivity ($\varepsilon'$) and 0.05 % for the loss tangent ($\varepsilon''/\varepsilon'$). For the purpose of the in-situ measurements an automation interface was developed using LabView. The conducted laboratory and in-situ measurements of the dielectric constant were in the frequency range of 1-19 GHz with a frequency resolution of 100 MHz. For the purpose of minimising the measurement noise the analyser IF bandwidth was set to 50 Hz.

![Diagram of a granulation vessel indicating also the location of the dielectric probe](image)

Fig. 1 A drawing of a granulation vessel indicating also the location of the dielectric probe [3].

2.2 In-Situ Measurements

The in-situ measurements of a granulation process using the above mentioned microwave equipment were carried on in a high-shear mixer apparatus (Mixer-Granulator P 1-6, DIOSNA Dierks & Soehne GmbH). The dielectric probe was interfaced to the four litre process vessel through installing a port with diameter of 19 mm at the vessel side and at a height just above the blades of the impeller. Fig. 1 displays a drawing of a granulation vessel with the location of the dielectric probe. The sampling rates used in the conducted in-situ measurements were 1 and 5 sec. All measurements were taken at an ambient temperature of 22°C.

2.3 Other Measurements

All material that we tested were measured using a Loss On Drying (HR73, Halogen Moisture Analyser, Mettler-Toledo AG) equipment having a repeatability of 0.1 % according to the instrument specifications [4].
3 Materials and Experiment Design

Normally, the granulation process consists of an initial mixing of an active(s) and a filler materials – a stage referred to as dry mixing, after which the liquid is added at a certain rate – liquid addition stage. When all of the binding liquid is added the process could either be stopped or continue in a wet massing stage, where further homogenisation could take place accompanied by changes in the particle size distribution. In this work we focus on measurements taken during the dry mixing stage of the mixing process, which in all our runs lasted for 2 minutes.

The main pharmaceutical material used in the conducted granulation experiments was microcrystalline cellulose (MCC), a commonly used filler material in pharmaceutical tablet formulations. In order to enable the formation of the granules in the mixing process a binding material was initially mixed with the MCC and then pure water was added.

In order to develop a calibration method relating measurements of the complex dielectric constant of our pharmaceutical material to its wetness we have preconditioned MCC in order to obtain different levels of wetness. We used climate boxes with different levels of relative humidity inside providing us with the following levels of MCC wetness: 2.1 %, 3.6 %, 4.5 %, 5.2 %, 5.8 %, and 8.1 %. The conditioned samples were then gently pressed in order to achieve a compact state and measured with the dielectric probe in the range of 1-19 GHz.

Then for the purpose of the in-situ measurements, a second set of preconditioned (in a climate chambers) MCC was generated. We performed four granulations and Table 3.1 summarises some of the parameters of those tests denoted as Exp.1 to Exp.4. The rotation speed of the impeller was constant in all of the tests (Speed A). The experiments were designed to have different initial MCC wetness ranging from 1.5 %, through 4.3 %, to 8.2 % and as it can be observed in Table 3.1 the amount of MCC used dominated the mixture (93 %) and was therefore the main responsible for the system behaviour. We should note that normally in the granulation mixture some active pharmaceutical substance(s) will also be added, but since the dominant substance often is the filler, in these tests we have decided to study the process when only using a filler (MCC) and a binder.

In addition to the above tests we have conducted seven supplementary granulations where MW measurements were taken in-situ. Different impeller speeds were applied while keeping a constant initial mixture wetness. A slight difference with the previously presented experiments was the presence of a certain percent of a non hygroscopic active substance (<0.5 % wetness) in the mixture. The ratio used in the mixture was 80 % MCC, 15 % active substance and 5 % binder, providing an average mixture initial wetness of 3.6 %. Two different impeller speeds were tested (denoted as A and B) while keeping all the rest of the process parameters not changed.

| Table 3.1 Initial condition in the conducted granulation experiments Exp.1 to Exp.4. |
|---|---|---|---|---|
| Mass filler (g) | Mass binder (g) | MCC initial wetness (%) | Binder initial wetness (%) | Mixture initial wetness (%) |
| Exp. 1 | 350 | 22 | 1.5 | 5.4 | 1.7 |
| Exp. 2 | 350 | 22 | 4.3 | 5.4 | 4.4 |
| Exp. 3 | 350 | 22 | 8.2 | 5.4 | 8.0 |
| Exp. 4 | 350 | 22 | 8.2 | 5.4 | 8.0 |
4 Measurements

4.1 Calibration Measurements

In Fig. 2 we present the measured complex dielectric constant of the preconditioned MCC for the frequency range of 2 to 14 GHz. The generated moisture levels were selected to cover the values which the initial material moisture might have (under normal ambient conditions the expected MCC wetness would be around 4%).

Fig. 2 Measured complex dielectric constant of MCC with different levels of wetness.

4.2 Moisture Measurements

During the process of mixing the complex dielectric constant of the material apart from the moisture content will be affected by a number of additional factors some of which are:

- varying density of the material present to the sensor. This is an inherent property of the granulation process, where for example using different impeller speeds will generate different average material density inside the vessel. With the granulation process evolution the material itself will also experience densification which will affect the measurement. Since this study is focusing on the dry mixing stage of the process, we will not be affected by the material densification but rather only experience the effect of the varying density inside the vessel due to the impeller speed effect.

- varying temperature of the material due to the friction forces during the mixing. The temperature increase in the liquid addition and the wet massing stage could be tens of degrees. The temperature change during the dry mixing phase is on the other hand negligible (in-situ temperature measurements have shown this change to be within one degree) and therefore in this work we assume that the temperature inside the process vessel was constant, being the ambient temperature (≈22°C)
Since a particular problem in our measurements is the difficulty to reproduce the density of the material inside the vessel, a function able to present a density independent quantity relating the material complex dielectric constant to the moisture content was sought. Assuming that the effects of the temperature and the material densification could be disregarded we have tested a number of density independent relations summarised and presented in [5]. We have identified the ratio:

\[ \xi(f) = \frac{\varepsilon''(f)}{\varepsilon'(f) - 1} \]  

(1)

to produce the best results and therefore used it in this work. We first calculated \( \xi(f) \) for the conditioned at different moisture levels samples and for each frequency. We then fit a linear model:

\[ M = \xi(f) \cdot a(f) + b(f) \]  

(2)

to relate the measured \( \xi(f) \) to the material wetness \( M \), where the model coefficients \( a(f) \) and \( b(f) \) were estimated from regression to the calibration measurements. In the results section we use the ratio (1) in combination with (2) having the estimated as described above regression coefficients.

5 Results

5.1 Density Independent Moisture Measurement

In Fig. 3 we present as an example the measured permittivity and loss factor for Exp.1, one minute after the beginning of the granulation process. In Fig. 4 we present the time evolution of the estimated wetness for all the experiments using the model in Eq.2 for 9 GHz. The sampling rate used was 5 seconds which was considered so in order to allow for the measurements with the frequency resolution and the instrument IF described above to take place. One can notice the increase of the wetness in the last sample coinciding with the beginning of the process of the water addition. We would like to point out the temporal stability and the lack of drifts in the estimated wetness.

In Table 5.1 we present the statistical results of the estimation error in Exp.1 to Exp.4. Presented is the absolute and the relative errors. The errors are based of the estimates from measurement at each frequency and time epoch in the particular experiment.

In Fig. 5 we present in more detail the estimation error as a function of the frequency used. As it can be observed it is difficult to find an optimum frequency producing equal accuracy in all wetness levels. Note the good repeatability of the measurements from Exp.3 and Exp.4. which had the same initial wetness, indicating a good instrument and method stability. A possible reason for the poorer estimation at the high levels of wetness could be the lack of calibration points in the wetness levels higher than 8.2 %. One reason that we limited us to 8.2 % was that this was the maximum MCC wetness that we could achieve using climate boxes. In the future additional calibration points having larger wetness content will also be considered.
Fig. 3 An example of measured in-situ complex dielectric constant (top – permittivity, bottom – loss factor) as a function of frequency. Displayed are the values measured 1 minute after the start of the mixing process (Exp.1).

Fig. 4 Estimated wetness for Exp.1 to Exp.4 using the measurements at 9 GHz.

Table 5.1 Estimation error (absolute and relative value) for Exp.1 to Exp.4.

<table>
<thead>
<tr>
<th></th>
<th>Exp.1</th>
<th>Exp.2</th>
<th>Exp.3</th>
<th>Exp.4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial wetness</td>
<td>1.7</td>
<td>4.4</td>
<td>8.0</td>
<td>8.0</td>
</tr>
<tr>
<td>Estimation error</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>- % wetness</td>
<td>0.6</td>
<td>0.6</td>
<td>1.3</td>
<td>1.3</td>
</tr>
<tr>
<td>- relative value</td>
<td>35</td>
<td>13</td>
<td>16</td>
<td>16</td>
</tr>
</tbody>
</table>
Fig. 5 (top) Estimated average wetness (Exp.1 to Exp.4) as a function of the frequency used and based on all 24 observations taken during the dry mixing of each experiment. (bottom) The estimation absolute error in percent wetness.

Fig. 6 (top) Estimated wetness from the seven verification tests as a function of the frequency used. All time epoch were included in the estimation. (bottom) The estimation absolute error in percent wetness. The initial mixture wetness was 3.6 %. Lines marked with dots and circles indicate experiments with impeller speeds A and B, respectively.

5.2 Verification Using Additional In-Situ Measurements

Fig. 6 presents the results of the estimated initial wetness in all of the additional test runs, where the initial mixture wetness was 3.6 %. The results indicate a good repeatability between
the processes, where the present systematic error indicates errors in the calibration rather than measurement errors. The robustness of the method is also indicated by the independence of the result on the impeller speed used, where the estimated wetness when using impeller speed A and B show similar accuracy. Table 5.2 presents some statistics of the estimation error for all test runs. In the error calculation estimates for all available times and experiments were used, but combining differently selected frequency ranges. As it can be observed the estimation error is somewhat better than in Exp.1 to Exp.4. One reason for this could be that in these verification runs we used a lower spectral resolution of 1 GHz, allowing us to sample every 1 sec providing in this way more data samples and thus minimising the effects of errors with white noise behaviour (not correlated in time).

Table 5.2 Estimation errors (absolute and relative) for the verification tests when using different frequency sets and for the two different impeller speeds.

<table>
<thead>
<tr>
<th>Frequency range used in the estimation</th>
<th>1 – 19 GHz</th>
<th>4 – 15 GHz</th>
<th>11 – 15 GHz</th>
</tr>
</thead>
<tbody>
<tr>
<td>Estimation Error</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>- % wetness (Speed A/ Speed B)</td>
<td>0.5 (0.5 / 0.5)</td>
<td>0.3 (0.3 / 0.2)</td>
<td>0.2 (0.2 / 0.1)</td>
</tr>
<tr>
<td>- relative value [%]</td>
<td>13</td>
<td>8</td>
<td>5</td>
</tr>
</tbody>
</table>

6 Conclusions

The importance of the initial material wetness for the outcome of the high-shear mixing process is critical, since it will affect the final outcome of the granulation process. The initial wetness is part of the complex granulation system where in addition to it, parameters such as the liquid addition rate and method, the liquid amount, the impeller speed and its shear angle affect the process outcome [2]. The achieved after the mixing final physical properties such as density and porosity, final wetness and particle size distribution are of crucial importance for the tableting properties of the mixture [2]. Therefore the demonstrated here method to measure the initial mixture wetness in-situ and with an accuracy at the sub-percent level, envisions the possibility to apply an adaptive process control of the mixing process through for example adjusting the liquid addition rate or amount, the mixer impeller speed or the total process time.

References

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4. www.mt.com/mt/product_detail/product.jsp?m=t&key=I2NDg4NjM1Nz

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Phone: +46 (0)31-706 55 85; Fax: +46 (0)31-776 37 44; E-mail: Lubomir.Gradinarsky@astrazeneca.com
Non-Destructive Microstrip Resonator Technique for the Measurement of Moisture / permittivity in Crude Oil

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ABSTRACT: An accurate, instantaneous and non-destructive Microstrip Resonator Technique has been set up for the measurement of permittivity and moisture of materials in solid, liquid and powder form. This paper reports the permittivity measurements in crude oil and water emulsions made in the laboratory. The resonant frequency of the microstrip resonator is measured in the presence of crude oil and the real part of the permittivity of the material under test is determined with the help of spectral domain analysis of multi-layer microstrips, which are embedded in dielectric cover. The relatively low values of permittivity of high oil percentage emulsions may be attributed to the in-pouch testing procedure.

Keywords: crude oil, emulsion, permittivity, watercut

1 Introduction

Many multiphase flow and watercut meters used for fiscal and quality purposes are based on established electrical impedance technique. Knowledge of the relative permittivity of flow components is fundamental to such technologies. Calibration of multiphase flow meters requires permittivity data of crude oil and its components [1]. The other reason for importance of the permittivity measurements lies in remote sensing applications. Microwave radiometry of regions with oil spills needs oil and emulsion permittivity data to establish the thickness of oil films from the emissivity data [2].

In the petroleum refinery industry, there is increasing demand for on-line determination of product quality. Dielectric spectroscopy has previously been shown to be a relevant method for quality characterization of petrochemical products. Several parameters describing the quality of the oil can be found from the measured permittivity spectrum. Oil has low permittivity and loss, long relaxation times and a broad frequency dispersion region. Hence, the permittivity spectrum has to be measured with high sensitivity over a broad frequency range (typically 1 kHz to 10 GHz), which requires sensitive measurement cells and adequate models for calculating the permittivity.

At low frequencies (1 kHz to 10 MHz), the impedance measurements of coaxial cells give high-precision measurement of permittivity [1]. At radio and microwave frequencies, transmission and reflection measurements of coaxial cells using network analyzers are a suitable method for fast and broad-band permittivity determination of low-loss liquids [3]. The accuracy in the measurements is lower than that which is obtained with resonator techniques, but the resonator methods are time-consuming when applied in a broad range of frequencies [4]. In comparison, a coaxial cell can typically be used over 1-2 frequency decades.

When measuring the parameters of petroleum products, a representative sample of the flow must be taken. Considering the industrial applications of dielectric spectroscopy, an
obvious drawback is that the cell is intrusive. Waveguide resonant cavity techniques have been widely used to perform non-destructive measurement of the moisture as well as complex permittivity of materials. Though considered accurate, the waveguide cavity technique is associated with difficulties like loading and unloading of samples. Out of the possible measurement principles, electromagnetic interaction is promising for cases where the fluid components have distinct dielectric properties.

ABB has published a report on a multiphase meter based on a special slow-wave resonator operating in the radio frequency range [4]. The dielectric properties of the fluid (permittivity, conductivity) determine the dispersion and, hence, the resonance spectrum of the measurement device. Microstrip resonator techniques, based on statistical or approximate closed form dependence of \( \varepsilon' \) and \( \varepsilon'' \), which are being used in the industry, do give sufficient accuracy for the crude oil industry.

To go a step ahead, we have implemented a novel technique for the measurement of complex permittivity for crude oil and water emulsions. The technique has used the microstrip resonator as a non-intrusive sensor, which can be used along with the flow meter itself without the need for sampling of the oil.

2 Experimental Set-up

The set up for measuring the rise in effective permittivity and the drop in the resonant frequency of the ring resonator with increase in the percentage of moisture in the crude oil sample were specifically designed for petroleum-drilling applications, keeping in mind that the occurrence of crude oil is always accompanied by water or moisture. It is this concurrent occurrence that could pose difficult challenges. Hence, by providing a non-destructive technique to measure the percentage of moisture in a sample of crude oil, these difficulties could be alleviated. In order to measure the percentage of moisture in the crude oil, firstly, the resonant frequency of the microstrip resonator is measured in the presence of crude oil while the real part of the permittivity of the material under test is determined with the help of spectral domain analysis of multi-layer microstrips embedded in dielectric cover.

The tests were performed using a 5 GHz ring resonator on substrate of dielectric of 4.7. We intended to quantify a relationship between the change in parameters of the resonator with change in its environment. The change in the composition of oil and water is effectively changed in the environment of the resonator circuit. We used this fact in our testing procedure.

3 Theoretical Background and Post-processing of Data

Microwave resonator technique is a powerful tool for the measurement of moisture / dielectric constant of the material under test since, with the availability of modern network analyzers, frequency measurement is not subject to measurement device errors like drift and repeatability. The present paper reports an accurate and fast watercut measurement, using a microstrip resonator. The material under test is kept as an overlay on the microstrip probe. The resonant frequency and the quality factor of microstrip
resonator are measured. The change in the effective permittivity of the microstrip probe is given by

\[ \frac{f_o^2}{f_s^2} = \frac{\varepsilon_{effs}}{\varepsilon_{effo}} \]

(1)

where suffixes “o” and “s” indicate resonant frequency (f) and effective permittivity (\(\varepsilon_{eff}\)) without and with the sample as an overlay on the resonator cavity. For low loss materials,

\[ \Delta f_o = f(\varepsilon_{rs'}) \]

(2)

\[ \Delta Q = f(\varepsilon_{rs'}, \varepsilon_{rs''}) \]

(3)

where \(\varepsilon_{rs}\) is the relative permittivity of the overlay sample and \(\varepsilon_{rs'}, \varepsilon_{rs''}\) indicate real and imaginary parts of the complex permittivity. \(\varepsilon_{rs'}\) of the material under test is determined with the help of spectral domain analysis[3,4].

Specially-created and dedicated software EPSILONCALCTM has been used to determine the real of the permittivity of the oil-water emulsions in the plastic pouch. EPSILONCALCTM The software for material permittivity measurement is applicable to the materials with infinite as well as finite depth. Thickness and width of the samples need to be supplied as parameters to the software.

### 4 Results and Discussion

Tables 1-4 give present and other published data on permittivity of crude oil, which seems to be around 2. The standard deviation is 5% in most cases. Figure 1 gives the experimental data on the variation of resonant frequency with water in the emulsion kept on ring resonators as overlay. The emulsions were kept in the zip-bag pouches for avoiding oil on the resonators. Figures 3-4 give simulated data using the software EpsilonCalcTM, which takes only a fraction of a second for single frequency point. The software has been validated using other commercial suppliers and experiments. The resonator data as well as material height needs to be supplied to the interactive software. Figures 4-6 give coaxial probe permittivity data for crude oil. However, it was not possible to compare higher moisture data due to unavailability. Table 4 gives a comparative review of the various publications.

<table>
<thead>
<tr>
<th>Fluid</th>
<th>Average permittivity</th>
<th>Standard deviation</th>
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<tbody>
<tr>
<td>p-xylene</td>
<td>2.327</td>
<td>0.005</td>
</tr>
<tr>
<td>Crude oil</td>
<td>2.034</td>
<td>0.113</td>
</tr>
</tbody>
</table>

Table 1 Average value and standard deviation for the high-frequency permittivity, \(\varepsilon_s\), of the 16 repeated measurements [50]

<table>
<thead>
<tr>
<th>Crude oil</th>
<th>(\varepsilon_s)</th>
<th>(\varepsilon_{\infty})</th>
<th>(\tau) (ns)</th>
<th>(\alpha)</th>
<th>(\sigma) (ns*m(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oil 1</td>
<td>2.295</td>
<td>2.169</td>
<td>3.35</td>
<td>0.54</td>
<td>42.9</td>
</tr>
<tr>
<td>Oil 2</td>
<td>2.324</td>
<td>2.198</td>
<td>11.3</td>
<td>0.52</td>
<td>2.0</td>
</tr>
<tr>
<td>Oil 3</td>
<td>2.248</td>
<td>2.151</td>
<td>2.40</td>
<td>0.50</td>
<td>11.4</td>
</tr>
</tbody>
</table>

Table 2 Estimated Cole–Cole parameters of three North Sea crude oils [3]
Table 3  Permittivity calculations from the present study using EPSILANCALC
Open Resonant Frequency 5.3GHz, (Material FR4-$\varepsilon_r=4.7$)

<table>
<thead>
<tr>
<th>Oil %</th>
<th>Refractive index equation $\varepsilon'_{oil}$</th>
<th>Present tech $\varepsilon'_{oil}$</th>
<th>Resonant Frequency (GHz)</th>
<th>Effective permittivity $\varepsilon_{eff}$</th>
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<tr>
<td>100</td>
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<td>4.915</td>
<td>4.159212</td>
</tr>
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<td>20</td>
<td>58.97</td>
<td>39.11376</td>
<td>3.709</td>
<td>7.140352</td>
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</tbody>
</table>

![Resonant frequency Vs Oil % in water oil suspension](image1)

Fig. 1 Resonant frequency variation with water percentage in crude oil.

![Effective permittivity](image2)

Fig. 2 EpsilonCalc™ results for variation of effective permittivity with dielectric cover thickness and w/h ratio of the microstrip $\varepsilon_{substrate}$ and $\varepsilon_{cover}=2.53$, h=1.27 mm
Fig. 3 EpsilonCalc™ results for variation of effective permittivity of a microstrip with dielectric cover thickness and cover permittivity $\varepsilon_{\text{substrate}} = 9.99$, $h = 0.635$ mm

Fig. 4 Measured permittivity (O) of n-heptane as a function of pressure and temperature at 20, 40 and 70°C (from top to bottom respectively) using an open-ended coaxial probe [5]

Fig. 5 Measured permittivity (O) of n-decane as a function of pressure and temperature at 20, 45 and 70°C (from top to bottom respectively) using an open-ended coaxial probe [5]
Table 4  High frequency permittivity values from the literature

<table>
<thead>
<tr>
<th>Liquid</th>
<th>Simple calibration</th>
<th>Bi-linear calibration</th>
<th>Literature values</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>n-pentane</td>
<td>1.80</td>
<td>1.84</td>
<td>1.844</td>
<td>6</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>1.841</td>
<td>7</td>
</tr>
<tr>
<td>n-decane</td>
<td>2.02</td>
<td>1.99</td>
<td>1.987</td>
<td>8</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>1.991</td>
<td>6</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>1.989</td>
<td>7</td>
</tr>
<tr>
<td>p-xylene</td>
<td>2.45</td>
<td>2.27</td>
<td>2.270</td>
<td>6</td>
</tr>
</tbody>
</table>

Fig. 6 Measured permittivity (O) of North Sea crude oil as a function of frequency using transmission type coaxial cell[9]

5 Conclusion

The results from the present work compared with the published data indicates that the novel numerical analysis technique can be used for crude oil watercut measurements. The technique is very fast in the numerical postprocessing and can give on-line measurement device with the measurement time in milliseconds. The possible applications would be well-logging, refinery flow meters, quality control and mixed fuel sensor. The lower values of permittivity are attributed to the in–pouch testing procedure. The measurements will be repeated for the direct contact and non-contact measurements to avoid contamination of the sensor surface.

Acknowledgements

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A Novel Application of Planar Electromagnetic Sensing Technique – Quality Inspection of Saxophone Reeds

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ABSTRACT. This paper has described some initial work in developing a planar electromagnetic sensor for use in non-destructive evaluation (NDE) of saxophone reeds. Both theoretical and experimental approaches were undertaken. The results obtained serve as a starting point for development of a commercial sensor. This sensor has many different possible applications including quality inspection for dairy products and metal products. Conclusions are drawn from the results such as the differences observed between ‘good’ and ‘bad’ reeds. Suggestions for further research are also stated to indicate possible directions that may be taken using this research as a starting point.

Keywords: Planar mesh sensor, Saxophone reeds, Non-destructive Evaluation, Analytical model.

1 Introduction

The researches using planar magnetic sensor has demonstrated the detection of defect in the printed circuit board and the estimation of near-surface material properties of conducting and magnetic materials. The outcome of the research is very encouraging and the successful results were published in international journals [1 - 6]. The planar electromagnetic sensing technique employing radio-frequency (RF) signal is based on the interaction between the measurement signal and the material under investigation. The configuration of electromagnetic sensors used for the inspection of the quality of the saxophone reeds is of planar type. The sensor consists of two coils: one coil known as exciting coil carrying radio frequency signal generates electromagnetic field. The generated electromagnetic field interacts with the nearby materials being measured. The resultant electromagnetic field is measured by the other coil, known as pick-up coil which is placed above the exciting coil. The ratio of the voltage of the pick-up coil to the current of the exciting coil is defined as the transfer impedance. The transfer impedance is a function of many parameters such as permittivity, conductivity, permeability of the materials being measured, the lift-off, operating frequency, thickness etc. There is no direct method available to determine them separately but it is possible to evaluate the material properties with the help of some computational method. It is possible to monitor the quality of the system under test by the measurement of transfer impedance of the sensor. The advantage of this over many other testing methods is that the test material is not physically touched or altered. The method is therefore a form of Non-Destructive Evaluation (NDE) and is useful in situations where it is impractical or infeasible to destroy or damage a test sample using chemical or physical testing means. This may be because a test sample is very expensive or because every item produced must undergo the test and therefore every item must survive the test unaltered.

Another advantage of NDE is that it may be applied as a safety check to items that are already in use. As NDE does not further damage a test material it may be used to evaluate parameters such as wear and fatigue and allows pre-emptive action, such as repair or replacement, to be taken on a test subject that is about to fail.
2 Applications of the Sensor

2.1 Saxophone Reeds

A saxophone reed as shown in Fig. 1 is a small piece of bamboo that is attached to the mouthpiece of a saxophone. When the player blows into the saxophone the reed vibrates creating sound. The reed is therefore, in part, responsible for the tone and ease of use of a saxophone. There is nothing more frustrating for a saxophone player than playing on a bad reed. Reeds wear out after a few weeks of playing and must be replaced. The problem with reeds is that currently the quality is very variable: in a box of ten, three or four reeds are found to be ‘bad’ when played and are discarded. As the player pays $10 per reed or more any improvements in quality control would be very worthwhile.

The parts of the reed that affect the quality of the reed the most are the ‘vamp’ and the ‘tip’ (See Figure 1).

Fig. 1 Tenor and Alto Saxophone Reeds, different sections of the reeds, the sensor and a saxophone

2.2 Quantitative Measurement

The sensor is composed of a copper ‘coil’ that is fabricated on a polyimide film. Fabrication in this manner allows the coil to be made very precisely, as the polyimide film keeps the thin, frail coil from deforming or breaking.

The technique used to apply the coil as a sensor involves passing a known alternating electrical current through a primary exciting coil which is of mesh type. This current was a frequency sweep generated by connecting the primary coil to the transmission port of a network analyser using low loss, high frequency cables as shown in Fig. 2. The range of the frequency sweep was defined as 300 kHz – 1.3 GHz. The current creates a magnetic field, the properties of which (magnitude and phase) change depending on the surrounding material’s (in our case the saxophone reed’s) composition and structure.
A secondary coil known as sensing coil of similar or identical construction to the primary coil (See Figure 1) was placed on top of the primary coil while current is passed through the primary. The magnetic field created by the current in the primary, generate an electromagnetic field which induces an electromotive force (emf) in the secondary, sensing coil. This emf was measured by connecting the secondary coil to the reception port of the network analyser. The magnitude and phase of this emf is compared to the exciting current in the primary coil. From this the network analyser calculated the transfer impedance of the sensor over the spectrum specified. This transfer impedance (both magnitude and phase) was then recorded used to predict the properties of the surrounding materials (i.e. the saxophone reed).

The sensor was placed above the reed so that it was directly above the tip and most of the vamp. The size of the gap between the sensor and the reed, called the “lift-off” (See Figure 2) was closely controlled. Lift-off affects the results dramatically, so it was set to a minimum (virtually zero) by adjusting the sensor height.

2.3 Playing The Reeds – Qualitative Measurement

One of the authors is a very keen saxophone player, whose musical achievements include teaching the saxophone part time, playing in various musical shows, leading a Jazz Quintet, playing in the Manawatu Jazz Club Big Band and also in the New Zealand National Youth Jazz Band. This experience created the initial interest in developing the sensor for use on saxophone reeds.

The experience also gives the author a good idea of what a ‘good’ and ‘bad’ reed sounds and ‘feels’ like when played. This allowed the reeds to be tested qualitatively by playing them for approximately ten minutes to gain an idea of how the reed performed in specific areas such as tone quality. This assessment was done before quantitatively testing the reeds so as not to make the qualitative test of playing a reed fit the quantitative results obtained from the sensor i.e. avoiding bias.

Reeds were rated on the following parameters: ease of attack, ease of sustain and tone quality in the low, mid and high ranges of the instrument as well as a score for volume. These parameters were then averaged to get the overall score.
3 Theoretical Model

The analytical model of the sensor of the Fig. 1 has been carried out assuming the sensor is placed on the reed under test as shown in Fig. 2 and the transfer impedance of the sensor as a function of the permittivity and other parameters such as permeability, conductivity etc. of the reed has been calculated. The detailed description of the development of analytical model has been described in [7, 8]. Figs. 3a and b show the variation of the resistive part and the reactive part of the transfer impedance as a function of the relative permittivity of the product at an operating frequency of 500 MHz. It is seen that the transfer impedance changes appreciably with the relative permittivity and the magnitude of the transfer impedance can very well be used to predict the permittivity by some computational technique. Figs. 4a and b show the variation of the resistive part and the reactive part of the transfer impedance as a function of the relative permittivity of the product at an operating frequency of 1 GHz. It is seen that the rate of change of both the real and imaginary part of the transfer impedance is much higher compared to that of frequency at 500 MHz.

Fig. 3a Variation of resistive part of transfer impedance with permittivity

Fig. 3b Variation of reactive part of transfer impedance with permittivity
So the selection of frequency is very important to deal with dielectric material like the reed. Usually an operating frequency of higher than 500 MHz is used for this purpose. It is seen from Figs. 3 and 4, that the reed behaves as a resonant circuit and gives a peak at a resonant frequency. In the analytical model the permittivity is assumed as a constant parameter but in practice the permittivity varies with the operating frequency. The experimental results thus show many peaks when the magnitudes are plotted as a function of frequency.

4 Experimental Results

Both qualitative (by playing reeds and scoring them out of ten) and quantitative (by measuring the response of the sensor) results were obtained. These results are shown in Table 1 and Figs. 5 and 6.

It should be noted that the sample of reeds tested have exactly the same response as each other except that reed 1 differs from the other reeds at one point in the magnitude plot, near 150 MHz and, more importantly, differences at the 579 MHz in the phase plot may be observed (Figs. 5, 6). This frequency corresponds to an impedance magnitude peak (local maximum) for all reeds. At this point the reeds give different responses and may be separated into two distinct groups based on their phase response.
Table 1 Saxophone reed quality

<table>
<thead>
<tr>
<th>Reed #</th>
<th>Alto/ Tenor</th>
<th>Played Much?</th>
<th>Strength</th>
<th>Overall Quality</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Tenor</td>
<td>Old</td>
<td>2.5</td>
<td>6</td>
</tr>
<tr>
<td>2</td>
<td>Tenor</td>
<td>Old</td>
<td>2.5</td>
<td>6</td>
</tr>
<tr>
<td>3</td>
<td>Tenor</td>
<td>Mid</td>
<td>2.5</td>
<td>9</td>
</tr>
<tr>
<td>4</td>
<td>Tenor</td>
<td>Mid</td>
<td>2.5</td>
<td>4</td>
</tr>
<tr>
<td>5</td>
<td>Tenor</td>
<td>New</td>
<td>2.5</td>
<td>9</td>
</tr>
<tr>
<td>11</td>
<td>Tenor</td>
<td>New</td>
<td>2.5</td>
<td>9</td>
</tr>
<tr>
<td>12</td>
<td>Tenor</td>
<td>New</td>
<td>2.5</td>
<td>8</td>
</tr>
<tr>
<td>13</td>
<td>Tenor</td>
<td>New</td>
<td>2.5</td>
<td>8</td>
</tr>
</tbody>
</table>

Fig. 5 Magnitude Response of Sensor

Bearing this grouping in mind, all the members of the group with the larger phase angle at 579 MHz have been bolded in the qualitative test results (Table 1). All these bolded entries scored lower and may be said to be the ‘bad reeds’, while the members of the other group all obtained very high overall scores.
Due to the small sample size analysed to this level, the results are promising, but not conclusive.

Fig. 6 Phase Response of Sensor

5 Conclusions

Non-destructive evaluation (NDE) is a technique that may be applied to many different areas. This paper looks at the specific case of saxophone reeds and begins to develop a sensor for this purpose.

Initial results for quality inspection of saxophone reeds are promising. There appears to be a measurable difference given by the sensor between ‘good’ and ‘bad reeds. A larger sample size must be taken to confirm this hypothesis, which is planned to be done now.

Further work must be completed to develop a theoretical model that adequately describes the sensor and reed. Initial work has involved the development of a 2-dimensional analytical model. Using the finite element method a 3-dimensional model is being planned to be developed taking into account all desirable factors along with the proper shape of the reed.

Further research would be to develop this sensor to a commercially viable product may be undertaken, using this paper as a starting point. The author has also undertaken considerable research into applying the sensor to other situations such as metallic and dielectric substances, with both theoretical and experimental results being available.
References


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1 New Sensor for High Moist Leaves in Green Tea Production  
S. Okamura, N. Tsukamoto; Shizuoka, Japan

2 Intangible but not Intractable: The Prediction of Food ‘Quality’ Variables Using Dielectric Spectroscopy  
M. Kent, R. Knöchel, F. Daschner, O. Schimmer; Kiel, Germany; J. Oehlenschläger, S. Mierke-Klemeyer, M. Kroeger; Hamburg, Germany; U.-K. Barr, P. Floberg; Göteborg, Sweden; M. Tejada, A. Huidobro; Madrid; Spain; L. Nunes, A. Martins, I. Batista, C. Cardoso; Lisbon; Portugal

3 Microwave Sensing for Food Structure Evaluation  
S. Clerjon, J.-L. Damez; Genes Champanelle, France

4 Effective Microwave Dielectric Properties of Food Materials Consisting of Large Particulates  
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5 RF Impedance Method for Nondestructive Moisture Content Determination in In-Shell Peanuts  
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K. Joshi; Pune, India

8 Frequency and Temperature Dependence of the Permittivity of Fresh Fruits and Vegetables  
S. O. Nelson; Athens, Georgia, USA

9 A Theoretical Relationship Between the Fractal Dimension and Moisture Content in Grains  
A. Albadri; Guildford, UK
New Sensor for High Moist Leaves in Green Tea Production

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ABSTRACT. This paper describes on a new sensor of practical use for producing green tea. It can measure moisture content as high as 160 to 300% on dry basis by use of microwave at 3GHz. The wave traveling down along a microstripline detects the information on the moisture contents of the green tea leaves overlaid on the microstripline. Moisture content of the leaves is calculated from the ratio of two signals on the attenuation and the phase shift of the waves traveling the microstripline. The error of the measurement was about 3 % of the readings.

Keywords: microstripline, attenuation, phase, ratio

1 Introduction

Green tea sold on the market has the moisture content of about 4 % on dry basis (d.b.). It is made from fresh tea leaves having the moisture content of 350-400 % d.b.. In a factory of green tea, several machines are used during the drying process of the tea leaves. Each machine has different role to make the leaves nice green tea with good flavor, mild taste, slender shape, and fresh green color. The machines are able to exercise their own maximum ability under proper conditions of moisture content of the leaves. Hence, the moisture measurement is very important to produce good green tea with high quality.

Generally used moisture measurement sensors are using electric resistance or capacitance of material under test operating at DC or radio frequency. These sensors are difficult to measure true moisture content of tea leaves because the current of DC or radio frequency is affected strongly by the surface moisture of material but affected slightly by the inside moisture. However, microwave can measure the moisture contained in the inside of material because microwave propagates through material and gathers information of the inside moisture. In factories of green tea two types of moisture sensors by microwave have been used. One is the microwave free space technique using a pair of horn antennas, which was developed about ten years ago [1]. It can measure the moisture contents of 30-190 % d.b. of the leaves. The sensor had a better ability for measuring the whole moisture content of leaves contained in both the surface and the inner part of the leaves and it has been used many factories in Japan. Another is the microstripline type sensor which was developed recently. It has been used already in many factories because of the smaller size than that of the horn type and the easiness in attaching the microstripline type sensor to a drying machine. The sensor can measure the moisture contents of 30 to 90% in a good accuracy.

The two types of sensor have contributed to high quality production of green tea and much labor-saving in the production process. However, more expansion of measurement range of the sensor is wanted in order to produce better green tea and save energy consumption on the process. This paper describes about a new sensor measuring high moisture content up to 300 % d.b..

Fresh tea leaves carried into a factory or steamed tea leaves in the first process of the green tea production are very wet and have high moisture content. Such leaves cannot be measured
even by microwave types described above. The difficulties in the measurement come from
next three matters. They are all caused by the too much water attaching over the tea leaves.

1. The first of the difficulties comes from irregularity of the length touching to the
microstripline and of the thickness of the sample leaves piled on the line.
2. The second is the density change of leaves accompanied by moisture content.
3. The third is that real moisture content is masked by the surface water, because
the leaves have much water on the surface as well as in the inner part of them.

The new sensor has overcome the three difficulties using the two signals of amplitude and
phase of microwaves, and using a new arrangement of the sensor board.

2 Principle

The moisture content of material M, on dry basis, is usually defined as:

\[
M = \frac{W_w}{W_d} \times 100\%
\]  

(1)

where \( W_w \) and \( W_d \) are the masses of water and dry material, respectively. The method
described in this paper uses the microwave transmission technique of a microstripline to
measure high moisture content. The schematic diagram of the measurement is shown in the
Figure 1. The leaves with high moisture content are overlaid on the microstripline, and a
microwave is transmitted along the line. Changes of the attenuation and the phase shift of the
microwave after through the line are mainly affected by the moisture contained in the leaves.

![Schematic diagram of the measurement](image)

Fig. 1 The moisture sensor using a microstripline

When a microwave is transmitted along the line overlaid with the leaves, the attenuation and
the phase shift are mainly caused by the moisture content \( M \), affected by the density of the
material \( \rho \), and they are proportional to the length of the microstripline \( l \) which is covered
with the leaves. Additionally, they are also related to the frequency of the microwave and the
temperature of the leaves. Under the conditions of a certain frequency and a certain
temperature, the attenuation is simplified to be a function of \( M, \rho, l \). In general, the
attenuation of a microwave signal at a frequency can be expressed as following equation [2],
[3],

\[
\Delta A = R_A(\rho) \cdot g_A(M) \cdot l
\]  

(2)
where $\Delta A$ is the attenuation, $R_A(\rho)$ is dependence of the attenuation on the density, $g_A(M)$ is a function of the moisture content, and $l$ is the length of the microstrip line overlaid with the leaves. Similarly, the phase shift $\Delta \Phi$ can be also expressed as following.

$$\Delta \Phi = R_\Phi(\rho) \cdot g_\Phi(M) \cdot l$$  \hspace{1cm} (3)

where $R_\Phi(\rho)$ is dependence of the phase shift on the density and $g_\Phi(M)$ is a function of the moisture content.

Ratio of the attenuation to the phase shift is obtained by combining Eq.2 and Eq.3 as Eq.4,

$$\frac{\Delta A}{\Delta \Phi} = \frac{R_A(\rho) \cdot g_A(M)}{R_\Phi(\rho) \cdot g_\Phi(M)} = k(\rho) \cdot \Psi(M)$$  \hspace{1cm} (4)

where $k(\rho)$ is the ratio of $R_A(\rho)$ to $R_\Phi(\rho)$, and $\Psi(M)$ is the ratio of $g_A(M)$ to $g_\Phi(M)$. It shows by Eq.4 that the term of length is eliminated in the equation of $\Delta A/\Delta \Phi$. Then, the ratio of $\Delta A/\Delta \Phi$ is independent from the fluctuation of the $\Delta A$ and $\Delta \Phi$ by the change of the length of the microstrip line which is covered by tea leaves. Hence, the moisture content can be determined independent of the density based on Eq.4 if $k(\rho)$ is a constant. The $k(\rho)$ is not constant over wide range of the density but the influence of density in the $k(\rho)$ is smaller than that in $R_A(\rho)$ or $R_\Phi(\rho)$ itself. These characteristics of $\Delta A/\Delta \Phi$ are useful to conquer the difficulties in the measurement of high moisture content of tea leaves. Thus, the sensor described in this paper measures moisture content by use the ratio of $\Delta A/\Delta \Phi$ in Eq.4.

3 Experiment I

To know the effect of the ratio of $\Delta A/\Delta \Phi$ in the measurement of high moisture content, a microstrip line as shown in Fig. 1 was tested. The data for the attenuation and the phase shift by the leaves were measured using a network analyzer. Tea leaves used in the measurement were defrosted tea leaves of *Yabukita* which had been stored in a freezer for about a year. The *Yabukita* is the name of a kind of tea tree. Defrosted leaves are different from fresh ones in the hardness and elasticity, however, the usefulness of using the ratio in the measurement is made clear even by examining with the defrosted leaves. They had about 315% d.b. of moisture content at the first time of the measurement. After the measurement they were dried for about 2 hours in a room at a temperature of 20-23 degrees Celsius. Then the next measurement was done to the leaves. Similarly, drying and measurement were repeated until the moisture content became about 190% d.b.. The measurement results are shown in the Figure 2.

The data for the attenuation and the phase shift are indicated with dots and crosses, respectively. Their values are shown in the vertical line of the left side of the graph. The ratio of $\Delta A/\Delta \Phi$ is shown by circle and the values are indicated in the vertical line of the right side of the graph. The moisture contents of the samples were measured using an oven drying method and indicated in the horizontal axis of Figure 2 as “Oven moisture Content”. We can see that the results as for the attenuation and the phase shift have poor relations to the moisture content. Their correlation coefficients $R^2$ to their linear regression lines, which are not drawn in the graph, are small. They are 0.49 to the attenuation and 0.004 to the phase shift. The ratio of $\Delta A/\Delta \Phi$ which was calculated from the same data, however, has a fairly
good relation to the moisture. The $R^2$ is 0.8136. We also see from the experiment that using the ratio of $\Delta A/\Delta \Phi$ is a powerful way to measure moisture of tea leaves.

![Fig. 2 Measurement result of defrosted tea leaves](image)

**4 New Sensor**

The electronic board of the new sensor based on the idea explained above is shown in Figure 3. The left side of the board is the electronic circuit of the sensor and the right side is the place on which tea leaves are put to be measured. In an actual sensor system the electric board is fixed at the top position of the frame of the system. The leaves to be measured are lifted up and pressed with a proper pressure to the microstripline on the measurement place of the board. The reason of taking such arrangement is that the microstripline at the measuring place is not soaked in the excess water percolated from the leaves. The electronic circuit on the board has some devices to detect the information for the moisture content of the leaves from signals for the attenuation or the phase shift of the microwave propagated through the measuring place.

![Fig. 3 The electronic board of the new sensor](image)

As shown in Figure 4, the microwave of 3 GHz generated by a voltage controlled oscillator (VCO) travels down the microstripline being affected by the leaves itself and the moisture contained in the leaves. The information for the moisture of the leaves is transferred to the two signals of the attenuation and the phase shift through the Heterodyne Detector I and II, the Log-Amp., the Phase detector, and etc.. The output voltages of the attenuation signal or
the phase shift signal are adjusted to certain voltages proportional to decibel or degree, respectively. The moisture content of tea leaves is calculated by a computer using the two outputs correspond to $\Delta A$ and $\Delta \Phi$ in Eq.4.

Fig. 4 The block diagram of the new sensor

5 Experiment II

The characteristics of the new sensor were examined at a factory currently working for producing green tea using fresh tea leaves just cut from tea tree, not defrosted tea leaves. In a factory several machines are lined in series to make tea leaves good Japanese green tea. In the process cultivated tea leaves are steamed at first, and dried using several machines. The machine examining the new sensor was a second drying machine named Sojuki shown in Figure 5. It has an ability to process a batch of tea leaves of 180 kg within about 15 minutes. The new sensor system installed the same model of the sensor shown in Figure 3 was attached on the belly of the drum of the drying machine as the picture of Figure 6. The examination was done using two batches of leaves of Yabukita which is the most popular kind of tea tree in Japan. One of the batches was the leaves grown at a field in a plain near of sea and another was that grown in a mountains. The machine used in the test was the second dryer in the process, then the moisture content of tea leaves carried into the machine was not as high as 300% d.b.. In this experiment the moisture was around 200 % d.b..

Fig. 5 The photo is the machine used for the experience II. It is a second drying machine named “Sojuki”. The standing person was sampling the leaves for reference of the moisture content.

Fig. 6 The new sensor attached on the belly of the drum of the drying machine of Sojuki.
The tea leaves carried out from a first drying machine were conveyed and injected into the drum of the second drying machine of the Sojuki. After the leaves filled the drum, they were stirred in the rotating drum and dried by warmed air of 90-100 degree in Celsius. In the drying process some leaves being stirred in the drum were gathered into a sampling box of the sensor system. They were lifted up to the measurement place of the sensor board and measured. After that they were returned into the drum. It took about 35 seconds for one cycle of the measurement.

The results in Figure 7 show the data obtained from the sensor system at about 3 minutes interval of the measurement. The time interval needed to take out some leaves from the drum to know the moisture content of the leaves in the drum. They were sealed in an envelope as a reference leaves of the moisture content. The reference leaves were measured by the oven method. The moisture contents of the leaves measured by the sensor and the reference leaves packed in the envelope were different in each other. But, we supposed in the experiment that the two groups of leaves have the same moisture content and the true moisture content is obtained by the oven method to the reference leaves.

Fig. 7 Measurement results by the new sensor for fresh tea leaves

In Figure 7, the vertical axis of the left side is scaled in arbitrary unit corresponding to attenuation in decibel or phase shift in degree. Another vertical axis in the right side is also scaled in arbitrary unit corresponding to the ratio of $\Delta A/\Delta \Phi$. The attenuation plotted in dots, has little change corresponding to the change of oven moisture content of the leaves. Concerning to the phase shift plotted in crosses, the values of them are slightly decreased with increasing the oven moisture content. These two signals have poor correlation coefficient of 0.002 on the attenuation signal and 0.372 on the phase shift signal. It is evident that both of the output signals on the attenuation or on the phase shift by itself alone can not measure moisture content of the high moist leaves.

Contrary to the results of the attenuation or the phase shift, the ratio of the two output signals of $\Delta A/\Delta \Phi$, represented by circles, has a good linear relation whose correlation coefficient is 0.941. The error of the measurement was about 3 % in the readings of the sensor over the whole measured range of 155 to 215 % d.b.,
6 Conclusions

The high moist tea leaves put on the microstripline give complicated effects on the microwave propagating in the line. Hence, the output signals on the attenuation or the phase shift of the wave can not clearly show some information on the moisture content of the leaves. However, it is evident from the results of the experiment I and II that using the ratio of $\Delta A/\Delta \Phi$ is very effective to measure moisture contents of high moist or heavily wet tea leaves. The measured range in fresh tea leaves was confirmed to be 155 to 215 % d.b. from the experiment II. Moreover, it is expected that the measurable range by the ratio of $\Delta A/\Delta \Phi$ will be wider enough in measuring high moist fresh leaves as high as 300 % d.b. because the measurable range in the Experiment I was 190-315 % d.b..

7 Acknowledgement

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References


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ABSTRACT. In many fields the concept and measurement of quality is of prime importance. The fish industry is no exception to this and many sensory approaches have been devised to quantify this rather intangible property. There have been several reports published of late on the measurement of fish quality and emanating from the EU 5th framework project ‘SEQUID’. This project has concentrated on the measurement of the dielectric properties of fish tissue as a function of time both in frozen and chilled storage.

The many deteriorative biochemical processes that take place during the gradual spoilage of such materials have a subtle influence on the dielectric properties across the spectrum but notably in the microwave frequency region. In this region the complex interactions of water, solutes and structure forming proteins are systematically changed by death and decay. Chilling or freezing may slow these processes but such preservation techniques do not halt them entirely.

The SEQUID project has shown that it is possible, using a combination of time domain reflectometry and multivariate analysis, to predict certain quality related variables, both sensory and biochemical, with comparable accuracy to existing methods. These results are presented in this paper.

Keywords: Time domain reflectometry, quality, fish

1 Introduction

What is quality? That question is possibly one of the great imponderables of food science and is the inspiration for the ‘intangible’ part of the title of this paper. The short answer is that quality is whatever it is defined to be, whether it be a definition based on some semi-objective system of sensory scoring such as the Quality Index Method (QIM) or on some directly measurable variable such as time and temperature of cold storage, or even on the level of some biochemical degradation of the foodstuff. This work has concerned itself not with the definition of any such variables but with their tractability using a suitably calibrated instrumental method.

During storage, several post mortem changes in the fish muscle affect quality via mechanisms that could also alter the dielectric properties. Most obvious are changes affecting interactions
between water and proteins. However, degradation processes leading to the formation of polar compounds are also of importance. For obvious reasons sensory attributes are the ultimate and most important consequences of biochemical degradation of protein and lipids and focus has been on odour and appearance by expert panels, using for example Quality index methods QIM-schemes [1].

In the EU, new regulations concerning traceability of foods [2] have been in force since 1st January 2005. Determination of quality is now an even more important issue.

Against this background the objectives of the project ‘SEQUID’ (Seafood Quality Identification) were:

- To develop a new rapid method for determination of the quality of seafood products.
- To construct a prototype hand-held instrument suitable for use in the fish processing industry.

Some preliminary results using a commercial network analyser were presented at the last ISEMA conference in New Zealand [3].

2 Methods and Materials

2.1 Time Domain Reflectometry

The methodology used in earlier related work [3], [4] relied on measurement of the complex dielectric properties of the samples as a function of frequency and subsequent transformation of these spectra using the method of Principal Component Analysis (PCA). PCA effectively reconstructs the information in the input data into a new form in which the sources of variation are ‘modelled’ rather than some attempt at a physical model of the system. The PC scores can then be used either as descriptor variables for discriminant analysis (assigning samples to predefined groups) or as variables in regression equations designed to predict desired quality variables (PCR).

In the work to be described here, the time domain response of the samples to an input step pulse (rise time 60 psec) delivered to the surface of the samples by an open ended coaxial probe was measured. A family of several mathematical transformations links the frequency and time domains, the most well known being of course the Fourier transform and its inverse. For the time varying data acquired in this instrument the inverse Fourier transform in its most general form can be written as in equation (1).

\[
h(t) = \frac{1}{2\pi} \int_{-\pi}^{\pi} g(\omega)e^{-i\omega t} d\omega
\]

(1)

\(h(t)\) is a time dependent function the Fourier transform of which, is a frequency dependent spectrum \(g(\omega)\). Examination of this equation shows that at any instant \(t\) every component part of the spectrum contributes to the value of \(h\). Because the subsequent PCA depends only on variations in \(g\) then transformation of \(h(t)\) to the frequency domain for PCA is not required since the same variations are present in \(h(t)\). In the method described here, truncation of the pulse is permitted at any time; the same information on variation is still contained in the collected data.

The basis of the portable TDR system used was also described at the previous ISEMA conference [5]. The sample positions measured included measurements directly on the skin,
on the flesh side of fillets and on minced or blended samples. Several replicate measurements were made on the same samples and at the same times as the various quality related analyses and measurements described later. Such measurements were repeated at regular intervals on new samples.

When the fish quality reached its rejection level the TDR data were subjected to principal component analysis and regression (PCA and PCR), partial least squares regression (PLSR) and processing by a non-linear artificial neural network (ANN). The aim of these various approaches was to produce regression equations capable of predicting the values of the various quality related factors.

2.2 Fresh Fish

An adequate number of cod (Gadus morhua), salmon (Salmo salar), sardine (Sardina pilchardus), scad (Trachurus trachurus), silver and black scabbard fish (Lepidopus caudatus and Aphanopus carbo), Senegalese sole (Solea senegalensis), seabass (Dicentrarchus labrax) and gilthead sea bream (Sparus aurata) were obtained as soon as possible after slaughter. All fish were kept in ice under refrigerated conditions (2 ± 2°C), until the end of their commercial life, fresh ice being added as necessary. At 1-2 day intervals, samples were taken for measurement with the TDR prototype and for quality analysis.

2.3 Frozen Fish

Hake (Merluccius capensis and M. paradoxus), commercialised together under the generic name of hake, were caught in Namibian waters (South Atlantic). The fish, deep frozen at sea, and transported at -20°C ± 2°C, arrived at the laboratory after 2 months at sea. The fish was divided into 3 lots and stored up to 10 months at -10, -20 and -30°C. The fish was analysed initially for composition and periodically for various quality related variables until the end of storage.

Double frozen Alaska pollock (Theragra chalcogramma) and cod (Gadus morhua) of known history were obtained from commercial catches, then thawed, filleted and frozen in blocks before delivery to the laboratory. The four different lots of frozen fish blocks were divided on arrival into three cold stores at -10°C, -20°C and -40°C. The storage times covered within this study were 3 – 11 month and 5 – 13 month respectively.

Cod was also caught by a research vessel (RV Walther Herwig III) in the Barents Sea, for frozen storage under four different storage conditions (–14°C, –20°C, –28°C and a double freezing experiment at –20°C).

3 Results and Discussion

The multivariate analysis of the prototype output has yielded predictive equations for variables such as time of storage (in ice or in the frozen state), various sensory variables and others related to change in quality. In the following tables of regression parameters the range of the calibration variables has also been included to give some idea of the magnitude of the uncertainty represented by the error terms.
3.1 Time of Frozen Storage

The results for the prediction of time of frozen storage are summarised in Table 1. In that table a comparison may be seen between the PCR and ANN approaches. The RMSE (root mean square error) values are used to make a better comparison with ANN for which standard errors of calibration and prediction (SEC and SEP) are not easy to calculate, because of unknown degrees of freedom. In general no overall advantage was found in using PLSR instead of PCR but when there are sufficient samples, then ANN performs well.

Table 1 Regression parameters for frozen fish calibrations to predict time (days) of storage. The different measurement laboratories are indicated.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Range (days)</th>
<th>Samples</th>
<th>pcs</th>
<th>$R^2$</th>
<th>RMSE$_c$</th>
<th>RMSE$_v$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alaska Pollock (SIK) (fillet)</td>
<td>110-353</td>
<td>136</td>
<td>8</td>
<td>75.7</td>
<td>40.0</td>
<td>42.8</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>94.7</td>
<td>29.1</td>
</tr>
<tr>
<td>Alaska Pollock (SIK) (blended)</td>
<td>110-353</td>
<td>77</td>
<td>8</td>
<td>70.6</td>
<td>39.3</td>
<td>44.5</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>82.6</td>
<td>31.3</td>
</tr>
<tr>
<td>Cod (SIK) (blended)</td>
<td>110-406</td>
<td>144</td>
<td>8</td>
<td>72.5</td>
<td>32.0</td>
<td>34.2</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>95.0</td>
<td>19.6</td>
</tr>
<tr>
<td>Cod (SIK) (fillet)</td>
<td>110-406</td>
<td>139</td>
<td>9</td>
<td>76.6</td>
<td>38.6</td>
<td>41.5</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>88.2</td>
<td>20.3</td>
</tr>
<tr>
<td>Cod (BFEL) (double and single frozen)</td>
<td>38-260</td>
<td>100</td>
<td>8</td>
<td>89.0</td>
<td>36.8</td>
<td>40.2</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>98.8</td>
<td>25.3</td>
</tr>
<tr>
<td>Cod (BFEL) (fillet)</td>
<td>38-260</td>
<td>100</td>
<td>7</td>
<td>83.2</td>
<td>45.6</td>
<td>50.0</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>99.5</td>
<td>30.7</td>
</tr>
<tr>
<td>Cod (BFEL) (double frozen)</td>
<td>38-260</td>
<td>20</td>
<td>6</td>
<td>96.6</td>
<td>15.0</td>
<td>27.6</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>99.3</td>
<td>19.9</td>
</tr>
<tr>
<td>Cod (BFEL) (once frozen)</td>
<td>38-260</td>
<td>80</td>
<td>7</td>
<td>87.3</td>
<td>41.3</td>
<td>45.8</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>99.1</td>
<td>32.1</td>
</tr>
<tr>
<td>Cod (SIK+BFEL) (blended)</td>
<td>30-406</td>
<td>244</td>
<td>8</td>
<td>83.8</td>
<td>39.6</td>
<td>41.0</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>94.4</td>
<td>35.9</td>
</tr>
<tr>
<td>Cod (SIK+BFEL) (fillet)</td>
<td>38-406</td>
<td>209</td>
<td>8</td>
<td>70.8</td>
<td>54.0</td>
<td>56.2</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>98.0</td>
<td>31.0</td>
</tr>
<tr>
<td>Hake (IF-CSIC)</td>
<td>69-310</td>
<td>55</td>
<td>8</td>
<td>84.2</td>
<td>28.6</td>
<td>34.0</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>99.8</td>
<td>7.0</td>
</tr>
</tbody>
</table>

Although the results obtained with frozen hake and Alaska Pollock are satisfactory, particularly noteworthy in this table are the excellent results for the storage time prediction of frozen cod, both at BFEL and SIK separately. This was especially the case using the ANN approach to data analysis. Combination of these two independent data sets into one overall cod calibration, still gives an $R^2$ of 94.4% with the ANN, compared to the separate values of 98.8% and 95.0% at BFEL and SIK respectively. Plots of the combined ANN data are shown in fig 1.
Fig. 1 Predicted time of storage for the combined cod results of SIK and BFEL using an ANN.

This is an encouraging result since it demonstrates that two instruments used in different laboratories by different operators, on fish of quite different origins and treatments can give equivalent results. The prediction of temperature of storage was less successful, largely due to the variable temperature history of most of the samples i.e. different times of storage at – 20ºC, prior to being received at the laboratories.

Table 2 Regression parameters for chilled fish calibrations to predict time of storage in ice. Absence of ANN data implies too few samples or not calculated.

<table>
<thead>
<tr>
<th>Sample/ measurement position</th>
<th>Range (days)</th>
<th>Samples</th>
<th>pcs</th>
<th>$R^2$</th>
<th>RMSEc</th>
<th>RMSEv</th>
</tr>
</thead>
<tbody>
<tr>
<td>Black scabbard middle</td>
<td>PCR</td>
<td>0-9</td>
<td>16</td>
<td>4</td>
<td>53.5</td>
<td>2.0</td>
</tr>
<tr>
<td>Cod mince</td>
<td>PCR</td>
<td>0-18</td>
<td>75</td>
<td>8</td>
<td>87.8</td>
<td>2.1</td>
</tr>
<tr>
<td></td>
<td>ANN</td>
<td></td>
<td></td>
<td></td>
<td>99.5</td>
<td>0.9</td>
</tr>
<tr>
<td></td>
<td>PCR</td>
<td>0-18</td>
<td>75</td>
<td>8</td>
<td>88.7</td>
<td>2.0</td>
</tr>
<tr>
<td></td>
<td>ANN</td>
<td></td>
<td></td>
<td></td>
<td>99.7</td>
<td>1.2</td>
</tr>
<tr>
<td>Scad mince</td>
<td>PCR</td>
<td>0-7</td>
<td>14</td>
<td>4</td>
<td>77.0</td>
<td>1.1</td>
</tr>
<tr>
<td></td>
<td>PCR</td>
<td>0-7</td>
<td>14</td>
<td>4</td>
<td>83.5</td>
<td>0.9</td>
</tr>
<tr>
<td>Salmon (2-3 kg) skin</td>
<td>PCR</td>
<td>0-18</td>
<td>40</td>
<td>3</td>
<td>70.6</td>
<td>3.2</td>
</tr>
<tr>
<td>Sardine mince</td>
<td>PCR</td>
<td>0-4</td>
<td>12</td>
<td>8</td>
<td>74.1</td>
<td>0.3</td>
</tr>
<tr>
<td></td>
<td>PCR</td>
<td>0-4</td>
<td>12</td>
<td>8</td>
<td>85.0</td>
<td>0.2</td>
</tr>
<tr>
<td>Sea bass skin</td>
<td>PCR</td>
<td>2-23</td>
<td>50</td>
<td>8</td>
<td>82.0</td>
<td>2.7</td>
</tr>
<tr>
<td></td>
<td>ANN</td>
<td></td>
<td></td>
<td></td>
<td>98.7</td>
<td>1.3</td>
</tr>
<tr>
<td>Sea bream skin</td>
<td>PCR</td>
<td>1-17</td>
<td>40</td>
<td>8</td>
<td>68.4</td>
<td>3.1</td>
</tr>
<tr>
<td></td>
<td>ANN</td>
<td></td>
<td></td>
<td></td>
<td>82.7</td>
<td>3.5</td>
</tr>
<tr>
<td>Senegalese sole skin</td>
<td>PCR</td>
<td>1-27</td>
<td>60</td>
<td>8</td>
<td>68.4</td>
<td>4.6</td>
</tr>
<tr>
<td></td>
<td>ANN</td>
<td></td>
<td></td>
<td></td>
<td>87.6</td>
<td>4.9</td>
</tr>
<tr>
<td>Silver scabbard dorsal</td>
<td>PCR</td>
<td>0-11</td>
<td>16</td>
<td>4</td>
<td>63.2</td>
<td>1.9</td>
</tr>
</tbody>
</table>
3.2 Time of Chilled Storage

For the fish stored in ice, the predictions of time of storage are seen in table 2. The ANN results are often poor due to the limited number of samples. Apart from sample presentations described as fillet, minced or spine-side, all TDR measurements were made directly on the skin of the whole fish or fillets. In some cases the exact region has been specified e.g. dorsal, middle or ventral. These results demonstrate that there could be some problems in using the prototype with small fish such as sardine, or fish of a slender profile such as the two species of scabbard fish. With sardine for example, where high fat content predicates lower water content than in fat-free flesh, there could be significant reflections of the TD pulse from the opposing back surface into the sensor. This is perhaps a problem that needs some investigation.

3.3 k-Value

The k-value is a measure of the gradual loss of adenosine triphosphate (ATP) after death. For many of the samples it could be predicted to a reasonable accuracy. These results are shown in table 3. Only the PCR results are shown since in general there were too few samples for ANN analysis. In this case the values shown are the SEC and SEP with the adjusted coefficient of determination. Also only the best sample configurations for the measurements have been chosen.

Table 3 PC regression parameters for chilled fish calibrations to predict k-value.

<table>
<thead>
<tr>
<th>Sample and measurement position</th>
<th>Range of k-value</th>
<th>Samples</th>
<th>R² adj</th>
<th>SEC</th>
<th>SEP</th>
</tr>
</thead>
<tbody>
<tr>
<td>Black scabbard ventral</td>
<td>18-63</td>
<td>16</td>
<td>69.5</td>
<td>6.2</td>
<td>13.5</td>
</tr>
<tr>
<td>Scad fillet skin side</td>
<td>11-30</td>
<td>14</td>
<td>82.9</td>
<td>2.4</td>
<td>4.3</td>
</tr>
<tr>
<td>Sea bass skin</td>
<td>14-80</td>
<td>50</td>
<td>85.0</td>
<td>7.3</td>
<td>12.5</td>
</tr>
<tr>
<td>Sea bream skin</td>
<td>9-45</td>
<td>20</td>
<td>71.5</td>
<td>3.6</td>
<td>6.2</td>
</tr>
<tr>
<td>Senegalese sole skin</td>
<td>5-38</td>
<td>30</td>
<td>79.2</td>
<td>4.2</td>
<td>6.0</td>
</tr>
<tr>
<td>Silver scabbard ventral</td>
<td>13-43</td>
<td>16</td>
<td>75.2</td>
<td>3.9</td>
<td>6.7</td>
</tr>
</tbody>
</table>

3.4 Sensory Assessment

The k-value is an important quality variable for chilled fish but is not relevant to frozen fish. For both raw chilled and frozen fish it is possible to construct Quality Index Method scores (QIM) and others based on various aspects of appearance and sensory assessments. The prediction of these from the prototype data is shown in table 4. As can be seen there is good agreement between the sensory scores and the TDR data. For the frozen hake the best result is for the overall cooked sensory score. All these results are important since they mean that the instrument can relate directly to the consumers’ appreciation of quality. Even more significant is perhaps the fact that the errors of prediction are often comparable in magnitude to those inherent in the subjective determination of quality indices by expert panels. Fig 2 shows the results for cooked hake and it is immediately clear that both the time and temperature of storage are eliminated when dealing only with sensory attributes.
Table 4 PC regression parameters for chilled and frozen fish calibrations to predict QIM scores

<table>
<thead>
<tr>
<th>Sample</th>
<th>Range of QI</th>
<th>Samples</th>
<th>pcs</th>
<th>R²adj</th>
<th>SEC</th>
<th>SEP</th>
</tr>
</thead>
<tbody>
<tr>
<td>Black scabbard</td>
<td>2-19</td>
<td>14</td>
<td>8</td>
<td>89.1</td>
<td>2.3</td>
<td>3.8</td>
</tr>
<tr>
<td>Cod</td>
<td>0-20</td>
<td>75</td>
<td>5</td>
<td>86.0</td>
<td>2.4</td>
<td>2.5</td>
</tr>
<tr>
<td>Scad</td>
<td>4-18</td>
<td>12</td>
<td>4</td>
<td>87.8</td>
<td>2.0</td>
<td>2.3</td>
</tr>
<tr>
<td>Salmon (2-3 kg)</td>
<td>3-24</td>
<td>40</td>
<td>5</td>
<td>73.6</td>
<td>3.2</td>
<td>3.5</td>
</tr>
<tr>
<td>Sardine</td>
<td>1-17</td>
<td>24</td>
<td>9</td>
<td>72.7</td>
<td>3.0</td>
<td>4.0</td>
</tr>
<tr>
<td>Sea bass</td>
<td>2-21</td>
<td>50</td>
<td>4</td>
<td>80.4</td>
<td>2.8</td>
<td>2.9</td>
</tr>
<tr>
<td>Sea bream</td>
<td>0-17</td>
<td>40</td>
<td>4</td>
<td>61.2</td>
<td>3.2</td>
<td>3.4</td>
</tr>
<tr>
<td>Silver scabbard</td>
<td>1-16</td>
<td>16</td>
<td>9</td>
<td>82.1</td>
<td>2.1</td>
<td>3.2</td>
</tr>
<tr>
<td>Hake (frozen/raw)</td>
<td>7-13</td>
<td>50</td>
<td>3</td>
<td>60.2</td>
<td>1.4</td>
<td>1.5</td>
</tr>
<tr>
<td>Hake (frozen/cooked)</td>
<td>3-7</td>
<td>50</td>
<td>5</td>
<td>80.0</td>
<td>0.8</td>
<td>0.8</td>
</tr>
</tbody>
</table>

As noted above the errors due to the instrument are comparable to those inherent in the QIM assessment. In fact since the latter is used to calibrate the instrument then obviously it can never give a more accurate result than that provided by the calibration data.

![Predicted sensory scores for cooked frozen hake stored for up to 11 months.](image)

Fig. 2 Predicted sensory scores for cooked frozen hake stored for up to 11 months.

Figure 3(a) represents similar mean QIM for a combination of 3 species, each with comparable storage times. In Fig 3(b) the QIM data has been normalised by division with the maximum score attainable for each species. In the prediction of days in ice (Fig 4) there is also generally good accuracy, marred only by two apparent outliers for sea bass and salmon at the end of the storage period. However, this may be due to the fish already having passed the rejection level. This is a preliminary attempt to produce a generalised calibration for all species and could be improved by a better knowledge of the potential shelf-life of each species. The inclusion of fish with much shorter shelf life in the analysis was not successful.
QIM has the advantage that it is a system based on the summation of scores of individual attributes. This has a tendency to smooth out anomalous assessments of any particular variable. This is probably the reason why the use of single attributes was not found to correlate well with the instrument predictions.

![Graph showing predicted mean QIM using ANN for a combined set of data of cod, sea bass, and salmon stored in ice for up to 20 days.](image)

**Fig. 3a** Predicted mean QIM using ANN for a combined set of data of cod, sea bass, and salmon stored in ice for up to 20 days. $R^2 = 71.3$, RMSE$_C = 3.1$, RMSEP = 2.7

![Graph showing predicted mean QIM/QIMmax using ANN for the same combination.](image)

**Fig. 3b** Predicted normalised QIM using ANN for the same combination. $R^2 = 75.2$, RMSE$_C = 0.13$, RMSEP = 0.11

It must be stressed that when attempts are made to correlate the TDR measurements with other sensory (QIM) or metabolic (K-value) variables, by generating multivariate predictive equations, this is not in the belief that the dielectric properties are directly related to them but simply as a device for showing their common dependence on time and/or temperature and to exploit that dependence.

![Graph showing predicted storage time using ANN for a combined set of data of cod, sea bass, and salmon stored in ice for up to 20 days.](image)

**Fig. 4** Predicted storage time using ANN for a combined set of data of cod, sea bass, and salmon stored in ice for up to 20 days. $R^2 = 70.8$, RMSE$_C = 2.9$, RMSEP = 2.6
4 Conclusions

To recapitulate, the objectives of the project ‘SEQUID’ (Seafood Quality Identification) were;
- To develop a new rapid method for determination of the quality of seafood products.
- To construct a prototype hand-held instrument suitable for use in the fish processing industry for indicating quality.

Because the dielectric properties depend very much on the behaviour of the water molecules in the tissue, then any changes in solute concentration or water structuring by hydrophilic or hydrophobic mechanisms, will have an effect on the dielectric spectrum. Such effects may be so subtle as to be invisible to any methodology other than by applying multivariate analysis to the data. This is readily confirmed in practice where the changes due to spoilage are not usually evident to the eye in the shape of the time domain reflected pulses, but sound multivariate predictive equations can be generated.

In general the TDR prototype has managed to confirm the measurements made in the first exploratory phase of the project. Storage times of both chilled and frozen fish can be predicted to a reasonably high accuracy. Prediction of the QIM variables is equally satisfying and the overall accuracy of such predictions is of the same order as for the subjective scores. Obviously it can never be better than those, because they are the calibrants. Generally, other individual important sensory variables were not predicted well.

Summarising, the most important results have been
- Storage times of both chilled and frozen fish can be predicted to a reasonably high accuracy.
- Prediction of the QIM variables is as accurate and of the same overall magnitude as for the subjective scores.
- For both chilled and frozen fish the TDR measurements relate to the quality perceived by the consumer.
- K-value is predicted well for many species.
- Finally, for chilled fish, there are indications that a calibration based on remaining shelf life, as defined by other known variables, could be species independent.

5 Acknowledgements

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Microwave Sensing for Food Structure Evaluation

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ABSTRACT. Changes in muscle structure during bovine meat ageing and fish freezing and alteration are major industrials problems. For a better competitiveness, and responding to the consumer’s quality demand, structure has to be evaluated on line. We present here a polarimetric microwave (10 to 24 GHz) method based on dielectric anisotropy decrease during process. This method is illustrated with the discrimination of fresh and frozen-thawed fish fillets and with meat ageing monitoring.

Keywords: structure, muscle, polarimetry, microwaves

1 Introduction

Microwave characterization began in our laboratory in 90ths for bovine carcass tissues discrimination (bone, muscle, fat and connective tissue). Then, we specially worked on muscle quality evaluation and on muscle to meat transformation during ageing. Among striated muscle (mammals and fish) qualities, the most important is structure because it is directly linked to meat tenderness (texture) and fish alteration. Structure measurement, even if it is possible to access it with microwave spectroscopy (spectral changes are mainly due to intra cellular liquid diffusion), is easy to access by polarimetric measurements. This particular technique is presented here applied to muscle (bovine and fish) structure evaluation.

2 Context

Fish products consumption in France strongly increases. All the species taken into account, French annual consumption is about 1 300 000 tons (full fish weight equivalent), on which 500 000 tons are imported. Importation is mainly due to the decrease of natural resources in occidental countries. Commercial exchanges rise and high fragility of fish products lead to new tools development for fish freshness objective measurement. Moreover, fish products are more expensive than in other food industries, and then quality assurance is now a strong demand all along fish industry. Fresh and thawed differentiation is a great target because there is a large gap between fresh and frozen fish prices. For wild fishes, regular supplying is difficult and thawed products are often sale as fresh. It is possible to discriminate fresh and thawed entire fish with fish skin electrical properties using the Torrymeter [1]. This measure is not possible on peeled fillets, which is the more common commercial supply of fish. Concerning peeled fillets, it is possible to use different enzymatic measuring out (for instance [2]), but this method could not be considered as a reference. Other methods could be used to detect thawed fillets: nuclear magnetic resonance [3], frontal fluorescence spectroscopy [4], and magnetic imaging resonance [5], but they are not even now usable for on line measurements.
After slaughter, bovine carcasses are stored seven to twelve days. During this ageing, meat becomes tenderer. This time ageing depends on the animal and the tenderization rate of bovine meat is particularly slow, which poses storage problems and is cost effective. Meat is usually sold within one or two weeks and not all pieces of meat are fully aged at this time. Consequently, meat can be tough if sold to quickly (which is against demands of the consumers for a constant tender meat) or in the opposite side, a longer time of ageing is expensive and reduce competitiveness [6]. The solution is a sensing method that can predict the best ageing duration for each pieces of meat. Such sensing should be very fast (given online measurements), non-destructive, inexpensive, waterproof (for sanitary considerations) and safe.

Many efficient methods of non-destructive monitoring, based on the measurement of reflection and transmission coefficients of electromagnetic waves at microwave frequencies, have been proposed for medical applications [7] or industrial process control [8]. Some of these can also be applied in the food industry [9, 10]. However, this kind of sensing could be used much more widely; simple microwave measurements could be usefully applied to food process control, since microwaves are strongly absorbed in water, the main constituent of a lot of food products.

3 Polarimetric Measurement

3.1 Muscle Anisotropy

Muscles are very high anisotropic dielectric material because of their geometric organisation and their components nature (Fig. 1): from muscle to sarcomere filaments, components are elongated and more or less parallel to form kinds of bundles of conjunctive tissue and myofibers which have very different intrinsic dielectric properties [11]. During rigor and maturation, structural damages appear [12] that should produce a decrease in the dielectric anisotropy.

The anatomy of fish muscle [13] is different from the anatomy of terrestrial mammals, in that the fish lacks the tendinous system connecting muscle bundles to the skeleton of the animal. Instead, fish has muscle cells running in parallel and connected to sheaths of connective tissue (myocommata), which are anchored to the skeleton and the skin. The bundles of parallel muscle cells are called myotomes (Fig. 2).

All muscle cells extend the full length between two myocommata, and run parallel with the longitudinal direction of the fish.
The fillet is heterogeneous in that the length of the muscle cells varies from the head end (anterior) to the tail end (posterior). The longest muscle cells in cod are found at about the twelfth myotome counting from the head, with an average length around 10 mm in a fish that is 60 cm long. The diameter of the cells also varies, being widest in the ventral part of the fillet.

Both for mammals and fishes, the muscle tissue is composed of striated muscle. The cell is surrounded by a sheath of connective tissue called the sarcolemma. The myofibrils contain the contractile proteins, actin and myosin. These proteins or filaments are arranged in a characteristic alternating system making the muscle appear striated upon microscopic examination. All these characteristics contribute to dielectric anisotropy of muscle.

### 3.2 Method

The principle of the polarimetric method consists in the measurement of variations of interaction between a linearly polarised electromagnetic wave and an anisotropic material when the angle theta between the electric field $\vec{E}$ and the axis of the material varies, as shown in Fig. 3. A linearly polarised wave is an electromagnetic wave whose the direction of the electric field $\vec{E}$ doesn't change in space and time. When a linearly polarized wave is reflected on an anisotropic material, the reflected wave is no more linear but elliptic (Fig. 4). Ellipticity ($a/b$) and rotation ($\alpha$) depend on the anisotropic material. In an anisotropic material, dielectric permittivity ($\varepsilon$) is different according to the direction ($\theta$) in which the electric field $\vec{E}$ "see" the material. The intensity of a linearly polarised wave after passing through an anisotropic material is given by:

$$I = r_{E//} \cos^2 \theta + r_{H//} \sin^2 \theta$$

(1)

where $r_{E//}$ and $r_{H//}$ are anisotropy constants characterising the material. Linearly polarisation can be done thanks to a rectangular horn antenna for free space measurements, or thanks to a rectangular probe (Fig. 5) for contact (but non invasive) measurements.
This way for polarimetric measurements is the well known method called open ended rectangular waveguide [14]. The rectangular waveguide used as a probe allow the linear polarisation of the guided wave. Reflection is done at the probe-sample interface. As shown on Fig. 5, the probe is ended with a polymer joint which is almost transparent for the microwave. Measurements of complex reflection coefficients were performed with an automatic network analyser. The study described here cover the frequency range 8.2 to 12.4 GHz and was carried out using a X-band probe. Another experiment at 16 and 24 GHz is in progress.

4 Thawed Fish Fillets Discrimination

The work presented here concern 8 salmons (16 fillets). Each fillet is got fresh (3 or 4 days post mortem (PM)) and peeled. Fishes are farmed salmons from Norway, Scotland or Ireland. Dielectric properties of right fillets are measured before alteration (fresh fillets) at 3 or 4 days PM. Left fillets are stored for 10 days on ice in a refrigerated (5°C) chamber, and the dielectric properties are measured at 10 days PM. Immediately after measurements, all fillets are placed in bags without air. Half of them are frozen quickly (in -20°C alcohol) and the others are frozen slowly (in -20°C air). Before measurement, frozen fillets are thawed at 5°C during 12 hours. For each fillet, measurements are done in 9 places dispatched on its internal face (Fig. 6).
For each place, the 10 GHz probe is applied 4 times on the fish successively with electric field parallel to muscle fibres, then perpendicular, and again perpendicular and parallel. This series is done 4 times.

Data acquisition is automatically done by software, which also calculates the average anisotropy for each place on the fillet. Statistic treatment is performed by SAS/STAT® Release 6.03 Edition commercial software. Correlation is computed between the parameter "anisotropy" and the parameter: "fresh or thawed".

Statistical analyse shows a good correlation between anisotropy and the parameter fresh or thawed fillet whatever the other parameters “number of days PM” (alteration), “slow or quick freezing” and “place on the fillet” are. Average anisotropy changes from 1.007 for fresh fillets to 0.969 for thawed fillets. This shift is highly significant (alpha < 5%).
Figure 7 gives the frequencies of anisotropy values for fresh (black) and thawed (grey) fillets. We can see the shift of mean value and also a change in the distribution form. For the fresh fillets, distribution is almost Gaussian and become very scattered after freezing. In a homogeneous muscle, with well-oriented muscle fibres, microwave reflected coefficient is upper for electric field parallel to fibres. This is due to dielectric properties of an anisotropic media with alternative conducting (extra and intra cellular liquid) and insulating (cell membranes) media. So, in homogeneous fresh fish muscle, anisotropy should be > 1. Regarding the active area of our probe (1 cm x 2 cm), fish muscle is not homogeneous due to myocepts (fat deposition) presence. Myocepts anisotropy competes with myofibres anisotropy because these structures are orthogonal each other (myocept anisotropy < 1). Membranes disappear and myofibres anisotropy decreases during freezing. Instead, myocepts anisotropy does not change because freezing doesn't affect adipose tissues. This could explain the significant shift of the mean anisotropy from 1.007 to 0.969 during freezing. The change in the histogram representation (Fig. 7) (thawed fillets anisotropy values are more scattered) may be due to the heterogeneity of the myocepts organisation all along the fillets. Myocepts are very pronounced on the bottom of the fillets (measurements number 1 to 4 on Fig. 6) and almost non-existent on the lateral parts (measurements number 5 and 6). So, we observe that freezing (both slow and fast) leads to a significant change of the anisotropy values, but it is not possible to discriminate fresh and thawed fillets. This study was completed with the measurement of electrical impedance anisotropy and this last parameter seems to be better for fresh and thawed fillets discrimination. However, microwave measurement which is non invasive have to be improved. Use of higher frequencies (under way) is promising.

5 Bovine Meat Ageing Monitoring

In the study presented here, the muscle under test was a *semitendinosus* muscle, from a three years old Charolais cow. It is thick enough to ovoid a second reflection at the muscle-air interface.

![Fig. 8 Amplitude of reflected wave at 31 hours, for parallel and perpendicular position](image)

![Fig. 9 Anisotropy decrease during ageing](image)
The side under test is cut parallel to muscle fibres. Muscle was taking from the carcass just after slaughter and then stored at 4°C. Measurements were done at 31, 49, 56, 72, 79, 96 and 102 hours post mortem. At each post mortem time, two measurements were done with electric field parallel to muscle fibres, and two other with electric field perpendicular to muscle fibres. For each time post mortem, in order to minimise mistake measurements due to non homogeneity of meat, we compute the mean value between the two parallel reflection data and between the two perpendicular reflection data. Whatever is meat ageing, we observe an anisotropy in meat dielectric properties, as shown for instance on Fig. 8 for the 31 hours old meat. Parallel reflection is higher than perpendicular.

To point out the decrease of the dielectric anisotropy during ageing, we compute the mean anisotropic ratio on the frequency range:

\[
\frac{\sum |S_{11}|_{\text{parallel}}}{\sum |S_{11}|_{\text{perpendicular}}}
\]  

were \(|S_{11}|\) is the modulus of the reflected wave.

This decrease is plotted on Fig. 9. During the first four days after slaughter, dielectric anisotropy drops from 5% to 1% (ratio drops from 1.05 to 1.01). This is in agreement with structural damages which occur in meat during ageing.

6 Conclusion

Microwave polarimetric method presented here allows following dielectric anisotropy decrease in bovine muscle during ageing. It also allows pointing out significant dielectric anisotropy change in fish muscle during a freezing – thawing cycle. An extension of this study at 16 and 24 GHz is under progress. Frequency increase should lead to a better sensitivity. It is also possible to extend this method to non contact measurements for an industrial point of view. Rectangular waveguide probe must be changed by an antenna which gives a linear polarization of the microwave. Preliminary studies have been done [15] on this topic, but non contacting measurement presents some problems and the ratio signal/noise must be improved (for instance thanks to beam focalisation) to measure weak variations of dielectric anisotropy.

References


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Effective Microwave Dielectric Properties of Food Materials Consisting of Large Particulates

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ABSTRACT. Dielectric properties of food and other materials are stated either for a compacted state (e.g. crushed peas) or for a bulk geometry consisting of such small particulates (e.g. wheat kernels) that mixture formulae can be used without additional difficulties. There are, however, many materials which naturally occur as defined, larger particulates. Uncrushed raw peas is used as a primary example of how the bulk as well as individual particulate heating properties in for example microwave heating processes can be analysed by a dual layer model of effective permittivity.

Keywords: effective permittivity, particulate, microwave, retro-modelling

1 Obtaining the "Effective" Permittivity

A multiple degenerate mode resonant circular cavity operating in the 2.2 to 2.6 GHz frequency interval has been used to obtain "fingerprints" (by $|S_{11}|$ measurements) of food samples with heights up to 50 mm in containers which can be inserted through a fixed "chimney" with 41 mm inner diameter [1]. The chimney also acts as a wavetrap so that it does not need to be closed in use. The cavity is fed by a vertical off-centre rod antenna. The result for three resonant frequencies is used to fine-tune the cavity dimensions by adapting a microwave modelling scenario so that the measured and modelled frequencies coincide. Since these three major resonances are $TM_{012}$ (at about 2470 MHz), $TM_{110}$ ($\approx 2410 \text{ MHz}$), and $TM_{111}$ ($\approx 2590 \text{ MHz}$), all important applicator dimensions can be brought into the scenario. In subsequent steps, the conductivities of the cavity and antenna, the dimensions and permittivities of the container support and sample container are obtained.

When there is a lossy sample, the three TM fields become degenerate and the behaviour of the reflection factor as a function of frequency becomes highly variable, depending on the dielectric properties and geometry of the sample. In effect, a kind of "reflection factor bridge circuit" is created and may provide a superior sensitivity. As an example, it becomes possible to detect a temperature change of 1°C or less in water.

The system has been used for measurements on uncrushed fresh peas, cooked rice, spaghetti, and some additional materials with still larger particulates. With a diameter of typically about 6 mm, raw peas provide a good example for further investigations. Literature data of crushed peas at $+20^\circ\text{C}$ and 2450 MHz are approximately 63.4–j15.3. With this high permittivity, an isolated sphere under 2450 MHz plane wave conditions becomes resonant with the lowest spherical $TE_{111}$ mode at about 14 mm diameter. Since the
coupling is reduced by more than the Rayleigh $\lambda^{-4}$ law, 6 mm diameter should be possible to accommodate in establishing an effective permittivity. However, the peas at the container wall are not cut, so the packing density will be less in an outer layer which is set equal to the pea radius, i.e. 3 mm. This thickness of a fictitious, homogeneous outer layer with a lower effective permittivity is used at all outer sides of the load. The final result becomes: inner $\varepsilon=(30 \pm 0.4) - j(4.8 \pm 0.4)$; outer: $\varepsilon=(1.8 \pm 0.1) - j(1.2 \pm 0.1)$. The data refer to 2450 MHz, but the dispersion effect due to the bandwidth which is a necessary condition is not considered to influence the data much.

It is somewhat astonishing that the effective permittivity of the inner region is almost half of that of crushed peas. Since the outer peas are more "isolated", the much lower value for this region is, however, expected. It is also interesting that $\tan\delta$ for the inner region is lower than for crushed peas, whereas $\tan\delta$ of the outer layer is much higher. Obviously, these phenomena are caused by diffraction and current concentration phenomena, which are accounted for by the effective permittivity computed here.

2 Investigation of the 2450 MHz Heating Pattern in Stacked Raw Peas

Due to further advances in the modelling software used [2], the field distributions in and around the individual peas can now be studied at a finer scale, and the reasons why the data above were obtained can be investigated. Comparisons with the resulting field distribution in a two-layered homogeneous load having the permittivities obtained by retromodelling show some interesting effects.

Figure 1 shows the pea (square) stack in perspective. It consists of $4 \times 4 \times 4$ spherical bodies, all with 6 mm diameter. There is a defined airgap of 0.1 mm between adjacent peas. Figure 2 shows the local heating pattern in a horizontal plane through centres of one of the middle stacks, under the condition of a modal impinging field from the left, such that a vertical $E_z$ component maximum occurs in the left-right centreline of the figure, when the field is undisturbed. In Figure 3, the only change is that the impinging modal field corresponds to a horizontal $H_x$ ($x =$ left-right) along the same centreline. Explanations for the differences in heating pattern and the very uneven heating of the individual particulates will be discussed, in terms of impedances and displacement currents.
3 Relevance Investigation of the Dual-layered Effective Permittivity Model

The method of permittivity determination involves a sample placement, more than $\lambda_0/4$ away from metal surfaces and other objects. In practical situations, the item stack may be contacting metal or a dielectric on at least one side. From a principle standpoint, the obtained effective permittivity simulates the behaviour of the load item stack as "seen" from the outside, i.e. provides relevant outer boundary conditions – but the internal fields may be compromised since the equivalent load has an outer homogeneous layer (with a thickness corresponding to the roughness of the load stack boundary) with a homogeneous region inside. What happens in terms of heating distribution and wave reflection can be conveniently investigated by comparative modelling of the load item stack using the "real" geometry in the figures, and a compact dual-layer object of the same overall size.

Colour illustrations are very suitable for illustrating the heating distributions and will be presented.

4 Conclusions

The quantitative results can be used for determining the advantages and limitations of the practical usefulness of the dual layer effective permittivity data. The effects of closeness between the particulates (i.e. degree of packing pressure, contacting area size and wetting, or air distance between particulates) is very important when they have a high permittivity, but the standard mixture formulas become increasingly relevant for smaller particulates and lower permittivity. This is related to the diffraction by the particulates (as opposed to quasistatic conditions. As mentioned above, about 14 mm size of high permittivity particulates at 2450 MHz will result in too pronounced deviations in behaviour as compared to any effective permittivity model.

The dual layer model provides representative results on overall reflection from and absorption in the bulk geometry of the load item stack, but the internal power distribution becomes different: the power density is smaller in the outer layer of the two-layered body than in its inner region. In addition, the wave energy penetrates about as deeply into regions away from the surface in the real load item stack as into the two-layered body. This fact is evidenced also by the penetration depth $d_p$ calculated in the usual fashion being about twice as large in the inner region of the two-layered model of uncrushed peas, as in a compacted pea load.

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RF Impedance Method for Nondestructive Moisture Content Determination in In-Shell Peanuts

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ABSTRACT: A method was developed earlier for estimating the moisture content (mc) in samples of wheat, corn and peanut kernels, nondestructively, by measuring their complex impedance values. In this method, capacitance, phase angle and dissipation factor were measured with an impedance analyzer at 1 and 5 MHz on a parallel-plate capacitor holding a few kernels of a particular commodity between the plates. These values were then used in an empirical equation and the moisture contents were calculated successfully within 1% of their air-oven values for about 85% of the kernel samples tested in the moisture range of 6 to 20% for wheat, corn and peanuts. However, it would be useful during drying and processing if the moisture content could be determined, for commodities such as peanuts, without shelling them. In this work, the feasibility of determining the moisture content of in-shell peanuts (pods) by similar impedance measurements was investigated. Measured values of capacitance, phase angle and dissipation factor at three frequencies were used in a modified prediction equation and the moisture contents were estimated within 1% of their air-oven values, for over 80% of the pod samples tested, in the moisture range of 7 to 20%. The method is rapid and nondestructive and may be used in the development of a commercial instrument.

Keywords: Moisture content, RF Impedance, In-shell, Peanuts

1 Introduction

In the southeastern United States, freshly dug peanuts, Arachis hypogaea L., may have moisture contents1 as high as 40%. They are allowed to dry on the vines till they reach an average moisture value of about 20% before being combined. Soon after they are combined, the peanuts have to be dried to reduce the mc to less than 10.5% to meet the grading standards [1]. They also can be stored at this mc level with adequate ventilation. Measuring and controlling moisture content is an important aspect in the harvesting, storage, marketing and processing of peanuts.

Peanuts are dried artificially in trailers that have a perforated floor and an air plenum till they dry to the required moisture level. At some drying facilities, more than 80 trailers at a time are in use. It takes considerable time and considerable cost is incurred in achieving the required reduction in the mc of the peanuts. During the drying process, it is required to measure the mc of the peanuts in each trailer at regular intervals to determine whether the desired mc level has been obtained and when the dryers can be turned off. Drying the peanuts for a longer time after the required mc level has been reached, increases drying costs and may lower the quality of the peanuts.

The mc measurement is presently done using electronic moisture meters that are calibrated against standard air-oven determinations. For this method a peanut sample of about 500 grams from a trailer is collected by an operator, the peanuts are shelled and cleaned, and the kernels are placed in a sample holder. The moisture meter measures the capacitance of the sample holder with the sample. This capacitance value is a function of the dielectric properties of the peanuts.

1 Moisture contents are expressed in % wet basis throughout this paper
peanuts in the bulk sample and is highly correlated with the moisture content of the sample. Thus, the meter gives an average value of the mc of all the peanuts in the bulk sample. This is repeated for samples from all other trailers. This method is time consuming. It would be very useful if the peanut kernel moisture could be estimated from physical measurements on the peanut pods without the need to shell them and clean them. Further, when large samples are used for measurement, the average value may be acceptable, but there could be some high moisture peanuts in the sample that are undetected. Pockets of high moisture peanuts can be conducive to the growth of microorganisms that may lead to the spoilage of the peanuts. Thus, it may be useful to measure the mc in smaller samples of pods than in large samples of peanut kernels. Experiments conducted earlier showed a good correlation between the peanut pod moisture and the peanut kernel moisture [2]. Thus, from knowledge of the peanut pod moisture, the mc of the kernels inside the pod may be estimated to an acceptable accuracy. This method is nondestructive and would result in large savings in time and labor during the drying process.

2 Materials and Methods

2.1 Basic Principles

The variation of dielectric constant with mc of shelled, yellow-dent field corn at different frequencies from 1 MHz to 11 GHz was earlier investigated [3]. The increase in dielectric constant with mc was more pronounced at the lower frequencies of 1 and 5 MHz than at higher frequencies (Fig. 1).

Fig. 1 Moisture dependence of the dielectric constant of shelled yellow-dent field corn at indicated frequencies [3]

It was found earlier that the variation of the dielectric constant at these frequencies was a useful parameter to estimate the mc of single kernels of corn [4]. Similar variation in dielectric constant with frequency was assumed for peanut kernels (shelled peanuts), and these variations were used to estimate their moisture content [5]. In the case of peanut pods (in-shell peanuts) also, similar variation in dielectric constant as with the peanut kernels at 1 and 5MHz is assumed.
The capacitance of a parallel-plate capacitor with plate area A and plate separation d, filled with a dielectric material, at a frequency \( f_1 \) is given by:

\[
C_1 = \frac{\varepsilon_{r1} \varepsilon_0 A}{d} \quad (1)
\]

At a frequency \( f_2 \), it is given by:

\[
C_2 = \frac{\varepsilon_{r2} \varepsilon_0 A}{d} \quad (2)
\]

\( \varepsilon_{r1} \) and \( \varepsilon_{r2} \) are the dielectric constants of the material between the plates at the two frequencies and \( \varepsilon_0 \) is the permittivity of free space \((8.854 \times 10^{-12} \text{ farad/m})\). Using these two equations, we can write the difference in the dielectric constants as:

\[
\varepsilon_{r1} - \varepsilon_{r2} = \frac{(C_1 - C_2) d}{(\varepsilon_0 A)} \quad (3)
\]

It was earlier found that \((C_1-C_2)\) was a good estimate of the moisture content but was highly influenced by the size and shape of the peanut kernels [5]. Two other electrical parameters, dissipation factor, \( D \) and phase angle, \( \theta \) were also measured at the two frequencies. The measurements were made on single kernels (shelled peanuts) with the kernel held between and in contact with the two parallel-plates. The differences in the values of these two parameters at the two frequencies, \((D_1-D_2)\) and \((\theta_1-\theta_2)\) were incorporated into the following empirical equation along with \((C_1-C_2)\), and from this equation the moisture content \( M \) in a single peanut kernel was calculated as

\[
M = A_0 + A_1 (C_1 - C_2) + A_2 (C_1 - C_2)^2
+ A_3 \left[ \frac{(\theta_1 - \theta_2)}{(C_1 - C_2)^2 + (D_1 - D_2)^2} - (C_1 - C_2)(D_1 - D_2) \right]
\quad (4)
\]

where \( A_0, \ldots, A_3 \) are calibration constants.

Measurements made on peanut pods (in-shell peanuts) are influenced by the presence of the shell around the kernels, and the mc determinations calculated with equation (4) did not compare well with the air-oven determinations. However with a slightly modified equation (5), shown below, the mc in single peanut pods could be obtained to an accuracy of within 1% of the air-oven values for over 85% of the single peanut pods tested in the moisture range 5 to 20% [6].

\[
M = A_0 + A_1 (C_1 - C_2) + A_2 (C_1 - C_2)^2
+ A_3 \left[ \frac{(\theta_1 - \theta_2)}{(C_1 - C_2)^2 + 2(D_1 - D_2)^2} - (C_1 - C_2)(D_1 - D_2) \right]
\quad (5)
\]

where \( A_0, \ldots, A_3 \) are calibration constants.

When several peanut pods of different sizes were placed between the plates, each of them may not be in contact with both the plates, resulting in air gaps between some of the pods and the plates. These contributed to inaccuracies in the moisture values determined by using the values of \( C, \theta \) and \( D \) at the two frequencies in equation (5) for small samples of peanut pods. The mc values calculated with equation (5) did not compare well with those obtained by the standard air-oven method. To improve the accuracy of mc prediction in small peanut-pod
samples, measurements of C, θ and D were made at two more frequencies, 9 and 13 MHz and the differences in the values of C and θ at 1, 5 and 9 MHz were used in the following empirical equation to obtain the mc of the small samples of peanut pods:

\[ M = A_0 + A_1 \Delta C_1 + A_2 \Delta C_2 + A_3 \Delta \theta_1 + A_4 \Delta \theta_2 + A_5 (\Delta C_2)^2 \]  

(6)

where \( \Delta C_1 \) and \( \Delta \theta_1 \) are the differences in the values of C and θ at 1 and 5 MHz, \( \Delta C_2 \) and \( \Delta \theta_2 \) are the differences at 1 and 9 MHz. \( A_0, \ldots, A_5 \) are calibration constants for this equation. The contribution of the values of C and θ at 14 MHz for mc calculation was found to be minimal and they were not used. The values of the constants in equation (6) were obtained by making measurements on peanut pods of known moisture contents, as determined by the air-oven method, at five different moisture levels in the range from 6 to 25% and by applying a least-squares computation.

2.2 Equipment

A Hewlett-Packard 4192A LF Impedance Analyzer equipped with a 16096A Test Fixture was used to make the measurements on the peanut pods held between the parallel plates of a specially constructed electrode assembly [4] as shown in Fig. 2. The electrode assembly consists of two circular, 75-mm-diameter, brass plates mounted on insulating rods. The upper plate can be moved up and down under a light spring pressure, which facilitates the sample insertion and removal. The peanuts were placed on the lower plate and held in position by the upper electrode,

![Measuring system](image)

Fig. 2 Measuring system: 1. Balance. 2. Computer. 3. Parallel plates with peanuts. 4. Analyzer

the analyzer measured the capacitance, dissipation factor and phase angle of this electrode system at 1, 5 and 9 MHz, and a computer controlled and collected the data from the analyzer. A 3-mm-thick plastic ring with an inner diameter of 72 mm, fitted to the lower plate, limited the pods from extending to the edges of the plates. A Mettler AE 163 electronic balance was also interfaced to this computer to record the wet and dry weights of the peanut pod samples to obtain their air-oven moisture values. Using these measurements and equation (6), the computer was programmed to calculate the moisture content in each peanut pod sample.

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2 Mention of company or trade names is for the purpose of description only and does not imply endorsement by the U.S. Department of Agriculture.
2.3 Peanut Samples

Peanuts of the Runner variety, harvested, dried and stored in cold storage at the National Peanut Research Laboratory during the year 2002, were used for these studies. The initial moisture content (mc) of this lot was about 5% as measured with a Dickey-john Grain Analysis Computer. From these, five sub-lots, called the calibration lots, were placed in quart jars. Leaving one jar of peanuts at the original moisture level, appropriate quantities of water were added to samples in the other jars to raise their moisture levels to obtain five sub-lots with moisture levels ranging from 5% to 25%. Similarly from the original lot another four sub-lots called the validation lots were separated and placed in four jars. The peanuts in these jars were conditioned to different moisture levels ranging from 6 to 22% by adding appropriate amounts of water to samples in each jar. All the jars were sealed and held at 4 °C to equilibrate.

2.4 Procedures

A small sample, consisting of seven or eight pods, was taken out of one of the calibration lot jars and placed in a numbered metal container. The container was placed on the balance and its weight was recorded. Raising the upper plate electrode, the sample was placed on the lower plate to occupy the space between the plates within the plastic ring, and the upper plate was released to rest on the pods. Impedance measurements were taken for this sample at 1, 5, 9 and 13 MHz, and the sample was transferred back into its container. This procedure was repeated for nine more samples from this jar and for ten similar samples (7 or 8 pods) from the other four sub-lots from the calibration group. These procedures were repeated for measurements on the four sub-lots of the validation lots.

After the measurements were completed and each sample was placed in its container, the moisture content of each sample was determined by the standard air-oven method [7]. The containers were placed in a hot-air oven at 130 °C for 6 hours. At the end of the heating period the containers were removed and weighed to determine the dry weight of the samples. Moisture content (wet weight basis) is determined for each sample as the percentage ratio of the weight loss to the original wet weight of the sample. Using the mc values and the measured impedance values of the calibration group, the calibration constants of equation (6) were determined. By using these constants and the measured impedance values of the samples in the validation group, the mc of each pod sample was calculated and compared with the mc values obtained by the air-oven method.

3 Results

From the measured values of capacitance, phase angle, and the air-oven mc values of the five calibration lots, the values of the constants in equation (6) determined by the least-squares computation were:

\[
A_0 = 11.989, \quad A_1 = 4.060, \quad A_2 = 8.357, \quad A_3 = -11.104, \quad A_4 = 9.656, \quad A_5 = -2.439
\]

By using the values of these constants in equation (6), the mc of each pod sample in the five calibration lots was calculated, averaged over the ten samples in each moisture level, and the results are shown in Table I along with the mc values obtained by the air-oven method. The standard deviations for both methods are also shown. The coefficient of determination was 0.96.
Table 1 Comparison of oven and calculated mc values for 5 calibration lots (Average of 10 samples)

<table>
<thead>
<tr>
<th>Nominal % mc</th>
<th>Moisture content (%)</th>
<th>Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Oven</td>
<td>From Eq.(6)</td>
</tr>
<tr>
<td>6</td>
<td>6.87</td>
<td>7.47</td>
</tr>
<tr>
<td>10</td>
<td>10.18</td>
<td>9.85</td>
</tr>
<tr>
<td>14</td>
<td>14.29</td>
<td>14.38</td>
</tr>
<tr>
<td>18</td>
<td>18.95</td>
<td>19.80</td>
</tr>
<tr>
<td>23</td>
<td>22.70</td>
<td>21.78</td>
</tr>
</tbody>
</table>

The mc values of each of the pod samples in the four validation lots were similarly calculated and the results are shown in Table 2.

Table 2 Comparison of oven and calculated mc values for 4 validation lots (Average of 10 samples)

<table>
<thead>
<tr>
<th>Nominal % mc</th>
<th>Moisture content (%)</th>
<th>Predictability#</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Oven</td>
<td>From Eq.(6)</td>
</tr>
<tr>
<td>8</td>
<td>8.44</td>
<td>8.24</td>
</tr>
<tr>
<td>10</td>
<td>9.29</td>
<td>9.10</td>
</tr>
<tr>
<td>14</td>
<td>12.97</td>
<td>12.12</td>
</tr>
<tr>
<td>21</td>
<td>21.16</td>
<td>20.98</td>
</tr>
</tbody>
</table>

# Predictability is the percentage of pod samples for which mc was predicted within 1% of the air-oven values.

The predictability, as shown in the last column, indicates how well the moisture content determined from impedance measurements and calculated with equation (6) for each sample compared with its corresponding air-oven value. It is the percentage of pod samples for which mc was predicted within 1% of the air-oven value in each moisture level. The predictability was at least 70% at any level and averaged 85% over all the moisture levels in the validation group.

Fig. 3 Comparison of air-oven and impedance meter values of mc in peanut pod samples
A bar graph comparing the mc values determined by the oven and impedance methods at the four moisture levels in the validation group is shown in Fig. 3.
4 Conclusions

The results show that the RF Impedance method which was earlier applied to corn and peanut kernels to determine their moisture content is applicable to in-shell peanuts. Since no shelling and cleaning of peanuts is needed for the impedance measurements, it could save considerable amount of time and labor. Presently there do not seem to be any commercial instruments that measure moisture content in peanut pods nondestructively. Using this method, mc of small samples of peanuts can be estimated, which may be useful in detecting pockets of high-moisture peanuts in bulk samples during the drying process or in storage. A practical instrument developed along these lines may be useful in marketing, processing and storage of peanuts both shelled and in shell.

References


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ABSTRACT. This paper describes the results of elaboration of microwave moisture meter for continuous control of moisture grain, sugar and dry milk in technological processes of their production. Dielectric properties of these materials and technological requirements determine the choice of investigation methods.

Keywords: moisture measurement, microwave, grain, sugar, dry milk

1 Introduction

Dielectric properties of most agricultural products in the moisture function $\varepsilon^*(W)$ can be described precisely enough on the basis of the linear model, which suggests that the real $\varepsilon'$ - and the imaginary $\varepsilon''$ - parts of complex dielectric permittivity $\varepsilon^*$ (as well as $\alpha = (\pi / \lambda) (\varepsilon'' / \sqrt{\varepsilon'})$ and $\beta = (2\pi / \lambda) \sqrt{\varepsilon'}$, where $\lambda$ is a wavelength) depend not only on the moisture $W$, but also on the density of the considered stuff $\rho$. Therefore, here we shall consider two alternative methods of the microwave moisture meter application.

2 Methods and Results

2.1 Flour-Milling Industry

The process of grain dehydration allows a rather easy stabilizing of the stuff flow controlled. Here, it is enough to control only one dimension, e.g. the electromagnetic wave attenuation coefficient $\alpha$ (when the temperature is corrected automatically). Thus, the relation between the attenuation $N$ in the sample with the thickness $d$ and the moisture will be as follows:

$$N = 8,686 \cdot \alpha_{H} \cdot W \cdot \rho \cdot d$$

(1)

The factor of attenuation $\alpha_{H}$ within the framework of linear model will be determined by expression:

$$\alpha_{H} = \alpha_{HC} \cdot q_{HC} + \alpha_{HP} \cdot q_{HP} + \alpha_{H0} \cdot q_{H0}$$

(2)

where: $\alpha_{HC}, \alpha_{HP}, \alpha_{H0}$ - factors of attenuation of an electromagnetic wave in water connected, polysorption and free, respectively;

$q_{HC}, q_{HP}, q_{H0}$ - volumetric concentration of water connected, polysorption and free, respectively;

From where we get:
\[
\alpha_H = \Phi \frac{\rho_d}{\rho_H} \left[ \frac{W}{1-W} \alpha_{\text{ss}} + \frac{\mu S_S}{N_A \sigma} a_{\text{max}} f_1(w, s_S, a_m) (\alpha_{\text{ss}} - \alpha_{\text{so}}) + \frac{W_{cr}}{1-W_{cr}} (\alpha_{\text{ic}} - \alpha_{\text{so}}) \right]
\]  

(3)

where:

- \(\Phi\) - factor of filling;
- \(\rho_d, \rho_H\) - density of a dry material and water;
- \(\mu\) - gramme molecular weight of water;
- \(S_S\) - a specific surface of a grain (wheat);
- \(N_A = 6,022 \cdot 10^{23}\);
- \(\sigma = 3 \cdot 10^{-8}\) - landing place of one molecule of water on a surface of a firm phase;
- \(a_{\text{max}} = 100\) - a maximum quantity of monolayers of water occupied with a moisture polysorption in the given material;
- \(W_{cr} = 10\%\) - the critical moisture appropriate to transition from connected water in a grain to water polysorption.

\(f_1(w, s_S, a_m)\) - the function describing ratio between free and connected water.

Function \(f_1(w, s_S, a_m)\) according to model of Ivens-Busker [1] in which it is supposed, that speed of change of the given form of moisture in the process of moistening is directly proportional to a part sorption volume free from this moisture. Hence it is possible to write down:

\[
\frac{dq_{HP}}{dQ} = 1 - \frac{q_{HP}}{q_{HPmax}}
\]

(4)

where:

- \(q_{HP}\) - volumetric concentration of a moisture polysorption;
- \(Q = \frac{P_H}{\rho_H \cdot V}\) - relative volumetric moisture of a material, i.e. relative quantity of water \((P_H)\) in unit of volume \((V = q_{HC} + q_{HP} + q_{H0})\);

Integrating (3), with the account that at \(Q = Q_{HP}, q_{HP} = 0\), we receive

\[
q_{HP} = q_{HPmax} \cdot \left( 1 - e^{\frac{Q}{q_{HPmax}}} \right)
\]

(5)

In this kind expression (4) is inconvenient to use since all volumetric concentration of water included in it depends on the factor of filling \(\Phi\). Knowing the specific surface of the material \(S_S\) and the maximum quantity of monolayers of water polysorption \(a_{\text{max}}\), \(q_{HPmax}\) it is possible to find as follows:

\[
q_{HPmax} = \frac{\mu \cdot \rho_d \cdot S_S}{N_A \cdot \rho_H \cdot \sigma} \cdot a_{\text{max}} \cdot \Phi, \text{ then}
\]

\[
q_{HPmax} = \Phi \cdot \frac{\rho_d \cdot \mu \cdot S_S}{\rho_H \cdot N_A \cdot \sigma} \cdot a_{\text{max}} \left[ 1 - \exp \left( \frac{(W_{cr} - W) \cdot \sigma \cdot N_A}{(1-W_{cr}) \cdot (1-W) \cdot \mu \cdot S_S \cdot a_{\text{max}}} \right) \right]
\]

(6)
Hence function \( f_1(w, s_S, a_{\text{max}}) \) will be determined by expression

\[
f_1(w, s_S, a_{\text{max}}) = 1 - \exp \left( \frac{(W_{cr} - W) \cdot \sigma \cdot N_A}{(1 - W_{cr}) \cdot (1 - W) \cdot \mu \cdot S_S \cdot a_{\text{max}}} \right)
\]

(7)

The calculation of dielectric characteristics of wheat model can be made, having accepted the following values:

\[
\begin{align*}
\rho_0 &= 1.2g/cm^3; \quad S_S = 220m^2/g; \quad a_{\text{max}} = 100; \quad W_{cr} = 0.1; \\
\epsilon_{H0}\epsilon &= \epsilon_{H1}\epsilon = \epsilon_{HC}\epsilon = 5.1; \quad \epsilon_{HCS} = 60; \quad \epsilon_{HPS} = \epsilon_{HOS} = 80.4; \quad t = 20^\circ C; \\
\lambda_{H0m} &= 1.79cm; \quad \lambda_{H1m} = 377cm; \quad \lambda_{Hpm} = 9.5cm; \\
\Delta H_{HC} &= 15Kcal / mol; \quad \Delta H_{HPS} = 1Kcal / mol; \quad \Delta H_{HOS} = 4.6Kcal / mol;
\end{align*}
\]

The results of calculation, in view of that the factor of filling \( \Phi \) is function of moisture (table 1) content lead to the dependence different from graduation for Microradar 113 no more than on 10 % (Fig. 1.)

Table 1 Dependence of filling coefficient \( \Phi \) on wheat moisture

<table>
<thead>
<tr>
<th>W, %</th>
<th>8</th>
<th>12</th>
<th>16</th>
<th>20</th>
<th>24</th>
<th>28</th>
<th>32</th>
<th>36</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \Phi )</td>
<td>0.60</td>
<td>0.54</td>
<td>0.50</td>
<td>0.44</td>
<td>0.43</td>
<td>0.41</td>
<td>0.39</td>
<td>0.37</td>
</tr>
</tbody>
</table>

However, since the flour-milling industry sticks to additional damping of grain from 12-14% to 15.5-16%, it is essential that the influence of the free and bound water should be taken into consideration, which means switching to another calculation algorithm. To serve that purpose, a new modification of moisture indicator has been developed. It is equipped with two microwave sensors (placed at the entrance and output of the damping machine) and a computing unit, which functions along two patterns.

The second algorithm is also based on the equation (1), but \( \alpha_H = \alpha(W) \) is in the settlement way under known dielectric characteristics of free water.

\[
N = N_0 + \alpha(W) \cdot k \cdot (W - W_0);
\]

(8)

The results of calculation on the accepted model of wheat grain for temperature \( t=20^\circ C \) and wavelength \( \lambda = 3.2cm \) are given in Figure 1.

The researches of experimental results, well in accord with the theory, and the record of industrial application of the automatic additional damping systems based on Microradar 113-2 show the possibility of high-precision maintenance of the finite moisture of grain (less than \( \pm0.2\% \)) [2].

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Fig. 1 Calculated dependence \( N(W) \) for microwave sensor \( \#1 \) (---) and \( \#2 \) (---) in function \( W \), • - experiment for wheat grain of natural moisture

Fig. 2 Scheme of main units installation on damping machine
2.2 Sugar and Milk Solids Production

The dielectric properties of the original components at sugar and milk solids production require a more sensitive method as well as compulsory control of two dimensions related to $\varepsilon'$ and $\varepsilon''$ of these stuffs. Therefore, the resonator two-parameter method and the Microradar 114 based on it have been applied [2].

The control of moisture of a material with small and midget moisture content by resonator methods is possible when using two circuits of inclusion of the resonator - on passage and reflection. In the first case change of transitive easing $\Delta N$, change of good quality $\Delta Q$ or displacement of its resonant frequency $\Delta f$ is supervised. And change of good quality loaded $Q_L$ and non-loaded $Q_0$ resonator is connected with $\varepsilon''(W, \Phi, t)$ moisture containing material and leaving of resonant frequency $(f - f_0)$ from it $\varepsilon'(W, \Phi, t)$. Since value of complex dielectric permittivity $\varepsilon^*$ depends not only on moisture $W$ and temperature $t$, but also on the factor of filling $\Phi$ at materials with the big fluctuations of density, it is necessary to supervise two parameters (at automatic temperature indemnification), it: $\varepsilon'(W, \Phi, t)$ and $\varepsilon''(W, \Phi, t)$, or a pair $\alpha(W)$ and $\beta(W)$ where $\alpha(W)$ is found from (3) such us:

$$\alpha = \alpha_{HC} \frac{W}{1-W} \frac{\rho_d}{\rho_u}$$

$$\beta = \Phi \left[ \frac{W}{1-W} \rho_u (\beta_u - \beta_0) + (\beta_c - \beta_0) \right]$$

This system is solved be relative $W$ with exception $\Phi$ i.e.:

$$W = \left\{ \frac{1 - \rho_d}{\rho_H} \left[ \frac{\beta_H - \beta_0}{\beta_c - \beta_0} \frac{\alpha_H \left( \beta - \beta_0 \right)}{\alpha \left( \beta - \beta_0 \right)} \right] \right\}^{-1}$$

The other way exception of factor of filling $\Phi$ is an introduction A-parameter:

$$A(W) = \frac{\alpha}{\beta - \beta_0} = \frac{\alpha_{HC}}{[\beta_c - \beta_0] + \frac{\rho_H}{\rho_d} (\beta_c - \beta_0) (1 - \frac{W}{W})}$$

For practically any range of moisture content the A-parameter may be approximately described by the expression

$$A(W) = K \frac{\varepsilon''}{\varepsilon' - 1}$$

That is similar

$$R(\psi) = \frac{\varepsilon''}{\varepsilon'_0 - 1} = \frac{1}{2} \frac{1}{\frac{f_0}{f_1}} \frac{Q_1 - Q_0}{f_0 - f_1}$$
The results of calculation on the basis of linear model and research on modelling materials give good correlation between moisture and components $\varepsilon'$ and $\varepsilon''$ to complex dielectric permittivity $\varepsilon^*$ (Fig. 3).

![Fig. 3 The real $\varepsilon'$ and imaginary $\varepsilon''$ parts of the dielectric constant for octanol at 25°C as a function of frequency (GHz)](image)

The resonator microwave - moisturemeter Microradar 114 is applied for the continuous control of moisture content in sugar production. Its installation in various points of a technological line will allow to automate the process of boiling off utfel at a stage of crystallization in vacuum - devices. Installation of a moisturemeter after centrifuging allows to operate work and to submit on drying sugar of known moisture. Application of a moisturemeter on an output of drying installation enables automation of this technological process. Figure 4 shows the exterior of one of updatings the Microradar 114 and variants of installation of the device in various points of technological process. Figure 5 shows dependence of a moisturemeter, an absolute error of measurement of ready sugar no more $\pm 0.03\%$ (in a range of change of moisture content 0-3 %) calibration.

![Fig. 4 Exterior (a) and variant of installation (b) of MR-114 in different of technological process](image)
Production of dry milk now is based on drying of initial raw material in spray dryers, and moisture in these processes is the factor determining quality of a ready-made product.

As well as in sugar production technological process of reception of dry milk assumes the control of moisture in several points:
- On an output of a product from convectional the drying unit;
- After shaking sieve before submission of a ready-made product to packaging.

Application Microradar 114 for automation of technological process of drying milk allows to operate work of convectional dryers and to support moisture in necessary limits. Overdrying a product conducts to disintegration of fibers and deterioration of dry milk at the significant overexpenditure of thermal energy, underdrying also influences both on quality and decrease of a period of storage.

Figure 6 shows the appearance of a sensor variant (6a) and the technological circuit of process with variants of installation of the device (6b).

The device provides:
- 9 different graduations;
- averaging of result;
- connection to the computer in standard RS 485;
- opportunity of diagnostics;
- standard current an output;

Range of measurement of moisture of dry milk is 2-7 %. The basic absolute error of measurement in this range ≤0.3 %. Calibration dependence for dry milk is given on Fig. 6c.
3 Conclusion

The application of two-parameter method’s in the Microwave Aquametry of loose agricultural materials allows essentially to increase accuracy of measurement of moisture due to the account of influence of density (factor of filling), the specified methods, however, are effective at rather small fluctuations of density (within the limits of 2.5 - 3 times) and rather narrow range of change moisture content. The further increase in accuracy of measurement of moisture is possible due to a combination of Microwave methods with others physical, ones, for example Microwave - Acoustic.

Fig. 6 Variants of installation of MR – 114 in different points of technological process of dry milk production and its calibration
References


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High Resolution, Non-destructive and In-process Time Domain Aquametry for FMCG and other Products Using Microstrip sensors

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ABSTRACT. This paper reports a novel non-destructive, instantaneous microwave aquametry technique, implemented in time domain, useful for applications in the FMCG as well as space and defense industries. Resolution from 0.004% to 0.05% has been measured for moisture levels, ranging from 0.1% to 80% respectively, in substances like seeds and soil, soap, tissue paper and milk powder. Similar resolution has been obtained at moisture levels from 0.2% to 2% in ammonium per-chlorate, an oxidizer for rocket propellants. The numerical software-assisted technique has potential for biomedical applications in drug discovery, tomography, tumor and cancer detection.

Keywords: tdr, moisture, fmcg, ndt

1 Introduction

The in process, on assembly line, measurement of moisture content of materials – powders, granules, chunks, mixtures, gels, foams, syrups and squashes – is critical for the Fast Moving Consumer Goods (FMCG) and processed food industries. Levels of moisture influence packaging, storage and shelf life. Quality and, therefore, profits are determined by moisture content. Currently for aquametry, these industries depend on moisture balances, Karl Fisher titration and infrared moisture meters. The microwave free space absorption technique and microstrip attenuation techniques have been introduced in these consumer goods industry. But they are yet to receive the wide acceptance given to moisture balances and infrared moisture meters. The microwave free space absorption technique and microstrip attenuation techniques have been introduced in these consumer goods industry. But they are yet to receive the wide acceptance given to moisture balances and infrared moisture meters. The TDR technique [2, 3] has been limited to soil moisture measurements, using invasive fork type probe. In many areas of space technology critical moisture measurement is required at very low moisture levels. The Karl Fisher titration is the only available option today. An open-ended feasibility study was performed which has resulted in promising results.

This paper reports a novel technique of on-line, time domain, non-destructive microwave aquametry [1] for the FMCG industry. Using the fast computing abilities of desktop computers, and by using numerical analysis [4], an alternative technique, to purely experimental calibration methods, has been proposed. The technique may be extended to the broadband measurement of permittivity, in a non-destructive manner. The material under test is kept as an overlay on the microstrip probe. The time delay, characteristic impedance and reflection coefficient of microstrip are measured. The change in the effective permittivity of the microstrip probe is given by

$$t_s^2 / t_o^2 = \varepsilon_{\text{effs}} / \varepsilon_{\text{effo}}$$  \hspace{1cm} (1)

Where suffixes “o” and “s” indicate time delay ($t$) and effective permittivity ($\varepsilon_{\text{eff}}$) without and with the sample as an overlay on the microstrip.

$$\Delta t_o = f(\varepsilon_{rs})$$  \hspace{1cm} (2)

where $\varepsilon_{rs}$ is the relative permittivity of the overlay sample. $\varepsilon_{rs'}$ of the material under test is determined with the help of spectral domain analysis [4].
2 Experimental Details and Results

2.1 Experimental Set-up

The experimental set-up was basically a simple microstrip connected to the HP 54750 in a reflection mode, as shown in Fig. 1. A step input was fed to the microstrip, which was open ended. The material under test was kept in the plastic pouches on the probe to avoid contamination except the wet tissue paper. The soap data was collected on the probe with a plastic protective sheet as a protective layer. Ammonium per chlorate and silica rubber was characterized for low moisture content according to the requirement of the material measurement.

![Fig. 1 Experimental set-up](image)

2.2 Measurement Results

Four types of soap samples including soap noodles and cakes; propellant grade amorphous and crystalline ammonium per-chlorate samples, saturated tissue paper, rice grains and silica rubber used for rocket motor lining were characterized non-destructively using microwave integrated circuit probes and HP (Agilent) TDR 54750 with 18GHz bandwidth. Figures 2-6 show TDR responses of probes with and without sample overlays. Figures 2-3 show characterization of soap noodles and saturated tissue paper respectively. The soap sample was few centimeters in height whereas tissue paper was only 2mm, both were covering the complete probe. The data shown in Figures 4 is obtained by soaking the silica rubber in water for 2-6 hours. All decimal points could not be indicated on the time axis. The numbers correspond to the actual differential delay measured using 50% pulse height as a reference. Ammonium per chloride measurement was performed (as shown in Fig. 5-6) using the same sample in a plastic pouch. The samples were not removed from the probe but water drops were added with a 2ml syringe. This helped significantly as the sample obtained was very small in quantity and placement repeatability error was eliminated. The differential TDR response of various materials presented a high resolution. Fig. 7 illustrates various resolutions obtained in the moisture characterization with high-resolution sampling oscilloscope. Characterization of Ammonium per chloride with very low moisture content indicated a third decimal place resolution not reported for on-line moisture measurement. The dry and wet soap noodles at 12% and 26% moisture levels showed 568 and 1127 digitized units.
respectively, giving **resolution of 0.02% and 0.023% moisture** (Table 1). Luxury and medicated soap cakes indicate differential delays of 376 and 481 time units respectively for 7.5% and 7.67% moisture. The percentage resolution is calculated on the basis of number of data points, differential delay and the gravimetric moisture content. The lower moisture levels are observed to give better resolution.

![Fig. 2 TDR response of reference probe and soap noodles](image1)

![Fig. 3 TDR responses of reference probe and saturated tissue paper](image2)
Table 1  Differential delay and resolution achieved for soap samples

<table>
<thead>
<tr>
<th>Type of Sample</th>
<th>$\Delta t$ ps Due to sample</th>
<th>% Moisture (Oven method)</th>
<th>%Resolution Present technique</th>
</tr>
</thead>
<tbody>
<tr>
<td>Open probe</td>
<td>0.0</td>
<td>0.00</td>
<td>----</td>
</tr>
<tr>
<td>Standard calibration</td>
<td></td>
<td>80.2</td>
<td>----</td>
</tr>
<tr>
<td>sheets (4 nos.)</td>
<td></td>
<td></td>
<td>----</td>
</tr>
<tr>
<td>Dry noodles</td>
<td>56.8</td>
<td>12</td>
<td>0.02</td>
</tr>
<tr>
<td>Wet noodles</td>
<td>112.7</td>
<td>26</td>
<td>0.023</td>
</tr>
<tr>
<td>Pink Lux cake</td>
<td>32.6</td>
<td>7.5</td>
<td>0.023</td>
</tr>
<tr>
<td>Life buoy gold</td>
<td>48.1</td>
<td>7.67</td>
<td>0.016</td>
</tr>
<tr>
<td>Saturated Tissue paper</td>
<td>177.9</td>
<td>80.0</td>
<td>0.05</td>
</tr>
</tbody>
</table>

Fig. 4 TDR step response for silica rubber when soaked in water for 0-6 hour
Fig. 5 TDR step response for ammonium per chlorate when added water drops (crystalline sample)

Fig. 6 TDR step response for ammonium per chlorate when added water drops (amorphous sample)
Table 2  Differential delays for ammonium per-chlorate overlay with added water

<table>
<thead>
<tr>
<th>Water added in ml</th>
<th>Moisture percentage</th>
<th>Delay in ps</th>
<th>Differential delay in ps</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td>0.02</td>
<td>459.97</td>
<td>---</td>
</tr>
<tr>
<td>0.1</td>
<td>0.10</td>
<td>486.36</td>
<td>26.39</td>
</tr>
<tr>
<td>0.2</td>
<td>0.18</td>
<td>500.00</td>
<td>40.03</td>
</tr>
<tr>
<td>0.3</td>
<td>0.25</td>
<td>502.93</td>
<td>42.96</td>
</tr>
</tbody>
</table>

Table 3  Differential delays for ammonium per-chlorate overlay with added water

<table>
<thead>
<tr>
<th>Water added in ml</th>
<th>Moisture percentage</th>
<th>Delay in ps</th>
<th>Differential delay in ps</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td>0.02</td>
<td>486.33</td>
<td>---</td>
</tr>
<tr>
<td>0.1</td>
<td>0.19</td>
<td>521.52</td>
<td>35.19</td>
</tr>
<tr>
<td>0.2</td>
<td>0.35</td>
<td>527.34</td>
<td>41.01</td>
</tr>
<tr>
<td>0.3</td>
<td>0.51</td>
<td>527.5</td>
<td>41.17</td>
</tr>
</tbody>
</table>

Fig. 7  Resolution [%] achieved for various moisture levels in different materials
3 Conclusions

The moisture resolution of the second decimal place is being reported for the first time in online measurements. However, the resolution of the second decimal place is not required in the consumer industry. It may prove to be useful in the critical applications such detection of absorption of moisture in circuit board materials in a nondestructive manner. The probe-to-probe and sample-to-sample repeatability studies indicate high confidence levels for ammonium per-chlorate as shown in Tables 2&3. The technique can be used with relatively low cost instrumentation for the consumer industry and with the reported degree of accuracy for the critical applications.

Acknowledgement

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1. United State Patent nos. 6,204,670 and 6,407,555.

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Frequency and Temperature Dependence of the Permittivity of Fresh Fruits and Vegetables

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Richard B. Russell Agricultural Research Center, Athens, Georgia, USA

ABSTRACT. Dielectric properties over the frequency range from 10 MHz to 1.8 GHz and over the temperature range from 5 to 65 °C were measured for tissue samples cut from nine fresh fruits and vegetables. Dielectric constant and loss-factor data were obtained for apple, avocado, banana, cantaloupe, carrot, cucumber, grape, orange, and potato, showing dielectric constants ranging from values of several hundred at 10 MHz to less than 100 at 1.8 GHz and loss factors on the order of one thousand at 10 MHz to less than 20 at 1.8 GHz. The dielectric loss factor increased consistently with increasing temperature at frequencies below 1 GHz. The dielectric constant increased with temperature at lower frequencies, but it decreased with temperature at the higher frequencies. This reversal of the sign of the temperature coefficient occurred at some point in the frequency range between 20 and 120 MHz where the temperature dependence of the dielectric constant was zero. At frequencies below this point, ionic conduction dominates the dielectric behavior, but above that point dipolar relaxation appears to control the behavior. Multiple linear regression provided equations for calculation of the loss factor in the frequency range from 10 to 300 MHz at temperatures from 5 to 65 °C. The data provide new information useful in understanding dielectric heating behavior and evaluating dielectric properties of such agricultural products for quality sensing applications.

Keywords: dielectric properties, permittivity, fruit, vegetables

1 Introduction

In this paper, the term "permittivity" implies the relative complex permittivity, i.e., the permittivity of a material relative to free space, often called the complex dielectric constant, which is expressed as \( \varepsilon = \varepsilon' - j \varepsilon'' \), where \( \varepsilon' \) is the dielectric constant and \( \varepsilon'' \) is the dielectric loss factor. The dielectric constant is associated with the capability for energy storage in the electric field in the material, and the loss factor is associated with energy dissipation in the material or the conversion from electric energy to heat energy. Here, all loss mechanisms, both those due to dipole relaxation and ionic conduction, are included in the dielectric loss factor \( \varepsilon'' \).

With regard to agricultural products and materials, interest in dielectric properties has been associated with only a few applications [1]. These have included the sensing of moisture content in grain [2]; radio-frequency and microwave dielectric heating for pest control [3-5], seed treatment [6], product conditioning [7-10], remote sensing of crop condition [11], and potential uses for quality measurements other than moisture content [12,13].

In connection with quality sensing in fruits and vegetables, the dielectric properties of mature-green and full-ripe peaches at 2.45 GHz were examined to see whether these properties might be useful in distinguishing degree of maturity [12]. The same kinds of measurements were taken on normal sweet potatoes and those that had a hard-core condition induced by chilling injury in storage [12]. Permittivity measurements at 2.45 GHz did not offer promise for detecting either of these quality factors. Following permittivity characterization measurements for twenty-three kinds of common fresh fruits and vegetables over the frequency range from 200 MHz to 20 GHz at 23 °C [14,15], similar measurements were taken over a narrow range
of peach maturity, and evidence for possible distinction of maturity degree was obtained [13]. A permittivity-based maturity index was suggested, based on differences in both components of the permittivity, the dielectric constants at the low end of the frequency range and the loss factors at 10 GHz near the higher end of this frequency range. More research and developmental work was recommended for determining the potential for practical use of the technique, including measurements at frequencies lower than 200 MHz, since the curves for the dielectric constants of the two different maturities appeared to be diverging as they approached the lower end of the frequency range.

Measurements obtained in a study to explore the frequency and temperature dependence of the dielectric properties of a few kinds of fruits and vegetables were recently reported for a temperature range from 5 to 95 °C [16]. The data obtained in that study have been further analyzed, and new relationships and graphical presentations providing reference data for the higher frequencies, which were not legible in the graphs in the cited article, are presented in this paper. The new data are of interest in microwave heating and processing of such materials and are also helpful in understanding the dielectric behavior of these food materials.

2 Materials and Methods

2.1 Fruits and Vegetables

A few samples of different fresh fruits and vegetables were obtained at local grocery stores for these measurements to study the variation of permittivity with temperature and frequency in the range from 10 MHz to 1.8 GHz. They included the 'Red Delicious' apple, *Malus domestica* Borkh.; Navel orange, *Citrus aurantium* subsp. bergamia; 'Thompson Seedless' grape, *Vitis amurensis* Rupr.; 'Cavendish' banana, *Musa x paradisiaca* L. var. paradisiaca; 'Russett Burbank' potato, *Solanum tuberosum* L.; cucumber, *Cucumis sativus* L.; carrot, *Daucus carota* subsp. sativus (Hoffm.) Arcang.; cantaloupe, *Cucumis melo* L.; and avocado, *Persea Americana*, Miller var. americana.

2.2 Permittivity Measurements

The electrical measurements necessary for permittivity determination were obtained with the Hewlett-Packard 85070B open-ended coaxial-line probe, a Hewlett-Packard 4291A Impedance/Material Analyzer, and a temperature-controlled stainless steel sample cup and water jacket assembly designed and built for use with the 85070B probe [17]. Permittivities (dielectric constants and loss factors) were calculated with Agilent Technologies 85070D Dielectric Probe Kit Software, modified for use with the HP 4291A Analyzer by Innovative Measurement Solutions, which provided permittivity values from the reflection coefficient of the material in contact with the active tip of the probe [18]. For further information on the equipment used, sample preparation, physical measurements, and the procedures followed, the reader is referred to the earlier publication on this work, where detailed descriptions are provided. [16].

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1 Mention of company or trade names is for purpose of description only and does not imply endorsement by the U. S. Department of Agriculture.
3 Results and Discussion

Measured characteristics of the fruit and vegetable tissue samples, other than dielectric properties, are presented in Table 3.1.

Table 3.1 Characteristics of fruit and vegetable tissue samples

<table>
<thead>
<tr>
<th>Fruit or vegetable</th>
<th>Cultivar or other description</th>
<th>Moisture content %</th>
<th>Tissue density g/cm³</th>
<th>Total soluble solids %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Apple</td>
<td>'Red Delicious'</td>
<td>85</td>
<td>0.81</td>
<td>13.4</td>
</tr>
<tr>
<td>Avocado</td>
<td></td>
<td>82</td>
<td>0.99</td>
<td>8.1</td>
</tr>
<tr>
<td>Banana</td>
<td>'Cavendish'</td>
<td>74</td>
<td>0.98</td>
<td>22.3</td>
</tr>
<tr>
<td>Cantaloupe</td>
<td>Muskmelon</td>
<td>87</td>
<td>0.97</td>
<td>13.0</td>
</tr>
<tr>
<td>Carrot</td>
<td></td>
<td>87</td>
<td>1.00</td>
<td>8.6</td>
</tr>
<tr>
<td>Cucumber</td>
<td></td>
<td>97</td>
<td>0.94</td>
<td>2.4</td>
</tr>
<tr>
<td>Grape</td>
<td>'Thompson Seedless'</td>
<td>83</td>
<td>1.01</td>
<td>17.3</td>
</tr>
<tr>
<td>Orange</td>
<td>Navel</td>
<td>89</td>
<td>1.04</td>
<td>13.1</td>
</tr>
<tr>
<td>Potato</td>
<td>'Russett Burbank'</td>
<td>77</td>
<td>1.08</td>
<td>6.4</td>
</tr>
</tbody>
</table>

The variation of the dielectric properties of samples from these nine fruits and vegetables with frequency and temperatures from 5 to 95 °C was reported previously [16]. To provide graphical values for the dielectric constant and loss factor that can be better discerned at the higher frequencies, the data were plotted on logarithmic scales vs the log of frequency, and examples are presented for navel orange and cucumber in Figs. 1 and 2, respectively. As noted previously [16], at some frequency in the range between 10 and about 120 MHz, the temperature dependence of \( \varepsilon' \) disappears. This frequency is 50 MHz for orange tissue and 16 MHz for cucumber tissue. Both the dielectric constant and loss factor show monotonic decreases in value as frequency increases. Consistent trends with temperature in the range from 5 to 65 °C were noted as shown in Figs. 1 and 2. The high values of \( \varepsilon' \) at the lower end of the frequency range are no doubt attributable to the polarization contributed by ionic conduction, while the behavior of \( \varepsilon' \) at the higher end of the frequency range is characteristic of dipolar relaxation.

Values of the permittivity at 25°C are shown for all of the nine fruit and vegetable tissues at frequencies of 10 and 100 MHz and 1 GHz in Table 3.2. Comparing the magnitudes of the permittivity components, \( \varepsilon' \) and \( \varepsilon'' \) at low frequencies, carrot has the greatest values for both, followed, with respect to \( \varepsilon' \), by avocado and cantaloupe, orange and potato, banana,

Table 3.2 Permittivities of fresh fruits and vegetables at indicated frequencies at 25 °C

<table>
<thead>
<tr>
<th>Fruit or vegetable</th>
<th>10 MHz</th>
<th>100 MHz</th>
<th>1 GHz</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( \varepsilon' )</td>
<td>( \varepsilon'' )</td>
<td>( \varepsilon' )</td>
</tr>
<tr>
<td>Apple</td>
<td>109</td>
<td>281</td>
<td>71</td>
</tr>
<tr>
<td>Avocado</td>
<td>245</td>
<td>759</td>
<td>66</td>
</tr>
<tr>
<td>Banana</td>
<td>166</td>
<td>834</td>
<td>76</td>
</tr>
<tr>
<td>Cantaloupe</td>
<td>260</td>
<td>629</td>
<td>70</td>
</tr>
<tr>
<td>Carrot</td>
<td>598</td>
<td>1291</td>
<td>87</td>
</tr>
<tr>
<td>Cucumber</td>
<td>123</td>
<td>361</td>
<td>80</td>
</tr>
<tr>
<td>Grape</td>
<td>122</td>
<td>570</td>
<td>78</td>
</tr>
<tr>
<td>Orange</td>
<td>197</td>
<td>617</td>
<td>78</td>
</tr>
<tr>
<td>Potato</td>
<td>183</td>
<td>679</td>
<td>73</td>
</tr>
</tbody>
</table>
Fig. 1 Frequency and temperature dependence of navel orange tissue permittivity

cucumber and grape, and finally by apple with the lowest $\epsilon'$ range. With respect to $\epsilon''$, carrot is followed by avocado and banana, cantaloupe and potato, orange and grape, with cucumber and apple having the lowest range. Most of the data at 200 MHz compare very well with values reported for the dielectric constants and loss factors of similar fruits and vegetables at that frequency in earlier measurements with a different open-ended coaxial-line probe and microwave network analyzer for the frequency range from 200 MHz to 20 GHz [15].
Examination of Figs. 1 and 2 shows that there is a good linear relationship between the log of $\varepsilon''$ and the log of frequency over the range from 10 to about 300 MHz, and this was true also for all the other fruits and vegetables. Since, at any given frequency in this range, there also appears to be a linear relationship between the dielectric properties and temperature, three-dimensional plots were examined, which show that all points fall very close to a plane in three-dimensional space (Fig. 3). Thus, multiple linear regressions were calculated, and resulting statistics are presented in Table 3.3. Coefficients of determination, $r^2$, are all 0.996.
Table 3.3 Regression statistics for Equation 1 and zero-temperature-dependence frequency $T_0$ for $\varepsilon'$ of fresh fruits and vegetables (Table 3.1).

<table>
<thead>
<tr>
<th>Fruit or vegetable</th>
<th>$a_0$</th>
<th>$a_1$</th>
<th>$a_2$</th>
<th>$r^2$</th>
<th>$T_0$ MHz</th>
</tr>
</thead>
<tbody>
<tr>
<td>Apple</td>
<td>8.518</td>
<td>-0.892</td>
<td>0.0059</td>
<td>0.996</td>
<td>23</td>
</tr>
<tr>
<td>Avocado</td>
<td>9.196</td>
<td>-0.924</td>
<td>0.0055</td>
<td>0.996</td>
<td>90</td>
</tr>
<tr>
<td>Banana</td>
<td>9.343</td>
<td>-0.940</td>
<td>0.0056</td>
<td>0.999</td>
<td>50</td>
</tr>
<tr>
<td>Cantaloupe</td>
<td>9.232</td>
<td>-0.941</td>
<td>0.0063</td>
<td>0.999</td>
<td>80</td>
</tr>
<tr>
<td>Carrot</td>
<td>9.468</td>
<td>-0.928</td>
<td>0.0052</td>
<td>0.998</td>
<td>120</td>
</tr>
<tr>
<td>Cucumber</td>
<td>9.020</td>
<td>-0.944</td>
<td>0.0054</td>
<td>0.999</td>
<td>16</td>
</tr>
<tr>
<td>Grape</td>
<td>9.224</td>
<td>-0.950</td>
<td>0.0067</td>
<td>0.998</td>
<td>23</td>
</tr>
<tr>
<td>Orange</td>
<td>9.130</td>
<td>-0.931</td>
<td>0.0068</td>
<td>0.999</td>
<td>50</td>
</tr>
<tr>
<td>Potato</td>
<td>9.114</td>
<td>-0.922</td>
<td>0.0061</td>
<td>0.999</td>
<td>70</td>
</tr>
</tbody>
</table>

or greater. Therefore, very good values for the loss factor $\varepsilon'$ of these fresh fruit and vegetable tissues in the frequency range from 10 to 300 MHz over temperatures from 5 to 65 °C can be calculated with the following equation by using the appropriate coefficients for the particular fruit or vegetable:

$$\log \varepsilon' = a_0 + a_1 \log f + a_2 T$$  \hspace{1cm} (1)

where $f$ is in Hz and $T$ is in degrees C.

The log-log plots revealed evidence of a dielectric relaxation phenomenon above 2 GHz. Some plots for the dielectric constant (Fig. 2) indicated the beginning dispersion characteristics of such relaxations around 1 GHz, and the loss-factor plots showed the associated increase toward a peak value at a relaxation frequency above 2 GHz. Also, as temperature increased, the relaxation frequency shifted to higher frequencies. Earlier measurements on fresh fruit and vegetable tissues between 200 MHz and 20 GHz revealed the influence of dielectric relaxations associated with bound and free forms of water [14]. The influence of free liquid water was clearly evident, since it has a relaxation frequency of 19.2 GHz at 25 °C [19]. Because the relaxation frequency of free water at 5 °C is 10.7 GHz, it is not surprising to note the evidence of this relaxation in Figs.1 and 2 above 1 GHz. As temperature increases, the relaxation frequency of pure liquid water increases to 27.3 GHz at 40 °C and to 39.7 GHz at 60 °C. Thus, these are significant frequency shifts that explain the behavior noted in the dielectric properties above 1 GHz.
The new frequency and temperature-dependent permittivity data provide information that can be useful in understanding the dielectric behavior of such materials. They are useful in explaining the behavior of these materials in radio-frequency and microwave dielectric heating applications. They also provide background information that will be useful in studies aimed at sensing fruit and vegetable quality through measurements that utilize the dielectric properties of these kinds of agricultural products. The lack of temperature dependence at certain frequencies might provide an advantage in sensing qualities in a practical instrument.

4 Conclusions

Measurements of the permittivities of tissue samples cut from nine different fresh fruits and vegetables over the frequency range from 10 MHz to 1.8 GHz at temperatures ranging from 5 to 65 °C revealed dielectric constants decreasing from values of several hundred at 10 MHz to less than 100 at 1.8 GHz. Dielectric loss factors of about one thousand in value at 10 MHz decreased regularly to less than 20 at 1.8 GHz. The dielectric constants increased regularly with temperature at lower frequencies in this range but decreased with temperature at higher frequencies. Loss factors increased consistently with increasing temperature. For all fruit and vegetable tissue samples, there was a point in the region between 10 and 120 MHz, where temperature dependence of the dielectric constant disappeared. At frequencies below this point, ionic conduction dominated the dielectric behavior, and at higher frequencies, dipolar dielectric relaxation losses were dominant. In the frequency range from 10 to 300 MHz, the log of the dielectric loss factor is linear with the log of frequency. Linearity with temperature, also, permits the use of multiple linear regression for closely calculating the dielectric loss factor at frequencies from 10 to 300 MHz over the 5 to 65 °C temperature range. These data provide new information concerning the frequency and temperature-dependent behavior of the dielectric properties that may be useful in dielectric heating applications and as
background material in exploring the dielectric properties of fruits and vegetables for potential new quality sensing applications.

References


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A Theoretical Relationship Between the Fractal Dimension and Moisture Content in Grains

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ABSTRACT. Particles of a particulate (grains) material could have a dominant geometrical shape such as spheres, discs, ovals …etc. The bulk density of the material is affected by the shape of the particles, hence the shape has an effect on the way in which the particles rest on each other. As moisture content of the grains change the sizes of the spaces between the grains change as well, causing the grains to align or orientate themselves towards a dominant direction. For groups of grains, their boundaries can form profiles or contours which define regions that can be used to determine the fractal dimension for the grains.

Mathematical relationships have been suggested to determine the fractal dimensions of grains by using measured microwave parameters such as phase shift and attenuation. The fractal dimension has been used as a mean to detect the seating configurations and the spaces between the grains as the moisture content of the grains increase.

Keywords: fractal dimension, moisture content, dielectric constant

1 The Shapes of the Grains and the Fractal Dimension

Since Mandelbort [1] proposed geometrical fractal geometry, the fractal has been widely used in various fields to describe some irregular phenomena in nature. For example, the application of fractal geometry provides an effective tool in the study of highly irregular surfaces, and profiles.

The apparent length of a fractal profile, \(L_p\) is correlated to the measuring step, \(\zeta\), and the fractal dimension \(D\) is given by [2-10]:

\[
L_p(\zeta) = N(\zeta) \zeta^{(1-D)}
\]

Few studied have correlated the geometrical shape of the particles of the particulate material to microwave parameters such as frequency, bandwidth, amplitude, attenuation and phase shift. However in previous study we [11] used the fractal dimension as a tool to determine the effects for the physical spaces, orientations and alignments of the grains on the microwave parameters. The theoretical model and experimental results indicated that the fractal dimension values fluctuate as the moisture content of the particles increase, reflecting the change in the spaces between the particles and the orientations for the particles.

2 The Fractal Dimension and the Dielectric Properties

The relative complex permittivity, \(\varepsilon = \varepsilon' + j\varepsilon''\) where \(\varepsilon'\) is the dielectric constant and \(\varepsilon''\) is the loss factor, is often used to study the structure and composition of materials. This intrinsic parameter is dependent on several variables, such as frequency, temperature, density and moisture content. Therefore, it can be used to determine most of these variables if appropriate correlations are established.
If an electromagnetic plane wave passes through a homogenous dielectric medium, the complex transmission coefficient $T$ is [11];

$$T = \frac{(1 - \Gamma^2) e^{j\gamma L}}{(1 - \Gamma^2 e^{-\gamma L})}$$ (2)

$\Gamma$ = complex reflection coefficient.
$\gamma$ = complex propagation constant.
$L$ = thickness of the medium.

Where

$$\Gamma = \frac{(1 - \varepsilon^{1/2})}{(1 + \varepsilon^{1/2})}$$ (3)

and

$$\gamma = \alpha + j\beta = j(2\pi/\lambda)\varepsilon^{1/2}$$ (4)

$\alpha$ = the attenuation constant.
$\beta$ = phase constant.
$\lambda$ = free space wavelength.

If $\varepsilon >> \varepsilon^\prime$, solving equation 4 produces;

$$\alpha = \left(\pi\varepsilon^\prime/\lambda\varepsilon^{1/2}\right)$$ (5)

$$\beta = \left(2\pi\varepsilon^{1/2}/\lambda\right)$$ (6)

$$|\Gamma| = \left(\varepsilon^{1/2} - 1\right)/\left(\varepsilon^{1/2} + 1\right)$$ (7)

The complex transmission can be written in the polar mode as

$$T = |T|e^{j\theta}$$ (8)

The attenuation and phase shift are related to the complex transmission coefficient as:

$$A = -20 \log|T|$$ (9)

$$\Phi = \theta + 2\pi n$$ (10)

$n$ = zero or positive integer.
$\theta$ = argument.

Solving equations 5, 6, and 7 produces [12-14];

$$\varepsilon^\prime \approx \left(1 + (\Delta\Phi\lambda)/(360L)^2\right)^2$$ (11)

$$\varepsilon^\prime \approx \left(\Delta\lambda\varepsilon^{1/2}\right)/(8.686\pi L)$$ (12)

$\Phi$ = the phase shift in degree.
$\Delta\Phi = \Phi - \Phi^\circ$, with $\Phi^\circ = 2\pi/\lambda$ is the phase shift in empty cavity.
$\Delta\lambda$ = attenuation introduced by the sample.
Equation 11 is approximated to;

\[ \varepsilon \approx (\Delta \Phi c/f)(1/L_p) \]  \hspace{1cm} (13)

c=300 \times 10^6 \text{ m/s} = \text{speed of light.}

$L_p$=sample thickness =length of profile or contour around the regions, it will be used to determine the fractal dimension, $D$.

$f$= resonance frequency.

For each moisture content $L_p$, $\Phi$, and $\varepsilon$ are dependent on step size, $\zeta$, used to measure the total length of the profile or contour around the regions, thus;

\[ L_p(\zeta) \approx (c/f)(1/ \varepsilon'^2) (\Delta \Phi(\zeta)) \]  \hspace{1cm} (14)

\[ \ln(L_p(\zeta)) \approx (c/f) \ln(\Delta \Phi(\zeta)/ \varepsilon'^2) \]  \hspace{1cm} (15)

From equation 1

\[ \ln(L_p(\zeta)) = C+(1-D) \ln(\zeta) \]  \hspace{1cm} (16)

$C$= equation’s constant.

Then

\[ C+(1-D) \ln(\zeta) \approx (c/f) \ln(\Delta \Phi(\zeta)/ \varepsilon'^2) \]  \hspace{1cm} (17)

If

\[ K=(c/f) \]  \hspace{1cm} (18)

Then equation 17 can be rewritten to;

\[ C+(1-D) \ln(\zeta) \approx K \ln(\Delta \Phi(\zeta)/ \varepsilon'^2) \]  \hspace{1cm} (19)

If the step size, $\zeta$, is as large as the thickness of the sample $L_p$, then;

\[ C+(1-D) \ln(L_p) \approx K \ln(\Delta \Phi(\zeta)/ \varepsilon'^2) \]  \hspace{1cm} (20)

Plotting $\ln(L_p)$ against $K \ln(\Delta \Phi(\zeta)/ \varepsilon'^2)$ produces a liner relationship with a slope equal to 1- $D_\Phi$ ($D_\Phi$ =fractal dimension deduced from the phase shift measurements).

The fractal dimension can also be determined by assuming that the $L_p$, $\varepsilon'$, and $A$ are functions of step size, $\zeta$, for each moisture content. Equation 12 is simplified to;

\[ \varepsilon' '(\zeta) \approx (\Delta A(\zeta))(c/f) (\varepsilon'^{1/2}) (1/L_p(\zeta)) \]  \hspace{1cm} (21)

Then

\[ L_p(\zeta) \approx (c/f) (\varepsilon'^{1/2}) (\Delta A(\zeta))/ (\varepsilon' '(\zeta)) \]  \hspace{1cm} (22)

\[ \ln(L_p(\zeta)) = C' +(1-D) \ln(\zeta) \approx (c/f) \ln(\Delta A(\zeta) (\varepsilon' '(\zeta)^{1/2}))/ (\varepsilon' '(\zeta)) \]  \hspace{1cm} (23)

$C'$= equation’s constant

By assuming the step size is large = $L_p$=sample thickness, equation 22 can be rewritten as
\[ C' + (1-D_A) \ln(L_p) \approx K \ln(\Delta A(\zeta) (\varepsilon'(\zeta)^{1/2})/(\varepsilon''(\zeta))) \] 

(24)

Plotting \( \ln(L_p) \) against \( K \ln(\Delta A (\varepsilon'(\zeta)^{1/2})/(\varepsilon''(\zeta))) \) produces a liner relationship with a slope equal to 1- \( D_A \) (\( D_A \) =fractal dimension deduced from the attenuation measurements).

3 Results and Discussion

Kraszewski A. [15] admitted that variations and fluctuations of the bulk density of grain have a major factor on limiting the accuracy of moisture content measurements by dielectric technique. He emphasised that the bulk density depends on the grains shapes and physical dimension. However he concluded useful mathematical equations which correlate the attenuation and phase shift to sample thickness, moisture content and density. The results of Kraszewski‘s work has been used in this study. The concluded constants and equations were used to calculate the samples thicknesses, and moisture content of the grains. The dielectric constant and dielectric loss were calculated using equations 11 and 12.

Fig. 1 shows that the value for the fractal dimension of the grains decreases as the moisture content increase. Detail analysis to the curves suggests that the fractal dimension values of the grains calculated from the phase shift fluctuate as their moisture content increase. Fig. 2 show that the fractal dimension calculated from the attenuation increases with increasing the moisture content for all the type of grains except corn.

Further experimental work is required to prove the reliability and repeatability of the mathematical relationships using different grades of grains and food products.

![Graph showing the relation between fractal dimension and moisture content](image-url)

Fig. 1 Show the relation between the fractal dimension calculated using equation 20 against the moisture content.
Fig. 2 Show the relation between the fractal dimensions calculated using equation 25 against the moisture content.

4 Conclusions

The fractal dimensions of grains can be determined by using simple mathematical relationships which correlate the microwave parameters such phase shift and attenuation to the dielectric constants of the material. The fractal dimensions were correlated to the moisture content of the grains to show the impact of moisture on the spaces between the grains and on the orientations and alignments for the grains.

References

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Session 9

Moisture Content Determination in Soil, Snow and Waste Disposals
Chairmen: A. Brandelik, Ch. Hübner

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S. K. Goel, S.K.Mittal, N. Gupta; Chandigarh, India

2 Water Content Measurements in Soil Column Tests with a New Electromagnetic Moisture Sensor
F. Fabian; Blankenburg/Freiberg; R. M. Zeh, K. J. Witt; Weimar, Germany

3 Alternative Surface Covering of Landfill Using the TAUPE Sealing Control System
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4 Measurement Method for Detection of Moisture Profiles in a Saline Environment
K. Kupfer, E. Trinks, Th. Schäfer, N. Wagner; Weimar; Ch. Hübner; Mannheim, Germany
Development of a Sensor for In-situ Determination of Snow Moisture & Density

Shiv Kumar Goel¹, S.K. Mittal², Neena Gupta¹
¹P.E.C. Chandigarh, India
²CSIO-Chandigarh, India

ABSTRACT. In this paper the concept of development a portable snow moisture resonator for quick and non-destructive in situ determination of snow liquid water content and density is presented. The design of this sensor is based on the relationship between the frequency and complex dielectric constant of snow. The developed resonator is based on the measurement of dielectric properties of the snow and or any other porous, granular or liquid material around 900 MHz. The present sensor developed is open ended parallel wire transmission-line resonator. The open-ended structure at both end leads to improved quality factor and hence wide range of moisture measurement. Owing to very simple structure measurement is non-destructive. The sensor is calibrated in various material such as water, sand, salt sugar. The results are compared with the standard values that are already available using snow probe.

1 Introduction

The dielectric constant of snow is a weighted average of the dielectric constants of its components: air, ice and liquid water. The dielectric constant of water (\( \varepsilon_w = 88 - j9.8 \) at 1 GHz) is large relative to that of ice (\( \varepsilon_i = 3.15 - j0.001 \)) and therefore a very small amount of water causes a significant change in the overall dielectric properties of snow medium, particularly with respect to the imaginary part[1, 2].

Although the conventional techniques to measure the snow density and liquid water content offers best accuracy, but are too complex and time consuming[2, 3]. Measuring snow moisture is a complicated task because of the rapid snow metamorphism at and near melting point. Any disturbance of snow may change its structure, texture and moisture rapidly[4]. This creates a need to develop electromagnetic sensor for quick and easy determination of snow moisture and density.

2 The Instruments

A variety of instruments with different operating frequencies and different sensors are available in international market, for the field measurement of snow density and liquid water content [2, 4, 5, 6]. Among conventional instruments “freezing calorimeter” offers best accuracy (≈ 1%) but cumbersome and time consuming. Second, it measures mass fraction of liquid water in snow sample so to covert mass fraction in to volumetric fraction, a separate measurement of snow density is required [4].

There have in recent years a host of instruments which retrieve snow parameter quickly and non-destructively, by measuring the dielectric constant of snow and relating it to the physical parameters. The most attractive candidate among these has been the “Snow Fork”, a microwave instrument in Finland.
The strength of this technique are simplicity of equipment, speed of measurement, high spatial resolution and ability to measure both the real & imaginary parts of dielectric constant of snow allowing for more power algorithm of snow wetness and density with a single measurement[2,4].

The modified design called snow probe developed at Radiation Laboratory, University of Michigan, and Ann Arbor. is designed to improve the sensitivity of instrument to moisture content and the effective sampled volume of the snow medium ,thereby, improving the spatial resolution of the sensor. For calibration density measurement is performed using standard tube of known volume, whose weight is measured both empty and full of snow [2,4].

In the present work, the designed sensor is based on “Snow Fork”.

3 Resonator Design

The developed snow sensor consists of a resonator that can be pushed into snow or any other porous, granular or liquid material under measurement. The resonator is a parallel wire transmission-line resonator that is open-ended at both the ends. In the present work, the sensor designed is based on “Snow Fork” as mentioned above.

The purpose of the resonator is to find out the complex permittivity of the material under measurement. This is achieved by measuring the change in the resonance curve of the resonator when it is pushed into material. The sensor is a fork shaped quarter wavelength microwave resonator, which is resonant at the frequency for which the length of resonant structure is one forth of wavelength, which is open circuited at both the end as shown in fig.1.

Fig.1 Design of open ended parallel wire transmission-line resonator.

A resonant frequency in air around 900 MHz makes approximately 8.33 cm length of prongs. But actual length called effective length is 7.3 cm. The differences between theoretical and actual value of length can be explained on the basis of that wave velocity in conductor is not same as wave velocity in the free space. The radio frequency power is fed in and out of the structure through coaxial cables and coupling loops.

The sensor works on the principle that when prongs are inserted in snow the real part of permittivity of snow lowers the resonant frequency; and imaginary part broadens the resonance curve and increases the signal attenuation at the resonance frequency. , and Snow Fork is explicitly designed for field use; it is light, quick and easy to use. Snow Fork measures the electrical parameters: resonant frequency, attenuation and 3-dB bandwidth. The measuring results are used to calculate accurately the complex dielectric constant of snow. Further, the liquid water content and density of snow are calculated using semi-empirical
equations. All data will be shown immediately on the display and can be stored in a solid-state memory.

The measurement by the instrument with operating frequency below 100 MHz is quite destructive and at high frequencies, especially in the GHz region, closed systems such as waveguides are, in general, impractical for field use [1, 8]. Second, real part of dielectric constant of snow is frequency independent from 10 MHz to 1.3 GHz. Third, 1 GHz scattering losses are negligible for snow particle size [1, 2 and 3].

The prongs of sensor are made up of stainless steel. The distance between two prongs is about 1.8 cm. Prongs are thin enough (2mm for stainless steel & 3mm for copper) with sharpen ends for easy and deformation less insertion into snow pack even through the possible crust. Also, prongs are thick enough so that the measurement operation practically does not cause the bending of prongs when inserted in to the snow pack. The bending of prongs can be seen when pushed into an ice pack.

3.1 Material for Prongs

The best suited material for making prongs is stainless steel. It has excellent corrosion resistance, very high strength and good ductility. The very high chromium and nickel content provides improved high temperature strength and oxidation resistance. The properties of some material are shown in table1.

Table 1 Properties of materials

<table>
<thead>
<tr>
<th></th>
<th>Stainless Steel</th>
<th>Copper</th>
<th>Aluminium</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density in g/cc at 20°C</td>
<td>9.4</td>
<td>8.94</td>
<td>2.703</td>
</tr>
<tr>
<td>Electrical resistivity (10⁻⁹ Ω-m) at 20°C</td>
<td>30.6</td>
<td>17.2</td>
<td>28.3</td>
</tr>
<tr>
<td>Temp. coefficient of Resistivity( C⁻¹)</td>
<td>0.0036</td>
<td>0.0039</td>
<td>0.0039</td>
</tr>
</tbody>
</table>

The less strength of Aluminium and Copper causes radius of prongs to be thick as compared to stainless steel which results in more deformation of material under test. Even Copper gets oxidized in acidic solution.

3.2 Electroplating

The soldering of stainless steel with on copper printed circuit board is not possible because of large difference in melting point of stainless steel and copper. So the ends (3 mm) of stainless steel prongs are electroplated with copper before soldering.
3.3 Spacing between prongs

The capacitance of a pair of parallel wires of spacing \( s \) and radius \( r (s>>r) \) is given by

\[
C = \frac{\varepsilon \pi}{\ln \left( \frac{s}{r} \right)} \quad \text{F/m}
\]  

(1)

The inductance of a pair of parallel wires of spacing \( s \) and radius \( r (s>>r) \) is given by

\[
L = \left( \frac{\mu}{\pi} \right) \ln \left( \frac{s}{r} \right) \quad \text{H/m}
\]  

(2)

This parallel wire transmission line type resonator behave as series resonant circuit so resonant frequency is given by

\[
f_r = \frac{1}{2\pi\sqrt{LC}}
\]  

(3)

Substituting value of 1 & 2 in 3, we have

\[
f_r = \frac{1}{2\pi\sqrt{\pi\varepsilon}}
\]  

(4)

This is independent on spacing between prongs \( s \) and radius of prongs \( r \).

The Quality factor is Given By

\[
Q_0 = \left( \frac{1}{R} \right) \sqrt{\frac{L}{C}}
\]  

(5)

Substituting value of 1 & 2 in 5,we have

\[
Q_0 = \frac{1}{\pi R} \left( \frac{\mu}{\varepsilon} \right) \ln \left( \frac{s}{r} \right)
\]  

(6)

This depends on spacing between prongs \( s \) and radius of prongs \( r \).

For air \( \mu = \varepsilon = 1 \), \( Q_0 = \left( \frac{1}{\pi R} \right) \ln \left( \frac{s}{r} \right) 
\]  

(7)

The Quality factor is also given by

\[
Q_0 = \frac{f_0}{\Delta f}
\]  

(8)

Where \( \Delta f \) is 3-dB bandwidth corresponding to \( f_0 \), \( f_0 \) is the resonance frequency of sensor when it was in air at material temperature.

The high value of \( Q_0 \) (79.867) is achieved by maximizing \( s/r \). The higher value of \( Q_0 \) means higher voltage amplification & higher selectivity. More increase in value of \( Q_0 \) causes peak of resonance curve so sharp that accurate measurement of 3-db bandwidth is not possible.
Further, to maximize the value of $Q_0$ and to decrease the radiation losses whole assembly is protected by a epoxy plastic pack.

4 Applications

Life in snowbound high altitude areas throws up peculiar challenges, both physical and psychological. The harsh climate, rarefied atmosphere, ever-present threat of avalanches and crevasses make mere survival an experience by itself [8, 9].

Snow avalanche are powerful destructive forces of nature, which cause widespread damage to men and materials. Catastrophic avalanches are caused by sustained heavy snowfall under severe weather conditions on critical slopes. Prediction of avalanche occurrence demands the measurement of various snow and meteorological parameters of the avalanche formation zones and monitoring the changes at frequent time intervals [10]. Specific kind of state of the art instruments and systems are needed for unmanned and automatic data collection and processing. Data collected by these instruments will provide valuable information on:

- Advance forecasting of avalanches occurrence in the western and central Himalayas for the army as well as civilian population.
- The technology developed under the proposed program can be used directly for the instrumentation for ‘Antarctica Programme’ [11, 12 and 13].

5 Results and Conclusion

The radio frequency power is fed in and out of the structure through coaxial cables with the use of spectrum analyzer. The value of resonant frequency of resonator made of stainless steel and copper in different material under test is given in tabular form below:

<table>
<thead>
<tr>
<th></th>
<th>Air</th>
<th>Water</th>
<th>Sand</th>
<th>Salt</th>
<th>Sugar</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stainless steel</td>
<td>902.5</td>
<td>105.7</td>
<td>548</td>
<td>531.7</td>
<td>649.5</td>
</tr>
<tr>
<td>Copper</td>
<td>905</td>
<td>108</td>
<td>563</td>
<td>553</td>
<td>638</td>
</tr>
</tbody>
</table>

To determine the complex dielectric constant of snow and to calculate the snow moisture and density by making the use of a set of empirical relations [2, 5, 6, 7] described by various snow researchers. The real part of dielectric constant $\varepsilon'_r$ is determined from the resonant frequency $f_s$ when the sensor prongs are completely inserted in material under test:

$$\varepsilon'_r = \left(\frac{f_a}{f_s}\right)^2$$  \hspace{1cm} (9)

Where $f_a$ is the resonance frequency of sensor when it was in air at material temperature.

The value of real part of dielectric constant ($\varepsilon'_r$) of resonator made of stainless steel and copper in different material under test is given in tabular form below:
Table 3  Real part of dielectric constant ($\varepsilon'$) of resonators in test materials

<table>
<thead>
<tr>
<th></th>
<th>Water</th>
<th>Sand</th>
<th>Salt</th>
<th>Sugar</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stainless steel</td>
<td>72.9</td>
<td>2.71</td>
<td>2.88</td>
<td>1.93</td>
</tr>
<tr>
<td>Copper</td>
<td>70.2</td>
<td>2.58</td>
<td>2.67</td>
<td>1.42</td>
</tr>
<tr>
<td>Std. Value</td>
<td>75</td>
<td>2.78</td>
<td>2.88</td>
<td>1.98</td>
</tr>
</tbody>
</table>

Standard values are obtained using snow probe as published in paper [1]

The percentage error in real part of dielectric constant ($\varepsilon'$) of resonators made of stainless steel and copper in different material under test is given in tabular form below:

Table 4  The percentage error in real part of dielectric constant ($\varepsilon'$) of resonator in different material under test

<table>
<thead>
<tr>
<th></th>
<th>Water</th>
<th>Sand</th>
<th>Salt</th>
<th>Sugar</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stainless Steel</td>
<td>2.8</td>
<td>2.5</td>
<td>2.7</td>
<td>2.5</td>
</tr>
<tr>
<td>Copper</td>
<td>6.4</td>
<td>7.19</td>
<td>7.29</td>
<td>8.2</td>
</tr>
</tbody>
</table>

The table shows that results obtained from resonator made of stainless steel are better than resonator made of copper.

The value of 3-dB bandwidth ($\Delta f = f_2 - f_1$) of resonator made of stainless steel in different material under test is given in tabular form below:

Table 5  3-dB bandwidth ($\Delta f = f_2 - f_1$) of resonator in different material under test

<table>
<thead>
<tr>
<th></th>
<th>salt</th>
<th>air</th>
<th>water</th>
<th>sand</th>
<th>sugar</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stainless Steel</td>
<td>40</td>
<td>11.3</td>
<td>4.1</td>
<td>1.3</td>
<td>10</td>
</tr>
</tbody>
</table>

Imaginary part of dielectric constant $\varepsilon''$, which represents the dielectric losses, is determined by solving the equation

$$
\varepsilon'' = \left( \frac{f_s}{f_r} \right)^2 \left[ \frac{\Delta f_s}{f_s} \left( p + \frac{b}{f_s} \right) \right] 
$$

(10)

Where $\Delta f_s$ is 3-dB bandwidth corresponding to $f_s$, $p$ and $b$ are resonator constants. The value of constants $p$ & $b$ is to be set when resonator is tested into the snow.

6  Empirical Relations

Further more, from the dielectric constant, the snow moisture ($W$) & density ($\rho$) are calculated from the solution of empirical relations listed by the research work of various snow research groups, Helsinki University of Technology, University of Innsbruck etc, which are frequency independent in microwave region where ice is dispersion less. These are as follows:
\[
\begin{align*}
\varepsilon_d' &= 1 + 1.7 \rho_d + 0.7 \rho_d^2 \\
\Delta \varepsilon_d' &= \varepsilon_d' - \varepsilon_d' = (0.10W + 0.80W^2) \varepsilon_w' \\
\varepsilon_w'' &= (0.10W + 0.80W^2) \varepsilon_w'' 
\end{align*}
\]  
(11)  
(12)  
(13)

\( \varepsilon_d' \) is the real part of the dielectric constant of dry snow.

\( \varepsilon_s' \) is the real part of the dielectric constant of snow.

\( \varepsilon_s'' \) is the imaginary part of the dielectric constant of snow.

\( \rho_d \) is the density of snow when all liquid water in it is replaced with air.

\( \varepsilon_w = \varepsilon_w' - j \varepsilon_w'' \) is the complex dielectric constant of water.

\( \rho_d = \rho_s - W \)  
(14)

\( \rho_s \) is density of wet snow relative to the density of water.

\( W \) is wetness by volume.

Above relations are frequencies independent in microwave region.

7 Conclusion

The high value of quality factor will extend the moisture measurement range. The results for real part of dielectric constant are highly satisfactory. The results for imaginary part of dielectric constant are contradictory and can be approximated by changing the values of \( p \) & \( b \) corresponding to particular material.

8 Future Scope

The atomization of system can be done to directly display the values of density & water content. The extremely low power consuming Microcontroller based portable data acquisition unit for computing wetness & water content of snow pack can be used in future.

References


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Dr. Neena Gupta, Assistant Professor, P.E.C. Chandigarh, ng65@rediffmail.com
**Water Content Measurements in Soil Column Tests with a New Electromagnetic Moisture Sensor**

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**ABSTRACT.** In soil mechanics and in geotechnical engineering, the amount of water in soils plays an important role. Most of the soil characteristics, such as shear and tensile strength, compressibility, permeability etc. are directly dependent on the degree of water in the soil (mostly called ‘degree of saturation’).

Focusing on the desiccation behaviour of clay liners in cover lining systems of landfills, the authors conduct soil-column tests on multi-layered soil systems to examine the water flow processes caused by different temperature gradients (ZEH et al. 2004, FABIAN, 2004, ZEH & WITT, 2003). Before and after the tests, gravimetric water contents can be easily determined by using the oven-dry-method. During the tests, constant water content measurements are only possible by using fast indirect sensors/methods such as tensiometers, FDR- or TDR-sensors or electromagnetic moisture sensors.

In the test series, the authors installed micro-tensiometers (T5, UMS) and new electromagnetic moisture sensors called SISOMOP (Schlaeger et al., 2005, FABIAN, 2004, BECKER & HÜBNER, 2003, FABIAN, 2003), which can also measure the temperature.

In this article, the main focus is on the new sensor SISOMOP and its calibration with different soils (e. g. sand and clay). The sensor’s details and test results of the calibration are presented. Furthermore, the results of different soil-column tests are shown and analysed. A comparison of the test results with numerical calculations using the model SUMMIT (DÖLL, 1996) are also shown to provide evidence for good adaptations.

**Keywords:** Sisomop-sensor, micro-tensiometer, calibration, soil-column tests, numerical modelling

1 **Introduction**

In geotechnics and, as a specific application, in cover lining systems a lot of procedures are described by the water content and water transport in the soils. The water content in the clay liner, as the basic essential in a cover lining system of landfills, plays an important role in connection with the estimation of his integrity. Therefore, cracking of compacted clays in landfill liners (WITT & ZEH, 2004) often arise from the reduction of the water content which is caused by desiccation. A very interesting modification to prevent desiccation in clay liners of cover lining systems is the capillary breaking layer (KBS) as buffer layer above the mineral sealing components. To investigate the KBS efficiency the authors conducted some soil-column tests (FABIAN, 2004, ZEH & WITT, 2003).

2 **Basics – Water Transport in Soils**

In the conventional soil mechanics, a soil is a mixture of 3 parts, consisting of solids (aggregates or grains), water and air in the soil pores. The liquid phase is the principal element in the interaction of all parts. The theoretical interrelationship is based on the (potential-) concept of E. Buckingham (1907).

For this reason, different constituents of potentials exist in a soil. The soil ever aims for an energetic balance in the total potential, which is composed of gravitation, matric, air and osmotic potential.
If and only if the two main components of potentials (gravitation and matric) are unbalanced, a water transport is starting from higher to lower level of potential to reach an energetic equilibrium. All water movements in the soil are directly dependent on the soil suction, because the water tends from areas of high potential to those with lower potential. The water tension reflects the sum of the water holding forces in the soil.

Naturally deposited soils represent a wide spectrum of structure and texture. The soil-water dynamic processes are influenced by granulation, compaction, homogeneity, micro cracks, pore space etc. These spatial variable parameters should be regarded firstly. They are the basis to describe the research concept and to co-ordinate the measuring and sampling techniques.

2.1 Tensiometer Measuring Principle

Compared to all other measuring principles, the capillary bonding of water in soils can directly measured only by tensiometers. The water tension value (suction) shows the sum of holding forces. The forces are transferred straight to the water inside the tensiometer cup.

The accumulation of drain water, rising or lateral water and infiltration processes can be described by the suction and are brought on a denominator. The tensiometer pressure sensor is designed for this task and offers continuous readings.

The measuring range does not cover the complete range of possible suction. Thus, in the saturated zone (pilot water) tensiometers operate as piezometer or level probes. In insaturated zones they operate up to pF of 2.9 or matric suction of 850 hPa. Tensiometers must be filled with water. This requires a routine refilling. Tensiometers measure only punctually.

![Fig. 1 Measuring principal of a tensiometer](image)

Therefore, measured values cannot describe the overall status of an area. An interpretation is largely dependent on the soil type and structure. In general, it is difficult to describe macro pores, sandy or skeleton-rich soils as well as strongly heterogeneous soils, because the water movement can strongly vary spatially and temporally.
3 New Sensor – SISOMOP

For the water content measurements in the soil column tests, the authors used a new electromagnetic moisture sensor – SISOMOP (SCHLAEGER et al., 2005). Its functional principal based on the measurement of the dielectric count in the soil mixture. The basis of its application is the significant difference of dielectric counts from solid, water and air. Besides, the permittivity of air constitutes 1, of solid from 2 to 10 and finally the permittivity of liquid water 80. This relationship is described in equation 1.

$$\varepsilon_\alpha = n_W \cdot \varepsilon_\alpha + n_S \cdot \varepsilon_\alpha + n_A \cdot \varepsilon_\alpha$$

$\alpha$ – factor for nonlineary influences of permittivity

$$n_W, n_S, n_A \quad \text{– volumetric fractions (water, solid, air)}$$

$$\varepsilon_M, \varepsilon_W, \varepsilon_S, \varepsilon_A \quad \text{– dielectric permittivity (soil-mixture, liquid water, solid and air)}$$

The sensor based on the measurement the dielectric permittivity by arranging an electric field in the fitting soil mixture. The sensor’s measuring principal is specified in SCHLAEGER et al, (2005) and BECKER & HÜBNER (2003).

3.1 Calibration Curves – First Test Runs by SISOMOP

Before starting the soil column tests, the authors conducted preliminary tests for checking the capability of the new sensors SISOMOP. Therefore, an experimental testing rig were constructed.

The equipment consisted of a tube-ferrule (selected volume), a scale, the SISOMOP - sensor and a computer to record the measured data of the reduced weight by dessication and the moisture counts of SISOMOP.

In the tests, the authors used two different soils, a medium plasticity clay called Plessa (ML) and a sand (KBS and bedding-layer). The soils’ properties are represented in Table 1. A clay mineral analysis (MPA, 2003) showed that the soil has marginal active clay minerals.

Table 1 Soil properties

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Values</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>clay Plessa</td>
<td>sand</td>
<td></td>
</tr>
<tr>
<td>clay content</td>
<td>41.1 %</td>
<td>-</td>
</tr>
<tr>
<td>silt content</td>
<td>58.3 %</td>
<td>-</td>
</tr>
<tr>
<td>sand content</td>
<td>0.6 %</td>
<td>94.6 %</td>
</tr>
<tr>
<td>organic loss VGl</td>
<td>4.5 %</td>
<td>0.0 %</td>
</tr>
<tr>
<td>liquid limit w_l</td>
<td>44.9 %</td>
<td>-</td>
</tr>
<tr>
<td>plastic limit w_p</td>
<td>21.4 %</td>
<td>-</td>
</tr>
<tr>
<td>plasticity index I_p</td>
<td>23.5 %</td>
<td>-</td>
</tr>
<tr>
<td>consistency index I_c</td>
<td>0.94</td>
<td>-</td>
</tr>
<tr>
<td>water adsorption w_a</td>
<td>94.0 %</td>
<td>-</td>
</tr>
<tr>
<td>specific gravity $\rho_s$</td>
<td>2.676 g/cm³</td>
<td>-</td>
</tr>
<tr>
<td>Proctor density: Proctor optimum</td>
<td>1.65 g/cm³</td>
<td>1.715 g/cm³</td>
</tr>
<tr>
<td>optical water content</td>
<td>21.0 %</td>
<td>10.2 %</td>
</tr>
<tr>
<td>97 % of Proctor optimum</td>
<td>1.60 g/cm³</td>
<td>1.664 g/cm³</td>
</tr>
<tr>
<td>water content</td>
<td>16.6 %/24.4 %</td>
<td>5.3 %/ -</td>
</tr>
<tr>
<td>permeability kf, sat</td>
<td>8.1 E-11 m/s</td>
<td>1.3 E-4 m/s</td>
</tr>
<tr>
<td>shear strength $\varphi'$</td>
<td>25.0°</td>
<td>35.0°</td>
</tr>
<tr>
<td>cohesion c'</td>
<td>16.8 kN/m²</td>
<td>-</td>
</tr>
</tbody>
</table>

| 415 |
Totally, 4 tests were run, 3 with clay and 1 with sand. The test period was varied, because of the different desiccation behaviour of the two soils used. Table 2 represents the test conditions of the soils.

Table 2  soil conditions in front and end of test runs

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Soil type</th>
<th>1. test run</th>
<th>2. test run</th>
<th>3. test run</th>
<th>4. test run</th>
</tr>
</thead>
<tbody>
<tr>
<td>Built-in-density (humid)</td>
<td>clay</td>
<td>1994.13 kg/m³</td>
<td>1751.46 kg/m³</td>
<td>1754.46 kg/m³</td>
<td>1868.18 kg/m³</td>
</tr>
<tr>
<td>Built-in-density (dry)</td>
<td>clay</td>
<td>1622.56 kg/m³</td>
<td>1232.90 kg/m³</td>
<td>1244.74 kg/m³</td>
<td>1673.25 kg/m³</td>
</tr>
<tr>
<td>Built-in-water content</td>
<td>clay</td>
<td>22.90 %</td>
<td>42.06 %</td>
<td>40.95 %</td>
<td>11.05 %</td>
</tr>
<tr>
<td>Remove-density (humid)</td>
<td>sand</td>
<td>2010.23 kg/m³</td>
<td>1866.58 kg/m³</td>
<td>1859.56 kg/m³</td>
<td>1690.85 kg/m³</td>
</tr>
<tr>
<td>Remove-density (dry)</td>
<td>clay</td>
<td>1953.97 kg/m³</td>
<td>1748.88 kg/m³</td>
<td>1785.65 kg/m³</td>
<td>1673.25 kg/m³</td>
</tr>
<tr>
<td>Remove-water content</td>
<td>clay</td>
<td>2.88 %</td>
<td>6.73 %</td>
<td>4.14 %</td>
<td>1.05 %</td>
</tr>
</tbody>
</table>

The clay desiccated under the environmental condition of the laboratory (temperature). The weight and volume reduction of the soil was noticed. The soil moisture changes are also associated with soil density changes (increasing capillary soil strength). Therewith, the volumetric water content subject to measure moisture counts by SISOMOP can be specified. A characteristic soil count could be identified because of the very similar 3 test runs with the clay.

Figure 2 shows the measured moisture content of the clay in the second test run. It is not a pure linear relationship by the measured frequency and water content. Figure 3 shows similar effect. As a first linear approximation (approx. 25% and 35% volumetric water content) three zones by a curve linearity of SISOMOP moisture content relationship can be distinguished. All test were started on the humid zone 1. Besides, the clay desiccation can be defined as the loss of free liquid rates of water. For this reason, the dielectric count of soil mixture is falling nearly linear. In the transition of zone 2, the clay is conditioned by shrinkage.

Fig. 2  Distribution of measured moisture content by SISOMOP in clay Plessa – 2. test run

Fig. 3  Distribution of measured moisture content by SISOMOP in clay Plessa – 3. test run
The capillary bonding effects of water and solid is dominated. The density of clay increases and the volumetric solid fraction is become bigger in conjunction with the decrease of pore volume. Therefore, the dielectric count decreases in soil mixture, too. The measured frequencies are increasing, but the shrinkage initiates a change in the contact area of the soil and the sensor. Consequently, this coating of air involves a dielectric count declension and a rising of frequencies in zone 2, as you can see in figures 2, 3 and 4. In a specific volumetric water content (zone 1), the described effect above (relation of shrinkage and capillary bonding) decreases. All calibration curves in clay Plessa seems to be a characteristic.

In figure 5, the measured counts of the sand is presented. Besides, the capillary effects (adhesion, cohesion, pore size distribution) have small effects on the progression of the curve. It is nearly a linear relationship by the measured frequencies with the corresponding water content, because the sand densities is not really changed.

**4 Soil Column Tests**

In practice, lysimeters are used to investigate the water balance in mineral liners and components of cover lining systems in landfills under climatic conditions. This describes the in-situ-monitoring of the water content in multi-layer soil systems. Mini-lysimeters (e. g. soil columns) can be used to simulate vertical vapour and liquid water transport in unsaturated non-rigid porous media for analysis of desiccation and suction changes in soils, respectively, in the laboratory. Two multi-layer soil systems were investigated under the influence of a vertical temperature gradient in a mini-lysimeter, the soil column test. Details to the setup etc. are described in WITT et al. (2004) and FABIAN (2004). Figure 6 shows the multi-layer soils in our tests. Sand were used as a capillary breaking layer KBS. The test rig consists of a PVC rigid pipe, where the soil-layers were installed.
System A had a capillary breaking layer KBS over the mineral liner, System B did not have this. The temperature gradient was generated by water quench heaters, which were installed at the top and the bottom, each with different temperatures. Besides, a constant level were created. Temperature gradients were averaged a value of 10 K in System A and 7 K in System B. In each soil layer, a micro-tensiometer (T5, UMS) were installed, also two SISOMOP sensors in the clay liner to measure the moisture content in the soil column. Figure 8 shows the results of moisture content measured by the SISOMOP sensors, figure 9 measured by the tensiometers.

The tensiometers’ results are quoted in matric suction and not directly in volumetric water contents. The relationship of both values is given by water retention curves (SWCC or pF-curve). In both figures 7 and 8, the moisture content of the clay increases during the test period. This is demonstrated in figure 8 by increasing water content counts and in figure 7 by the downward drift of suction in the clay liner. In addition, the bedding layer and the capillary
breaking layer were desiccated (increasing values of suction in figure 7). In System B without a capillary breaking layer, the moisture content is also increasing during the test period as showed in figure 9 & 10.

But there is a significant difference dealing with the water content trends of the clay liner. In the System B, the matric suction in figure 9 increases after a first falling down and consequently the mineral liner desiccated slowly. This trend is not directly identified in figure 10 (measurement by SISOMOP).

But the test periods (10 days) were very short for a noticeable desiccation. Only in the measured results of the matric suction (fig. 10), the desiccation trend is visible, caused by the very small changes in water content by a clearly measurable changing in matric suction of the fine grained soil (cf. SWCC).

5 Numerical Simulations by SUMMIT

The programme SUMMIT (Döll, 1997a, 1996) is a numerical model to describe the one-dimensional transport of water in unsaturated porous soils. This programme was developed for the modelling and the examination of the desiccation risk in mineral (clay) liners of landfill lining systems under the influence of temperature gradients (Döll, 1997b). It also can be used to conduct numerical experiments to simulate vertical vapour and liquid water transport in unsaturated non-rigid porous media.

Hydraulic properties of the porous media can be estimated from pore-size distributions considering microscopic conceptual models for pore connectivity and pore tortuosity. The relative hydraulic conductivity function is indirectly derived from the water-retention curve by
using statistical models which calculate the pore-size distribution by deducing the water-retention curve.

To model the soil column tests, it is necessary to integrate the significant soil parameters into the programme. Table 3 shows the properties of the used soils.

Table 3  Soil properties

<table>
<thead>
<tr>
<th>Soil - Parameter</th>
<th>( n_p ) [-]</th>
<th>( \theta_s ) [-]</th>
<th>( \theta_r ) [-]</th>
<th>( \alpha ) [cm(^{-1})]</th>
<th>( n ) [-]</th>
<th>( l ) [-]</th>
<th>( K_{sat} ) [m/s]</th>
<th>( f_{Q} ) [-]</th>
<th>( f_{AM} ) [-]</th>
<th>( f_{OM} ) [-]</th>
<th>( \theta_b ) [-]</th>
</tr>
</thead>
<tbody>
<tr>
<td>KBS (sand)</td>
<td>0.415</td>
<td>0.415</td>
<td>0.002</td>
<td>0.15099</td>
<td>1.9476</td>
<td>0.5</td>
<td>1.3 \times 10^{-4}</td>
<td>0.50</td>
<td>0.50</td>
<td>0.00</td>
<td>0.06</td>
</tr>
<tr>
<td>Mineral liner (Clay Plessa)</td>
<td>0.372</td>
<td>0.372</td>
<td>0.000</td>
<td>0.00109</td>
<td>1.1202</td>
<td>0.5</td>
<td>8.11 \times 10^{-11}</td>
<td>0.20</td>
<td>0.60</td>
<td>0.00</td>
<td>0.10</td>
</tr>
<tr>
<td>Bedding layer (sand)</td>
<td>0.415</td>
<td>0.415</td>
<td>0.002</td>
<td>0.15099</td>
<td>1.9476</td>
<td>0.5</td>
<td>1.3 \times 10^{-4}</td>
<td>0.50</td>
<td>0.50</td>
<td>0.00</td>
<td>0.06</td>
</tr>
</tbody>
</table>

The results of the numerical modelling systems A and B were showed in figure 11 and 12. The trend (drawn and dashed circles) of a moisture content increasing in the clay liner can also be seen in the decreasing of the matric suction during the test period (10 days). As well as in systems A and B,

![Fig. 11 Time dependency of suction in modelling soil layers – System A](image1.png)

![Fig. 12 Time dependency of suction in modelling soil layers – System B](image2.png)

the moisture content is increasing at the location, where the sensors in our practical soil column tests were installed. In figure 12 (system without capillary breaking layer) you can see the desiccation-trend of the mineral layer, the matric suction increases at the top of the mineral layer.
6 Conclusions

In this paper, the new sensor SISOMOP, which can indirectly measure the soil moisture content and the temperature, is presented. As input parameters, SISOMOP measures moisture counts (dielectric values) which can be recalculated to volumetric water contents by using calibration curves. The calibration are necessary but not so time-consuming as SWCC tests which have to conduct to calculate water contents by tensiometer test results. The sensor is a practicable and efficient instrument, applicable both in-situ and in the laboratory. The water content range and the examined area of a SISOMOP sensor is greater than the commonly used tensiometers. The tolerance of SISOMOP is only about ± 1 % volumetric water content and it is almost maintenance-free.

In chapter 3, the setup and the calibration of the SISOMOP sensor were shown. Two soils were used in the preliminary tests. Their soil characteristics were also represented here.

Chapter 4 shows an application for using the sensors. In soil column tests where desiccation processes of cover lining systems of landfills were examined, the sensors measured permanently the soil moisture changes. A comparison of these test results with numerical calculations (model SUMMIT) is represented in chapter 5. The trends and values are rather similar.

Acknowledgement

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References


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Alternative Surface Covering of Landfill Using the TAUPE Sealing Control System

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1 Introduction

According to the German Federal Waste Disposal Ordinance, surface covers of landfills of the category DK II must represent so-called combination sealings. Apart from the compensation and drainage layer on the waste slope and the recultivation layer as top cover, the surface sealing consists of two sealing components fulfilling the function of a so-called long-term element and short-term element, respectively. One of the suitable long-term elements is the capillary barrier system.

In the past, a number of projects were performed to study the function of this system in large channel tests and field lysimeters. For a close-to-reality investigation of the functioning and time behavior of this long-term “capillary barrier” element outside of a lysimeter (plastic sealing below, margins), it was applied for the layout of a test field of about 20,000 m² in area on the landfill “Hintere Dollert” in Gaggenau-Oberweier, district of Rastatt, in 2002.

Upon the approval in 2003, the test field was nearly completed by autumn 2004. In early spring 2005, extensive studies shall be started in this test field and performed for three years at least. Establishment of this test field was supported financially by the state of Baden-Württemberg.

2 Test Field

2.1 Decisions Taken

The landfill ”Hintere Dollert“ has a total surface area to be sealed of about 130,000 m². Of these, 65,000 m² have already been sealed using a combination sealing according to the Technical Regulations for the Treatment of Municipal Waste (TASi).

Due to the positive experience gathered from the use of “capillary barrier” as sealing element, the district decided to have further areas sealed with such a capillary barrier as a long-term element. As far as the second sealing component is concerned, a number of alternatives are discussed.

In the test field, functioning of the sealing component “capillary barrier” shall be tested under real conditions – i.e. under the influence of landfill settings, landfill gas formation, and leachate directly from the recultivation layer – without lysimeter units or other “short-term sealing elements” impairing or preventing these effects.

To control and monitor the functioning of the sealing in the test field, a number of control measurements and control studies are envisaged. Control of the “tightness” using the sealing
control system TAUPE (French for “mole”) installed in the test field will be one of the major activities.

2.2 Layout of the Test Field

The test field covers an area of about 100 m x 200 m with a slope of 1 : 2.6 to 1 : 4.0.

Fig. 1 Location of the test field

The test field ends with the landfill cap at the top and the landfill bypass road at the bottom. The layout of the surface sealing in the test field is shown in Fig. 2.

Fig. 2 Layout of the sealing in the test field

The profiled and post-compacted landfill slope (that is already covered by an earth layer of at least 50 cm in thickness) is covered by a relief and drainage layer of 20 cm thickness (k_f value > 10^{-4} m/s) and monitoring layer of 15 cm thickness on top. This monitoring layer accommodates the lower part of the TAUPE sealing control system. On top of both layers, the capillary barrier system of the dimensions known is installed. Above, the upper layer of the sealing control system TAUPE is arranged in an underfloor
layer (15 cm thick) for reasons of soil mechanics. On top, the 1.85 m thick vegetation layer is located (lower part slightly compacted, upper part loose).

At the foot of the test field, outflows from the layer breaking the capillaries (gravel) and the capillary layer are collected separately and measured in a control building.

Inside the test field, a so-called lysimeter field (24 x 43 m) is located. Here, the TAUPE sealing control system is laid in smaller meshes and in addition in the capillary layer.

Fig. 3 Edge of an installation section in the test field with the sealing layers

3 Functioning and Installation of the Sealing Control System

3.1 Functioning of the TAUPE Sealing Control System

Non-metal materials, e.g. soil, may be classified by measuring their dielectric properties, i.e. the dielectricity constant. Per definition, the dielectricity constant of air equals 1, that of dry soil materials ranges from 2 to 6. Water has a dielectricity constant of 80 at 20°C. If water is added to the dry soil material, the dielectricity constant of the soil/water mixture rises. Thus, the soil’s water content can be determined via the dielectricity constant.

A method to determine the dielectricity constant is time-domain reflectometry (TDR). By a transmitter, a steep-flank impulse is fed into a cable. This impulse runs along the cable and is reflected at all points of disturbances, e.g. at the end of the cable. At the beginning of the line, the reflected impulse is detected and its runtime is measured. Its propagation speed depends on the dielectric properties of the material surrounding the line. If the dielectricity constant of soil is changed, e.g. by the inflow of water, the difference in the dielectricity constant also represents a disturbance and is recorded as a reflection at the beginning of the cable.

To monitor a representative measurement volume in the surrounding material, a special sensor cable consisting of three individual conductors is used. This cable acts like an antenna. The transmitter pulse is passed through a shielded cable to this sensor cable that may be up to 20 m long.
Consequently, the distance between the transmitter and the sensor cable may be up to 100 m. To cover a larger area, the supply cable is provided with multiplexers that distribute the impulse transmitted to several sensor cables. Calibration yields the relationship between the runtime measured and the water content to be determined. Evaluation of the reflections along the cable additionally allows for a local resolution. Consequently, also local moisture changes can be detected.

3.2 Installation of the Sealing Control System

The sensor cables are arranged in the supporting layer and under floor. Both layers have a thickness of about 15 cm and consist of the same silty sand (Su). In the limited area of the lysimeter, an additional layer of sensor cables is laid into the capillary layer.

For this, the respective layers are manufactured completely. Then, cable trenches of about 10 x 10 cm are dug out manually according to the arrangement plan. The pre-confectioned sensors are placed into the cable trenches. These trenches are then backfilled and slightly compacted. During backfilling, stones and disturbing materials in the range of the sensor cables are removed.
The coaxial cables connected to the sensor cables are bundled and stored as a cable bundle at a pile driven into the soil. Here, the cables are passed upwards through the top layers. After the application of root soil, the piles are withdrawn. Following the application of the complete root soil, the multiplexers are installed in the range of the cable bundles at a depth of about 0.8 m. The connections of the multiplexers to the central data acquisition units are also arranged about 0.8 m deep in the vegetation layer. Thus, the multiplexers and connection cables are protected from mechanical damage.

3.3 Integration in Construction Work

Prior to the laying of the sensor cables, about 0.5 ha of supporting layer and root soil are applied, planed, and compacted. Then, a day is needed for laying the sensors on the area. After another day to set the sensor cables and carry out control measurements, the layer may be covered. On both days, the next step of sensor laying can be prepared already.

The cables are laid by the experts responsible for the TAUPE system and covered the staff of the construction company. Due to this proceeding, construction work is not delayed or inhibited, the expenditure needed for the digging and backfilling of the trenches is small. The insertion of the cables does not require any additional expenditure, as this work is carried out in parallel by the experts.
3.4 Quality Assurance

According to the existing quality assurance plan, the sensors confectioned in the cable work are tested by the manufacturer of the sealing control system prior to their delivery to the construction site. The local experts control the laying of the sensors according to the plan. Immediately after laying, the sensors are adjusted by the construction company and tested again by the manufacturer.

Fig. 8 Control measurements directly after laying the sensors

Upon the complete installation, the sealing control system is taken into operation by the manufacturer. According to the recommendations of the working group on sealing control systems, functioning of the system is checked by the detection of test leaks. Then, the system is handed over to the owner.

4 Control and Investigation Program

The control and investigation program in the test field will cover a period of three years. This program will comprise the following activities:

- Measurement of variations of the water content in the layers above and below the capillary barrier using the TAUPE system and, thus, determination of flows
- Measurement of variations of the water content in the capillary layer by means of the TAUPE system
- Control measurements of variations of the water content with neutron probes
- Measurement of the outflows from the test field by a continuous outflow measurement system
- Balancing of the water resources
- Measurement of gas emissions and gas migrations by surface control, gas probes, and “gas bells”
- Investigation of the modification of sealing components by excavations and materials studies

The resulting measurements and balances will be used to adapt the water resources model such that future modeling of the water resources in the sealing system can be improved.
First measurements to determine the state of completion have already been performed. The program proper will start in spring 2005 after the complete vegetation layer will have been applied.

5 Summary and Outlook

The measurement program presented above shall contribute to verifying the suitability of the capillary barrier as a long-term sealing element within the next three years. Prior control of an alternative sealing element, possibly for the later completion of the multi-barrier system, is a challenge for the TAUPE sealing control system.

At the same time, it will be possible to verify the requirements made in the recommendations by the Federal Authority for Materials Research and Testing (BAM): Leaks have to be detected with an accuracy of < 100 m² and variations of the water content in the sealing with an accuracy of a power of 10. Functioning of the TAUPE system has already been tested successfully on smaller test fields of landfills and in a lysimeter on the premises of Forschungszentrum Karlsruhe.

It is additionally aimed at confirming that the TAUPE system allows for the qualitative and quantitative estimation of water inflow through the sealing. This information will enable the landfill operator to estimate the damage potential caused by a leak detected and to take suitable counter measures. The operator remains the acting person, and not the measurement device.

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Measurement Method for Detection of Moisture Profiles in a Saline Environment

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ABSTRACT. Hazardous and non-recyclable wastes are permanently disposed of in salt mines. The measurement of moisture distribution in gate structures designed to be kept free of monitoring after site closure is stringently necessary for the indefinable trend of fluid access and for data recording of long-term security analyses. The aim of the project is to create a dielectric moisture measurement system using flexible electrodes. Electromagnetic field simulation of both the sensor and the surrounding material helps to optimise the cable sensor. By using TDR, it was possible to detect the moisture distribution along a cable sensor. This was located in a trough and surrounded by materials of varying moisture content. For further investigations the cable sensor is then enclosed by compacted bentonite in a pressure test stand. The results of simulations and experiments will be presented.

Keywords: Waste, moisture profiles, TDR, saline environment

1 Introduction

The measurement of moisture distribution in gate structures designed for free monitoring after site closure is stringently necessary for the indefinable trend of fluid ingress and for data recording of long-term security analyses. Because of the absence of long-term experiences measurement control under the influence of disturbances is imperative over a long period of observation in test barriers. Measurement monitoring not only will be required for quality assurance during the final building inspections but also it will determine the discharge of the monitoring period and shorten it essentially. Measurements showed for this application that the accuracy and reliability of most moisture measurement methods are limited or even unacceptable [1, 3]. A time domain reflectometry (TDR) moisture measurement system using flexible electrodes will be presented here for bentonite-sealing material in a saline environment. Electromagnetic field simulation of the TDR-sensor characteristic under consideration of the surrounding material will help to optimize the cable sensor to increase the accuracy and reliability of the measurement method. By application of the TDR-cable sensor it was possible to record moisture profiles in a trough with different moist chambers. In addition the moisture profile in a small bentonite layer could be determined during measurements in a compression test stand at pressures > 40 bar.

2 TDR-Measurement Method

The propagation of an electromagnetic wave in a cable is characterised by the propagation constant $\gamma$ which is determined by the attenuation constant $\alpha$ and the phase constant $\beta$. The Eq. 1 shows the propagation constant dependent on the cable parameters:

$$\gamma = \alpha + j\beta = \sqrt{(R+j\omega L)(G+j\omega C)} \approx \sqrt{j\omega L(G'+j\omega C')}$$

(1)
The travel time \( t \) of a pulse along a cable \( l \) has the following form,

\[
t = \frac{2l}{c} \sqrt{\frac{\epsilon_r}{2} \left[ 1 + \left(1 + \tan^2 \delta \right) \right]^{1/2}}
\]  

(2)

it depends on the relative permittivity \( \epsilon_r \) and the loss factor \( \tan \delta \) of the surrounding material. In Figure 2 the TDR-waveform of a 1m flat band cable is represented for different moist materials.

![TDR-waveform of a 1m flat band cable embedded in different materials (air, moist sand, water) as a function of pulse travel time](image)

In the case of a zero loss line the losses of surrounding material influence the values of capacity and conductivity. The loss factor will be characterised by the \( \tan \delta \):

\[
\tan \delta = \frac{\epsilon_r'(\omega) + \frac{\sigma}{\omega_0 \epsilon_0 \epsilon_r}}{\epsilon_r'(\omega)}
\]

\[
\tan \delta = \frac{\sigma}{\omega_0 \epsilon_0 \epsilon_r} \quad \text{for frequencies } < 3 \text{ GHz}
\]

\[
\tan \delta = \frac{\epsilon_r'(\omega)}{\epsilon_r'(\omega)} \quad \text{for frequencies } > 3 \text{ GHz}
\]

(3)

where \( \sigma = \text{conductivity} \)
3 Calibration Methods

3.1 Experimental Method

In order to quantify the pulse travel time versus moisture content (MC) an experimental setup was used consisting of a cable tester with a rise time of 200 ps, a cable sensor, and a computer. The cable sensor was embedded in a bentonite-sand mixture with different moisture contents (c.f. Fig. 3) to record the pulse travel time dependent on MC.

![Calibration curve of the measured pulse travel time versus MC at Calcigel-sand mixtures and at a MX-80 mixture](image)

Fig. 3 Measured pulse travel time of a Calcigel-sand mixture as a function of MC as parameter

![Calibration curve of the measured pulse travel time versus MC at Calcigel-sand mixtures and at a MX-80 mixture](image)

Fig. 4 Calibration curve of the measured pulse travel time versus MC at Calcigel-sand mixtures and at a MX-80 mixture

The measured pulse travel time versus MC at Calcigel-sand mixtures and at a MX-80 mixture is represented in the calibration curves (s. Fig. 4).
3.2 Simulation

The following method was carried out for calibration of TDR-sensors using HFSS. The band cable sensor has been designed on the HFSS-Modeler. It was embedded in a cylinder which could be filled with different materials. The dielectric material properties of bentonite of different MC ($\varepsilon_r$; tan $\delta$) has been measured with a network analyser. These frequency dependent material properties has been used as input data for the HFSS-Material-Manager. Because of the wide spectrum of the TDR-pulse the simulation was made in the frequency range from 1 MHz – 12.5 GHz. The calculation was performed for each value of MC.

![Fig. 5 Simulated step responses for a Calcigel-sand mixture of different MC](image)

In Fig. 5 the simulated step responses for a Calcigel-sand mixture of different MC are represented. The recorded calibration curves were the same as received during the experiments.

4 Moisture Profiles

![Fig. 6 Trough with cable sensor and chambers of different dielectrics](image)
A trough subdivided in chambers was used to investigate moisture profiles.

Fig. 7 Change of pulse form along the trough; in each case one other chamber has been filled with dry sand.

The change of pulse form during the measurements along the trough will be shown in Fig. 7. The chambers 1 – 4 have been filled one after the other with dry sand. In all four cases the change of pulse travel time is constant, it results only from one chamber.

Fig. 8 Beginning at the cable end the chambers has been filled with dry sand.

The change of pulse form results in Fig. 8 from the filling of chambers. Thus, the pulse travel time is determined by the number of filled chambers.
Fig. 9 Trough with chamber divisions; the middle chamber has been filled with sand of different moisture content.

In Fig. 9 the change of pulsform results from the filling of the middle chamber with sand of 0.1%; 5.6% and 11.6% MC. With increasing MC the pulse travel times increase and the amplitudes of the step responses decrease. The resulting pulse form represented in Fig. 10 is caused by chambers of different MC (2 chambers blank; 2 ch. 0.55% MC; 2 ch. 15.8% MC; 2 ch. 30.4% MC).

Fig. 10 Pulse form dependent on filling the chambers with material of different MC
5 Change of Pulse Travel Time by the Influence of Density

The following experiments with a 2 m cable sensor were carried out to investigate the influence of density on the pulse travel time at a constant MC.

Fig. 11 Measuring through with divisions of blank chambers and material chamber

Density changes lead at constant MC to the increasing of pulse travel time (s. Fig. 12). In comparison it is shown the step response in air. An increasing density results in a compression of water paths and is connected by an increasing permittivity.

Fig. 12a TDR-pulse form dependent on different densities where $t_1$ = pulse travel time in the blank chamber 1; $t_{11}$ = pulse travel time of the 1. reflection of pulse edge 1; $t_{12}$ = pulse travel time of the 2. reflection of pulse edge 1; $t_{2A}$, $t_{2B}$, $t_{2C}$ = pulse travel time in the material chamber with the densities A, B, C (chamber 2 blank).
The following approximations for the reflection of the 1. pulse edge can be deduced from the measuring values with:

\[ t_1 \cong t_{11} \cong t_{12} \cong 2.83 \text{ ns} \]

Fig. 12b Pulse form and pulse travel times dependent on different densities
where \( t_{12} = 7.47 \text{ ns} \) – pulse travel time in the blank measuring through; \( t_{34} = 1.56 \text{ ns} \) pulse travel time in the blank material chamber; \( t_{12A} = 8.99 \text{ ns} \) - pulse travel time of the through by a filled material chamber with a density \( A = 1.32 \text{ g/cm}^3 \); \( t_{12B} = 9.40 \text{ ns} \) - pulse travel time of the through by a filled material chamber with a density \( B = 1.49 \text{ g/cm}^3 \); \( t_{12C} \) - pulse travel time of the through by a filled material chamber with a density \( C = 1.66 \text{ g/cm}^3 \)

The values of the pulse travel times could be measured in the blank chambers:

\[ t_{2\text{leer}} \cong t_{2A} \cong t_{2B} \cong t_{2C} \]

The following approximations results from Fig. 12b:

\[ \Delta t_{2A} = t_{12A} - t_{12\text{leer}} \]  \hspace{1cm} (8.3)
\[ \Delta t_{2B} = t_{12B} - t_{12\text{leer}} \]  \hspace{1cm} (8.4)
\[ \Delta t_{2C} = t_{12C} - t_{12\text{leer}} \]  \hspace{1cm} (8.5)
\[ \Delta t_{4A} \cong \Delta t_{2A} ; \hspace{0.5cm} \Delta t_{4B} \cong \Delta t_{2B} ; \hspace{0.5cm} \Delta t_{4C} \cong \Delta t_{2C} \]  \hspace{1cm} (8.6)
7 Determination of Moisture Profiles at Compressions up to 40 bar

Investigations influenced by pressure are necessary to simulate the conditions in the depth of a mine. A compression test stand (Fig. 13) was used to detect moisture profiles and to investigate the sealing of bentonite. The schematic representation shows the material layers and the embedding of the cable sensor. The experiments were carried out with pressures up to 40 bar. The small progress of the moisture front in the bentonite was detected using TDR with a cable sensor. The measuring unit was developed for bentonite-sand-mixtures and salt solutions to investigate the long time behaviour. The cylinder has a length of 50/100 cm and a diameter of 15 cm. Three compression sensors were installed in the outer polycarbonate cylinder to measure the pressure in the bentonite layer.

Fig. 13 Detection of moisture profiles in bentonite using a compression test stand; left the schematic representation of filling

The pressure vessel which is open at the upper end, is filled from the bottom with deionised water or salt solution. The schematic representation shows the material layers and the embedding of the cable sensor. The experiments were carried out at pressures up to 40 bar.

The gradual progress of the moisture front in the bentonite was detected using TDR with a cable sensor. The truncation of locations in bentonite layers can be determined using reversal points in the pulse form (Fig. 14). The pressure is active at the end of the cable in the first instance. The step response starts at the measuring point MP1 at the beginning of the cable connector. The voltage is increased up to the beginning of the upper sand layer 1 and then remains relatively constant. The increase of \( \varepsilon_r' \) is connected with the increase of pulse travel time, and the drop in voltage is shown over the bentonite layer between MP3 and MP4. Thereafter that the voltage is increased up to the end of the cable MP2.
Figure 14 indicates the progress of the moisture profile in bentonite as a result of many TDR measurements over 200 days. The upper curve shows the initial state and the bottom curve the final state of measurement. The water comes from the pump (Fig. 13) at the end of the cable sensor (MP2), and penetrates the sand layer very quickly (1 day) up to the bentonite-sand2-boundary layer (MP4). The water can penetrate into the bentonite only very slowly. The bentonite swells with the injected water and produces a sealing effect (measuring time 2 months). The pressure is increased in steps up to 40 bar. Higher pressures produce better barrier properties in bentonite. The dot series at MP5 show the moisture profile first in the sand layer and then, after penetrating the boundary MP4, that in the bentonite layer. The widening of the group of curves behind the moisture front between MP4 and MP3 results from density changes and not from water leakage along the cable sensor. Salt solutions were used to get practical knowledge for applications. The swelling pressure of bentonite is decreased and the front of the solution penetrates the bentonite layer faster than that from using water.

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Session 10

Multi-parameter Measurements for Determination of Properties such as Conductivity, Moisture, Density, etc.
Chairmen: S.O. Nelson, S. Okamura

1 Density-independent Moisture Measurements in Polymer Powders Using Millimeter Wave Quasi-optical Resonator
   B.Kapilevich and B.Litvak; Ariel, Israel; V.Wainstein and D.Moshe; Tel Aviv, Israel

2 Granular and Powdered Material Permittivity-density Relationships
   S. O. Nelson; Athens, Georgia, USA

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4 Comparing Near-Field and Far-Field Dielectric Properties Measurements for Accuracy of Bulk Density and Moisture Content Determination in Grain
   S. Trabelsi and S. O. Nelson; Athens, Georgia, USA
Density-independent Moisture Measurements in Polymer Powders Using Millimeter Wave Quasi-optical Resonator

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ABSTRACT. A quasi-optical resonator is implemented to characterize low loss polymer powders at mm waves. Based on suggested resonator configuration, density-independent moisture measurements have been carried out in W-band. The resonant frequencies and Q factor were measured to reconstruct real and imaginary parts of a dielectric constant to be used for density and moisture determinations. The model needed to describe this quasi-optical resonator including the reconstructing algorithm has also been developed. Examples illustrating the method proposed are reported.

Keywords: mm-wave measurements, resonators, moisture, powders

1 Introduction

Since many polymer powders being in the natural (not compressed state) have a low loss tangent (10^{-4} or less) and low density, an attenuation of propagating wave becomes too small at microwaves conventionally employed for moisture measurements. Therefore, it is reasonable to develop moisture metering technique at mm-wave range. A shift toward mm-waves allows increasing accuracy of moisture and density determinations due to higher attenuation and better resolution when free-space technique is used. Density-independent moisture method [1,2] is an efficient approach for improving accuracy of the moisture content determinations of variety materials such as solids, grain, sands, tobacco etc. at microwaves. Extension of this method toward mm-waves is considered in the paper.

We have suggested employing a section of overmoded rectangular waveguide filled with powders. Waveguide is closed by a metallic plate (short) on its output port and the array of wire grids operating as a coupler on its input. Such a configuration forms short-circuited quasi-optical resonator that is excited by horn antenna. Measured reflection coefficient data are downloaded to PC via GPIB cable and employed then for a determination of real and imaginary parts of powder’s dielectric constant using proper reconstructing procedure. Since both the real and imaginary parts of complex permittivity are obtained from the same measured data, density invariant moisture function $A(\psi)=(\epsilon’-1)/\epsilon’$ [2] can be directly determined.

The paper presents the model describing a behavior of a quasi-optical resonator filled with powder. The model links an input reflection coefficient with attenuation constant and power reflectance of the wire grid coupler. Based on this model reconstructing algorithm has been realized. Effects of asymmetric shape of the resonance curve are discussed. Since the reconstructing procedure involves solution of non-linear equations, its stability has also been investigated. Experimental setup and its calibrations are considered. Examples of moisture measurement of low loss polymer powder at W-band are reported.
2 Description of Experimental Setup

The low loss powder characterization has been performed using experimental setup shown in Fig. 1a. The section of short-circuited overmoded guide is filled with a powder under test. Polarizing wire grid at the input of this section is used as a coupling element. It has a large power reflectance, about 0.9. As a result, the short-circuited quasi-optical resonator filled with powder is formed.

![Experimental setup](image)

Fig. 1 Experimental setup (a) and equivalent circuit (b) of quasi-optical resonator.

The horn antenna connected with W-band Network Analyzer excites the resonator. Measured reflection coefficient data are transmitted to PC and stored for a further processing. Frequency synthesizer Agilent-83623B being as a part of Agilent-8757D Network Analyzer provides frequency resolution 1 MHz and up to 1600 sampling points within a fixed sweeping range. It is sufficient to observe rather narrow resonance curves of low loss materials under test.
3 Description of the Model

A quasi-optical resonator used for characterization of powder at mm-waves can be presented by its equivalent circuit depicted in Fig 1b, where \( Z_{mn} \) is the impedance of a resonating waveguide mode \( \text{TE}_{mn} \), \( \alpha_{mn} \) is an attenuation constant; \( \beta_{mn} = 2\pi f_{mn}/c \); \( m \), \( n \) are integer numbers. The resonance frequencies \( f_{mn} \) of ideal resonator can be calculated [3, p.430] using formula:

\[
f_{mn} = \frac{0.3}{\sqrt{\varepsilon_r}} \sqrt{\frac{2l}{\lambda_c}} \left( p^2 + 1 \right), \text{ GHz}
\]  

(1)

where \( \varepsilon_r \) is relative dielectric constant of medium filled a resonator, \( \lambda_c \) is critical wavelength, \( p = 1, 2, \ldots \) are integer numbers, \( l \) is the length of resonator. Input coupling element causes a shift of a resonance frequency which is not easy to predict at mm waves due to complexity of coupler. As a result, uncertainty in identification of true values of \( m, n \) and \( p \) may occur in a practice. The procedure discussed below permits to avoid this undesirable effect.

Using polarizing properties of grid’s coupler we can provide an excitation of some selected modes which demonstrate a regular distribution of resonant frequencies as a function of longitudinal number \( p \). In this case, the resonance frequency, \( f_{mn(p+1)} \) corresponding to the next number \( p+1 \) is written as:

\[
f_{mn(p+1)} = \frac{0.3}{\sqrt{\varepsilon_r}} \sqrt{\frac{2l}{\lambda_c}} \left( (p+1)^2 \right), \text{ GHz}
\]  

(2)

Assuming that both frequencies \( f_{mn} \) and \( f_{mn(p+1)} \) are known from measurements we can write the following equation derived from (1) and (2) that determines longitudinal number \( p \):

\[
r = \frac{q + (p + 1)^2}{q + p^2}
\]  

(3)

where: \( r = (f_{mn(p+1)}/f_{mn})^2 \) and \( q = (2l/\lambda_c)^2 \). The solution of (3) yields the two values of \( p \)

\[
p_{1,2} = \frac{1 \pm \sqrt{r - q + 2rq - qr^2}}{r - 1}
\]  

(4)

There is no universal rule for distinguishing these roots formally. However, in many practical situations associated with quasi-optical resonators \( p_1 >> p_2 \) and the first solution should be used for reconstructing \( \varepsilon_r \) from measured data resulting in the following expression:

\[
\varepsilon_r = \left( \frac{0.3}{f_{mn}} \right)^2 \left( \frac{1}{\lambda_c} \right)^2 + \left( \frac{p_1}{2l} \right)^2
\]  

(5)

In order to reconstruct dielectric loss associated with \( \varepsilon'' \) from the measured data it is necessary to evaluate behavior of the reflection coefficient in vicinity of resonance frequency of the specified mode. Let’s assume that the grid’s coupler can be characterized by power
reflectance $R_p$. It can be derived that the input reflection coefficient of the shorted resonator shown in Fig. 1 is written as follows [4]:

$$\Gamma(R_p, \alpha, f) = \frac{(1-2i\sqrt{R_p}) \tanh(\alpha + i\beta)l - 1}{(1+2i\sqrt{R_p}) \tanh(\alpha + i\beta)l + 1}$$  \hspace{1cm} (6)

It should be pointed out that (6) is valid for any loss and can be used for direct reconstruction of $\varepsilon''$ from measured values of $\Gamma(R_p, \alpha, f)$.

### 4 Reconstructing Algorithm

To apply the procedure for reconstructing $\varepsilon''$, the two measurements of $\Gamma$ must be done at some specified frequencies, namely, $\Gamma(R_p, \alpha, f_0)$ and $\Gamma(R_p, \alpha, f_1)$, where $f_0$ is the resonance frequency and $f_1$ is an arbitrary frequency located just near the resonance one. In standard measurements the $f_1$ corresponds to a 3 dB level assuming that the resonance curve has symmetrical shape relatively its resonance frequency. Since, the measured resonance curve may be asymmetric we do not use the 3dB criterion. If the measured values $A_0$ and $A_1$, correspond to frequencies $f_0$ and $f_1$, the following system of two nonlinear equations can be written for unknown $R_p$ and $\alpha$:

$$\Gamma(R_p, \alpha, f_0) = A_0$$
$$\Gamma(R_p, \alpha, f_1) = A_1$$  \hspace{1cm} (7)

The system (7) can be solved numerically. In practice, the resonance curve becomes asymmetrical due to excitation of parasitic modes, and measurements of a reflection coefficient must be done on both sides of the resonance curve (below and above of the resonance frequency). The value of the frequency detuning $Af = f_1 - f_2$ depends on asymmetry of a resonance curve. The real trade-off corresponds to almost the same resonator parameters determined for both detuned frequencies $f_1$ and $f_2$. The averaged value of $R_p$ and $\alpha$ estimated for these detuned frequencies are used later as a final result of the measurement carried out. It should be pointed out that the basic assumptions used in the model formulations are:

- the resonant frequency of the selected mode is determined by a real part of dielectric constant;
- the width of resonant curve of the same mode is determined by imaginary part of dielectric constant.

Hence, strictly speaking, the above assumptions are valid for low loss powders which are the subject of this paper.

### 5 Calibration of Experimental Setup

In measurements described below we used a section of overmoded rectangular guide with dimensions 10.5x25x189.8 mm$^3$ fabricated from Al-alloy. The input coupler was as an array of one dimension wire grids with the period 0.5mm and wire diameter 0.02mm resulting in power reflection of about 0.9 that was sufficient to form a quasi-optical resonator. To provide
an excitation of quasi-T$_{01p}$ mode the wires were oriented parallel to the wall having a width of 25 mm. Agilent-8757D Network Analyzer was employed for measurement of input reflection coefficient. Its output was connected with a rectangular horn antenna illuminating the grid coupler. The distance between them was adjusted to provide the best suppression of undesirable modes. First of all, the calibration of experimental setup must be done in order to estimate unloaded $Q_{\text{metal}}$ – factor of quasi-optical resonator associated with loss energy in metal walls. Such a calibration has been performed for the air-filled resonator.

Typical measured reflectance (return loss in dB) as a function of frequency corresponding to air filled resonator is shown in Fig. 2. There are several observable resonances within measured frequency range 97 –100 GHz with averaged frequency shift between the two nearest resonances $\Delta f = 0.7725$ GHz. Lets select from Fig. 2 value $f_1 = 97.33$ GHz as a resonance frequency of the selected mode with index $p$ and $f_2 = f_1 + \Delta f = 98.1025$ GHz as a resonance frequency of the nearest mode with index $p+1$. From (4) we can calculate the two values of $p$: $p_1 = 122.37$ and $p_2 = 2.161$. The first one after rounding to nearest integer number $p_1 \approx 122$ corresponds to real experiment conditions and should be used for reconstructing dielectric constant while the second one must be neglected. Based on (5) we can determine relative dielectric constant of a medium filled with the quasi-optical resonator, $\varepsilon_r = 1.01$. This result is very close to the air's dielectric constant proving that the assumptions introduced into the model were valid.

![Frequency response of air-filled quasi-optical resonator](image)

**Fig. 2** Measured return loss of air-filled quasi-optical resonator as a function of frequency.

To estimate unloaded $Q_{\text{metal}}$ it is necessary to consider (6) and (7). The solution of (7) at the resonance frequency $f_2$ yields $R_p = 0.89$ and $\alpha = 0.0526$ (1/m). Now, we can calculate resulting $Q_{\text{metal}}$ using formula $Q = \beta / 2 \alpha$ [3, p.420]. In our case we have determined that $Q_{\text{metal}} = 19520$. Then, we can employ (6) to validate an accuracy of reconstructing procedure. Figure 3 shows return losses $RL$ in dB measured (dotted line) and reconstructed (solid line) from (6) for the calibrated resonator. Neglecting excitation of parasitic modes we can state that reconstructing procedure works rather effectively.
Fig. 3 Measured (dotted line) and reconstructed from (6) (solid line) values of return losses \( RL \text{ (dB)} \) for the calibrated resonator.

The following range of guess values providing the same solutions of the system (7) have been determined: 0.01 < \( \alpha < 0.5 \) [1/m] and 0.8 < \( Rp < 0.99 \). So that, a stability of reconstructing procedure is quite well.

6 Low Loss Powder Characterization

The same resonator as described in section 5 has been filled with low loss polymer powder (Fluoropolymer Resine). Its measured reflectance (return loss in dB) is depicted in Fig. 4. Now we can reconstruct both real and imaginary parts of complex permittivity the powder under test.

Reconstructing real part of complex permittivity: Repeating calculations of the section 5 we have determined from measured data, Fig. 4:

- averaged frequency shift between the two nearest resonances is \( \Delta f = 0.685 \text{ GHz} \);
- index \( p_1 \) corresponding to the given experiment conditions is \( p_1 = 140 \) at the resonance frequency \( f = 97.44 \text{ GHz} \);
- real part of the dielectric constant reconstructed from (5) is \( \varepsilon_r = 1.286 \) that corresponds to density \( d = 0.8 \text{g/cm}^3 \).

It should be pointed out that a real part of dielectric constant is a function of powder density and may vary in a wide range depending on a compressed powder state.

Reconstructing imaginary part of complex permittivity: At first, it is necessary to estimate the unloaded \( Q_{\text{filled}} \) -factor of the resonator filled with a powder using well known expression:
\[
\frac{1}{Q_{\text{filled}}} = \frac{1}{Q_{\text{metal}}} + \frac{1}{Q_{\text{powder}}}
\]  

Fig. 4 Measured return loss of quasi-optical resonator filled with powder as a function of frequency.

Fig. 5 Measured (dotted line) and reconstructed (solid line) from (6) values of return loss $RL$ (dB) for the resonator filled with powder.

For $\epsilon_r$ determined above the following parameters were found out at the resonance frequency 98.13 GHz: $R_p = 0.863$, and $\alpha = 0.0688$ (1/m) resulting in $Q_{\text{filled}} = 16920$. The $Q_{\text{powder}}$
determined from (8) is approximately 127000 that corresponds to \( \tan \delta = 1/Q_{\text{powder}} \approx 0.8 \times 10^{-5} \) resulting \( \varepsilon_r = 1.013 \times 10^5 \).

Figure 5 demonstrates both measured result (dotted line) and reconstructed from (6) return losses \( RL \, dB \) (solid line) for the resonator filled with the measured polymer powder. Calculation has been made for the resonance mode located near frequency 98.13 GHz. We can see that measured and reconstructed curves are rather close to each other proving an efficiency of the method proposed.

Based on the above measurements we can determine density invariant moisture function \( A(\psi) = (\varepsilon' - 1) / \varepsilon'' \) for the given material, namely, \( A(\psi) \approx 2.8 \times 10^4 \). Other results are presented in the Table 1 for different compressed state (density) of the powder under testing.

Table 1 Some results of measurements density independent moisture \( \psi \% \)

<table>
<thead>
<tr>
<th>( d , [g/cm^3] )</th>
<th>0.8</th>
<th>0.85</th>
<th>0.9</th>
<th>0.95</th>
<th>1.0</th>
<th>1.05</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \varepsilon_r )</td>
<td>1.286</td>
<td>1.321</td>
<td>1.34</td>
<td>1.379</td>
<td>1.42</td>
<td>1.46</td>
</tr>
<tr>
<td>( 10^5 \varepsilon_r )</td>
<td>1.013</td>
<td>1.14</td>
<td>1.21</td>
<td>1.36</td>
<td>1.51</td>
<td>1.62</td>
</tr>
<tr>
<td>( \psi % )</td>
<td>0.49</td>
<td>0.5</td>
<td>0.51</td>
<td>0.5</td>
<td>0.52</td>
<td>0.51</td>
</tr>
</tbody>
</table>

7 Conclusion

In this paper, we have suggested density-independent moisture method base on an application of mm-wave quasi-optical resonator. Measurements of real and imaginary parts of dielectric constant of low loss powders filling this resonator can be easily performed. The model required for doing the reconstruction procedure has been suggested. Examples illustrating measurement technique have validated the basic assumptions used in the method developed.

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Granular and Powdered Material Permittivity-density Relationships

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ABSTRACT. When knowledge of the dielectric properties, or permittivity, of granular or powdered materials is important, as in dielectric heating applications or in the sensing of moisture content by radio-frequency or microwave instruments, some reliable relationship between the dielectric properties and the bulk density of the material is required. Linear relationships between functions of the permittivity and bulk density have been observed, and dielectric mixture equations have been explored to obtain useful relationships for determining the dielectric properties of granular and powdered materials. These findings are reviewed, useful permittivity-density relationships are identified, and an equation is provided that permits reliable calculation of the permittivity of a particulate material at any given bulk density, if the permittivity is known or measured at another known bulk density. The relationships are illustrated for pulverized coal, wheat, and ground wheat. The useful mixture equation is valid for granular and powdered materials with permittivities in the same range as those for grain and coal.

Keyword: permittivity, bulk density, granular materials, powdered materials

1 Introduction

When the permittivity, or dielectric properties, of granular or powdered solid materials are important, some reliable relationship between the permittivity and the bulk density of the air-particle mixture is required. Examples of such applications include radio-frequency or microwave dielectric heating of these materials and the use of correlations between permittivity and water content of hygroscopic materials for sensing moisture content. Also, relationships between the permittivities of pulverized or granular samples of materials and those of the solid materials have long been of interest for use in determining the permittivities of the solids from measured permittivities of the particulate samples. Dielectric mixture equations have been developed and investigated for this purpose [1-3]. An extrapolation procedure, based on the linearity with density of functions of the real and imaginary parts of the permittivity of pulverized and granular materials, has also been used to obtain estimates of the permittivity of the solid material [4,5]. Both of these methods, calculation by dielectric mixture equations and the extrapolation procedure, will be described in the following sections for estimating the permittivity of solids from measured permittivity data on granular or powdered samples of those materials.

2 Dielectric Mixture Equations

Many different dielectric mixture equations have been proposed to represent the effective permittivity of mixtures of dielectric materials, and the characteristics and performance of these equations for various applications have been discussed in the literature [6-11]. The complex permittivity relative to free space is represented here as \( \epsilon = \epsilon' - j\epsilon'' \), where \( \epsilon' \) is the dielectric constant and \( \epsilon'' \) is the dielectric loss factor. Dielectric mixture equations for a two-phase mixture can be used to calculate the dielectric properties of a solid material from the dielectric properties of an air-particle mixture of air and pulverized or granular particles of the solid. To use the mixture equations, one needs to know the permittivity of the pulverized
sample at its bulk density (air-particle mixture density), \( \rho \), and the specific gravity or density of the solid material, \( \rho_2 \). The fractional part of the total volume of the mixture occupied by the particles (volume fraction), \( v_2 \), is then given by \( \rho / \rho_2 \).

Several well-known dielectric mixture equations have been studied to relate the permittivities of the solid material to the permittivities of granular or pulverized samples of the same material [5,12-15]. Two of these equations are the Complex Refractive Index mixture equation [16-22],

\[
(\varepsilon)^{1/2} = v_1(\varepsilon_1)^{1/2} + v_2(\varepsilon_2)^{1/2}
\]

and the Landau & Lifshitz, Looyenga equation [23,24],

\[
(\varepsilon)^{1/3} = v_1(\varepsilon_1)^{1/3} + v_2(\varepsilon_2)^{1/3}
\]

where \( \varepsilon \) represents the complex permittivity of the mixture, \( \varepsilon_1 \) is the permittivity of the medium in which particles of permittivity \( \varepsilon_2 \) are dispersed, and \( v_1 \) and \( v_2 \) are the volume fractions of the respective components, where \( v_1 + v_2 = 1 \).

Each of these equations can be used to calculate the complex permittivity of the solid material by substituting \( I - j \theta \) for \( \varepsilon_1 \), the complex permittivity of air, and solving for \( \varepsilon_2 \). The corresponding equations are:

Complex Refractive Index (1)

\[
\varepsilon_2 = \left( \frac{\sqrt{\varepsilon + v_2 - I}}{v_2} \right)^2
\]

Landau & Lifshitz, Looyenga (2)

\[
\varepsilon_2 = \left( \frac{\varepsilon^{1/3} + v_2 - I}{v_2} \right)^3
\]

3 Linear Extrapolation Technique

Essentially linear relationships between functions of the real and imaginary components of the complex permittivity of particulate materials such as pulverized coal, wheat, and whole-wheat flour and their bulk densities were identified previously [4,25,26]. These findings were based on earlier work in which Klein [27] observed the linearity of \( \sqrt{\varepsilon'} \) with the bulk density of granular coal, and in which Kent [28], working with fish meal, found that \( \varepsilon' \) and \( \varepsilon'' \) were both quadratic functions of the bulk density of the fish meal as follows:

\[
\varepsilon' = a \rho^2 + b \rho + l
\]

\[
\varepsilon'' = c \rho^2 + d \rho
\]

where \( \rho \) represents the density of the air-particle mixture, \( a, b, c \) and \( d \) are constants for a given particulate material and \( \varepsilon' \) and \( \varepsilon'' \) have values of \( l \) and \( \theta \), respectively, for air alone.
(ρ = 0). Equations (5) and (6) are equivalent to expressing the relative complex permittivity as a quadratic function of bulk density, \( \varepsilon = a^* ρ^2 + b^* ρ + I \), where the constants \( a^* = a - je \) and \( b^* = b - jd \) are complex numbers.

The linearity of \((\varepsilon')^{1/2}\) with bulk density was confirmed with data on pulverized coal (Fig. 1) [25] and this relationship is expressed as

\[
(\varepsilon')^{1/2} = m\rho + I
\]

Fig. 1 Linear relationships between the square root of the dielectric constant of pulverized coal samples and the bulk density measured at indicated frequencies [25].

Equations (5) and (7) are equivalent if \( a = m^2 \) and \( b = 2m \). Thus, measurement of the dielectric constant of a particulate material at one bulk density, along with the \((\rho = 0, \varepsilon' = 1)\) intercept, provides information on the dielectric constant at all densities, including that of the solid material if its density is known. Examining the expression for the loss factor (6), the square can be completed by adding a constant, \( e \), to each side, and a linear function is obtained

\[
(\varepsilon'' + e)^{1/2} = c^{1/2} \rho + e^{1/2}
\]

where \( e = d^2 / 4c \). Thus, to describe the density dependence of the complex permittivity of a particulate material, one needs to obtain the values for \( a, b, c \), and \( d \). Measurement of the permittivity of the particulate material at a given density establishes slope \( m \) of (7), thus determining values for \( a \) and \( b \). Measurements at a few additional densities are necessary for determination of \( c \) and \( d \), as explained previously [12,29]. It is interesting to note that (5) and (6) are consistent with the Complex Refractive Index mixture equation (1), when used for the real part of the complex permittivity. Since \( v_z = \rho / \rho' \), where \( \rho \) is the density of the air-particle mixture, and \( \rho_z \) is the density of the particles, this substitution in (1) yields the following for an air-particle mixture:

\[
(\varepsilon')^{1/2} = \frac{(\varepsilon_0')^{1/2} - 1}{\rho_z} \rho + I
\]
which is equivalent to (7), where \( m = (\sqrt{\varepsilon'_2} - 1)/\rho_2 \). In an analogous manner, it can be shown that the linearity of the cube root of the dielectric constant of an air-particle mixture with its density is consistent with the Landau and Lifshitz, Looyenga mixture equation (2) [4],

\[
(\varepsilon'_2)^{1/3} = \frac{(\varepsilon'_2)^{1/3} - 1}{\rho_2} \rho + 1
\]

(10)

4 Experimental Data for Wheat

Both components of the permittivity are shown in Fig. 2 for a measurement sequence at different densities for whole-wheat flour of 10.9% moisture at 11.67 GHz [26], illustrating the quadratic behavior for both the dielectric constant and loss factor in accordance with (5) and (6). The same data are presented in Fig. 3, showing the linearity of the square roots and the cube roots of the dielectric constant with bulk density as defined in (7), (9), and (10). The linearity of the square root of the loss-factor function is also illustrated for these data.

![Dielectric constant and loss factor of whole-wheat flour of 10.9% moisture at 11.67 GHz as functions of the bulk density of the flour.](image)

Fig. 2 Dielectric constant and loss factor of whole-wheat flour of 10.9% moisture at 11.67 GHz as functions of the bulk density of the flour [26].

Results of similar sequences of measurements over a range of densities are shown in Figs. 5 and 6 for whole-wheat flour (ground wheat) and whole-kernel hard red winter wheat, respectively. These were different wheat samples and the measurements were taken at different frequencies. However, for these two different particle sizes of similar material, results showed that both the square root and cube root relationships fit the data reasonably well. The cube-root relationship, however, was a bit superior to the square-root relationship in fitting the data for both the whole-kernel wheat and the ground wheat. In these studies, the permittivity data were extrapolated to the densities of the flour and grain particle densities, as determined by air comparison pycnometer measurements [26], to obtain estimates of the permittivities of the solid materials at their densities as indicated by the vertical dashed lines in Figs. 5 and 6.
5 Discussion and Conclusions

Applications of these techniques have been discussed in detail for coal [4,5,13,25,29], wheat and wheat flour [4,14,15,26], plastics [12,13], and coal and limestone and their mixtures [30,31]. As a result of these studies, the Landau & Lifshitz, Looyenga equation generally provided the best relationship between the permittivities of the solid and particulate materials. Therefore, a relationship based on this equation can be recommended for correcting permittivities of granular and powdered materials, with permittivities in the same range as these materials, for changes in bulk density as follows:

Fig. 3 Straight-line relationship between the square root and cube root of the dielectric constant of whole-wheat flour at 10.9% moisture and 11.67 GHz and the bulk density of the flour [26]. As shown in Fig. 3, the cube-root relationship of (10) provides a slightly better fit than the square-root relationship of (9).

Fig. 4 Straight-line relationship between the function of the loss factor of whole-wheat flour of 10.9% moisture at 11.67 GHz and the bulk density of the flour. Solid line is the regression line with the \((0, \sqrt{\varepsilon})\) intercept included, and the dashed line is the regression line with the intercept excluded from the data set [26].
\[ \varepsilon_b = \left[ \left( \left( \frac{\varepsilon_a}{\rho_b / \rho_a + 1} \right)^{1/3} - 1 \right) \rho_b / \rho_a + 1 \right]^2 \]  

(11)

where \( \varepsilon_a \) is the complex permittivity of the air-particle mixture at a given density \( \rho_a \), and \( \varepsilon_b \) is the permittivity of the mixture at a different density \( \rho_b \). Use of complex calculation provides values for both the dielectric constant and loss factor.

Fig. 5 Density dependence of the dielectric constant and its square root and cube root for whole-wheat flour at 11.7 GHz and 22 °C. Solid lines are regression lines for second-order polynomial regression for the dielectric constant and linear regression for the square root and cube root. Short- and long-dashed lines for the dielectric constant are predictions from the regression of the cube root and square root of the dielectric constant, respectively [4].

Fig. 6 Density dependence of the dielectric constant and its square root and cube root for whole-kernel wheat at 9.4 GHz and 24 °C. Solid lines are regression lines for second-order polynomial regression for the dielectric constant and linear regression for the square root and cube root. Short- and long-dashed lines for the dielectric constant are predictions from the regression of the cube root and square root of the dielectric constant, respectively [4].
References


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Moisture and Ion Measurement Using Microstripline

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ABSTRACT. This paper describes a new sensor used for soil. It aims to measure the moisture content and ion concentration of soil. It is based on an idea of using a ratio of microwave signals propagating through a microstripline. In the experiment by the microwaves at 2GHz and 10GHz the idea was examined on a sample of sand. Although the measurement result of the experiment had non-negligible error, it confirmed that the new sensor had an ability of measuring the moisture content and salt concentration.

Keywords: moisture, soil, microwave, salt

1 Introduction

Measurement of moisture content and salt concentration contained in soil is very important for farming. It is necessary especially for controlling of irrigation to grow much plant with preventing the salt graining at the surface of the ground in dried region. However, there is few sensors having the ability of measuring the moisture and the concentration, and development of a new sensor is desired. If such a sensor is popular in the world, a farmland in dried region helped by irrigation will be prevented from salt damage and that will contribute to the increase of food production.

The popular sensor that measures moisture content of material containing much water is using microwaves. In this paper a transmission method was discussed to realize the sensor for the moisture content and the salt concentration measurement of soil. In the transmission method the sensor using a microstripline was studied for the measurement because it has good result in the measurement of moisture content of high moist material. The principle of the measurement of the sensor is using the ratio of the attenuation to the phase shift of the microwave transmitted through a microstripline surrounded by soil [1].

2 Measurement Method

2.1 Principle

The sensor used in this paper is made by a microstripline that detects the information about the material under test put on the line. The microwave which come into the line propagates through the line being affected by the material on the line. The output microwave from the line has the information about the material as the changes of the amplitude or the phase of the wave. The two changes are measured as the attenuation or the phase shift. In the measurement system of the sensor the two changes are combined to one signal. The signal is the ratio of the attenuation to the phase shift.

Both of the attenuation and the phase shift are affected by moisture content \( M \), ion concentration \( S \), density of material \( \rho \), and the effective sensor length \( l \). The attenuation and the phase shift denoted by \( \Delta A \) and \( \Delta \Phi \), respectively, are expressed in Eq. 1 and Eq. 2 [2].
\[ \Delta A = R_A(\rho) \cdot g_A(M) \cdot C_A(S) \cdot l \]  
\[ \Delta \Phi = R_{\Phi}(\rho) \cdot g_{\Phi}(M) \cdot C_{\Phi}(S) \cdot l \]

where \( R_A(\rho) \) and \( R_{\Phi}(\rho) \) are the functions for the attenuation and the phase shift on the density, \( g_A(M) \) and \( g_{\Phi}(M) \) are the functions for the attenuation and the phase shift on moisture content, \( C_A(S) \) and \( C_{\Phi}(S) \) are the functions for the attenuation and the phase shift on ion concentration, respectively.

The ratio of Eq. 1 to Eq. 2 is shown in Eq. 3.

\[
\frac{\Delta A}{\Delta \Phi} = \frac{R_A(\rho) \cdot g_A(M) \cdot C_A(S) \cdot l}{R_{\Phi}(\rho) \cdot g_{\Phi}(M) \cdot C_{\Phi}(S) \cdot l} = k(\rho) \cdot \varphi(M) \cdot \eta(S) 
\]

\[
k(\rho) = \frac{R_A(\rho)}{R_{\Phi}(\rho)} \quad (4)
\]

\[
\varphi(M) = \frac{g_A(M)}{g_{\Phi}(M)} \quad (5)
\]

\[
\eta(S) = \frac{C_A(S)}{C_{\Phi}(S)} \quad (6)
\]

where \( k(\rho), \varphi(M) \), and \( \eta(S) \) are the functions of the ratio on the density, moisture content, and ion concentration, respectively.

The ratio expressed in Eq. 3 has no variable for the effective sensor length of \( l \). Then the detected signals from the sensor is not affected by the length of material or roughness at the surface of the material. Also, it has less dependence to the change of density of material. The fact that the ratio is useful for the measurement of moisture content has been confirmed experimentally [3]. It was reconfirmed in this paper. It is shown in the section of 3.1.

2.2 Sample

The aim of this research is to know the possibility of measuring the moisture content and the ion concentration of soil, but sand was tested instead of soil because of the easiness in handling. The moisture content of sand on wet basis is defined as following.

\[
M = \frac{W_w}{W_d + W_w} \times 100\% 
\]

where \( W_w \) and \( W_d \) are the masses of water and dry material, respectively. The mass of perfect dried sand, i.e. 0 % sand, was measured after drying more than 8 hours at temperature of 80 degree in Celsius.

The moisture content and the ion concentration of sand were adjusted by adding tap water and salt. Moisture contents are 0, 5, 10, 15, and 20 %, and salt concentrations are 0, 1, 2, and 3 %.

2.3 Setup

The setup of the sensor is constructed with a board of microstripline, a network analyzer (Anritsu 37347C), and a personal computer as shown in Fig. 1. The microstripline board was made of a copper double side epoxy glass board. Microwave generated at the analyzer propagates through the microstripline and detected by the analyzer. The sand under test is put
on the microstripline, and the moisture and the ion contained in the sand are measured. The strip of the line is covered with polyethylene film and protected from the damage caused by the sand pressing on it. The measured data from the analyzer were processed and stored by the computer.

Fig. 1 Microstripline in the Sensor Setup

2.4 Decision of Frequency

Measurement frequency was determined from the frequency characteristics of sand, water, and salt water. The permittivity of dried sand was about 3 and has little dependence in the measured frequency. The permittivity of water and salt water was measured by a dielectric probe of a network analyzer (HP85070) and the result is shown in Fig. 2. Measured samples were tap water and the weight concentration of salt were 1, 2, and 3%.

It is seen from the Fig. 2 that there is difference between the measured values in the salt concentrations in both the real part and imaginary part. However, the difference becomes smaller in increasing of the measuring frequency and that is conspicuous in the imaginary part. The attenuation of microwave comes mainly from the imaginary part of the permittivity. From these results, the frequency used in the measurement was decided to 2 GHz and 10 GHz.

Fig. 2 Permittivity of water and salt water. The left figure is the real part and the right is the imaginary part.
3 Experiment on Sensor

3.1 Ratio of Attenuation to Phase Shift

Considering the difference in the nature of soil, the sand under test was pressed by some weights to change the density of the sand. To prevent the effects due to the difference of the size and of the density of sample sand, the method using the ratio of the attenuation to the phase shift of microwave was adopted in this research.

The sample sand contained tap water of 10% on the weight. The density of the sand was changed by pressing the top of the sand by putting some weights. The maximum weight was 8 kg and the pressed area of sand was about $7 \times 15 \text{ cm}^2$, then the maximum pressure was about 0.08 kg/cm$^2$. The influence due to the change of the pressure is seen in the attenuation in Fig. 3 and the phase shift in Fig. 4. These results express that both the attenuation and phase shift are changed easily by the difference in the nature of soil.

![Fig. 3 The influence to the attenuation due to the change of pressure.](image1)

![Fig. 4 The influence to phase shift due to the change of pressure.](image2)

On the other hand the influence due to the change of pressure was decreased by using the ratio of the attenuation to the phase shift shown in Fig. 5. It was decreased by one fifth at 10 GHz than that of the attenuation or the phase shift itself. Hence, the ratio method is used in this paper to measure moisture content and ion concentration.

![Fig. 5 The ratio of the attenuation and the phase shift](image3)
3.2 Moisture and Ion Measurement Result

The moisture content and the ion concentration containing in the sample of sand were measured by using the ratio method. The moisture content of the sample was made to 5, 10, 15, and 20%. The salt concentration on each sample was 0, 1, 2, and 3%. The ratio of the attenuation to the phase shift at 2 GHz is shown in Fig. 6, and that at 10 GHz is shown in Fig. 7.

![Fig. 6 The ratio at 2GHz](image1)
![Fig. 7 The ratio at 10 GHz](image2)

The ratio at 2 GHz in Fig.6 changed by both the moisture content and the salt concentration. Contrarily, the difference of the ratio due to the change of the salt concentration was small at 10 GHz in Fig. 7. The ratio is considered to be mainly changed only by the moisture content of the sample sand at 10 GHz.

4 Possibility of Measurement

As shown in Fig. 7, the moisture content of sand can be measured by using the microwave at 10 GHz in spite of whether salt is existing in the sand or not. Therefore, the moisture content or the salt concentration of a sample of sand under measurement can be measured by next procedure.

1. To know the moisture content of a sample, measure the ratio explained above by using the microwave at 10 GHz.
2. From the measured ratio at 10 GHz, the moisture content of the sand is predicted using a chart or a table. In this paper Fig. 7 can be used as the chart.
3. To know the salt concentration of the sample, measure the ratio by using the microwave at 2GHz.
4. From the measured ratio at 2GHz and the predicted moisture content, the salt concentration of the sand is predicted using a chart or a table. Fig. 6 can be used as the chart.
5. The moisture content and the salt concentration are the predicted values at the step 2 and 4, respectively.

To check the above procedure, the samples used in the measurement in Fig. 6 and Fig. 7 were measured and the moisture content and the salt concentration of the samples were predicted.
The results are shown in Fig. 8 and Fig. 9. The straight line in each figure corresponds to the ideal relation. The predicted results on moisture content have small error but the predicted results on salt concentration have no small error. However, the possibility of simultaneous measurement of the moisture content and the ion concentration has been confirmed.

Fig. 8 The predicted moisture content measured at 10 GHz.  
Fig. 9 The predicted salt concentration measured at 2 GHz

5 Conclusion

A new idea of a sensor for measuring moisture content and ion concentration has been presented. One of the features in the sensor is made of a microstripline operating at 2 and 10 GHz, on which a sample under test is put for the measurement. Another is the adoption of the ratio of the attenuation to the phase shift on the microwaves traveling through the line. Although the measurement result of the experiment on the sand had non-negligible error, it confirmed that the new sensor had an ability of measuring the moisture content and salt concentration. In next step of the research on the sensor more research will be needed to reduce the measurement error.

Reference:


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Comparing Near-Field and Far-Field Dielectric Properties Measurements for Accuracy of Bulk Density and Moisture Content Determination in Grain

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ABSTRACT. Near-field microwave measurements of the dielectric properties of wheat and soybeans with a pair of single-patch microstrip antennas at 5.8 GHz and 24 °C are compared to those taken in the far field with a pair of focused-beam horn/lens antennas. Effects of near-field measurements with microstrip antennas on the accuracy of determining bulk density and moisture content from measured dielectric properties are examined.

Keywords: Near field, far field, dielectric properties, microwaves, microstrip, horn/lens, bulk density, moisture content

1 Introduction

Dielectric-based microwave sensors provide versatile tools for real-time determination of physical properties of materials [1, 2]. These sensors can meet requirements of modern industries and their highly computerized environments.

However, widespread use of these sensors depends on many factors, including efficient and robust calibration methods and cost-effective designs. Both of these aspects are addressed in this paper. Several calibration methods have been proposed [3-7], and many microwave components are now available at affordable prices, which make the development of such sensors attractive both economically and technically.

Dielectric-based microwave sensors rely on measuring the two components of the relative complex permittivity, i.e., the dielectric constant \( \varepsilon' \) and the dielectric loss factor \( \varepsilon'' \), and identifying appropriate correlations between these properties and the desired physical properties of the material. Therefore, their effectiveness in sensing any physical property depends on the accuracy with which \( \varepsilon' \) and \( \varepsilon'' \) are measured and the validity of established calibration equations [8-10].

Granular and particulate materials constitute a large category of organic and inorganic materials, which are encountered in diverse industries including food and agriculture, pharmaceutical, mining and construction. Rapid characterization of these materials is important for process and quality control. The aim of this study is to examine the level of accuracy achievable in predicting bulk density and moisture content from near-field measurement of dielectric properties of granular moist materials with an inexpensive microstrip free space antenna system. The dielectric properties of wheat and soybeans measured in free space with a pair of single-patch microstrip antennas placed very close to the sample interfaces are compared to those obtained in far-field measurements with a pair of horn/lens antennas [11]. Results of bulk density and moisture content prediction from dielectric properties measured with both antenna systems are compared through computation of the standard error of calibration \( (SEC) \). Calibration equations correlating dielectric
properties with bulk density and with moisture content in wheat and soybeans are given for each antenna system along with corresponding standard error of calibration.

2 Free-Space Measurement

Among the microwave techniques available for dielectrics characterization [12], free-space measurement techniques (reflection/transmission) have the advantage of being nondestructive, they do not require contact with the sample, and very often they involve minimum sample preparation. For nonmagnetic materials such as cereal grain and oilseed, measurement of either the reflection coefficient or transmission coefficient is sufficient to determine the dielectric properties. In this study, the transmission technique was selected, because measurement of the scattering transmission coefficient \( S_{21} \) provides information relative to the whole volume of materials interacting with the incident electric field. The dielectric properties are calculated from the measured modulus, \(|S_{21}|\), and phase, \(\phi\), of \( S_{21} \) assuming a plane wave is incident on a low-loss material \((\varepsilon'' \vee \varepsilon')\) as follows [11,13]:

\[
\varepsilon' = \left[ 1 - \left( \frac{\phi - 360n}{360d} \right) \frac{c}{f} \right]^2 \\
\varepsilon'' = \frac{-20\log|S_{21}|}{8.686\pi d} \frac{c}{f} \sqrt{\varepsilon'}
\]

where \(c\) is the speed of light in m/s, \(f\) is the frequency in Hz, \(d\) is the thickness of the layer of material in meters, and \(n\) is an integer to be determined [14]. The two components of the relative complex permittivity, as defined by equations (1) and (2) are the average values for the air-material mixture.

Errors in determining \(\varepsilon'\) and \(\varepsilon''\) from free-space measurements are usually attributed to near-field effects, multiple reflections within the sample and between the antennas through the sample, diffraction at the edges of the sample, multiple-path transmission, and residual post-calibration mismatches. Measurement precautions [11] were taken to minimize, or at best eliminate, all these sources of error with the exception of the near-field effect, which is the object of this study for its influence on the accuracy of dielectric properties measurement and implications on predicted bulk density and moisture content.

2.1 Description of Far-Field Measurement System

Far-field conditions are met when the sample to be tested is placed at a distance equal to or greater than \(2D^2/\lambda\) from the antenna, where \(D\) is the largest dimension of the antenna and \(\lambda\) is the wavelength. For this to be fulfilled, it implies, in some instances, large distances between the antennas and the sample and samples with impractical dimensions, which make them difficult to handle and require large amounts of material. With an antenna characterized by a focused beam, far-field conditions are met at a short distance from the antenna with samples of reasonable size.

The far-field measurement system, shown in Figure 1, consists of two linearly polarized ultrabroadband horn/lens antennas (2 to 21 GHz), BAE SYSTEMS\(^1\) model AHO-2077-N,

\(^1\) Mention of company or trade names is for purpose of description only and does not imply endorsement by the U. S. Department of Agriculture or the University of Georgia.
connected to a HP 8510C vector network analyzer (VNA) with high quality coaxial cables. The antennas, facing each other, were placed 37 cm apart on a wooden structure, which kept them well aligned. The antennas have a cross-polarization level of -25 dB, on average, over the 2 to 18 GHz frequency range, and the E-plane 3-dB and 10-dB beamwidths at 10 GHz were 10.3° and 13.0°, respectively, based on the radiation pattern provided by the manufacturer. The horn/lens antennas collimate the electromagnetic energy in a relatively narrow beam and provide a plane wave at a short distance from the transmitting antenna. These features allow reduced sample size and minimize both the diffraction effects at the edges of the sample and any interference from the surroundings.

Fig. 1 Diagram of measurement setup showing horn/lens antennas.

The sample holder was a box of rectangular cross section made of 2.5 cm-thick Styrofoam sheet material. The internal dimensions of the sample holder were 25 cm transverse, 25 cm in height, and a maximum thickness of 15.4 cm. Different sample thicknesses (between 4 and 15.4 cm) were obtained by placing Styrofoam spacers inside the box. For each moisture level, the sample thickness was selected to ensure a 10-dB one-way attenuation in the sample to minimize the effect of multiple reflections. The grain and seed samples were poured into the sample holder, which was then placed between the two antennas. To isolate the sample from surroundings, it was surrounded by a tunnel-shaped enclosure made of radiation-absorbing material, ECCOSORB AN-79, with a reflectivity of −20 to −25 dB between 2 and 18 GHz. Finally, for each sample, time-domain gating was applied to \( S_{21} \) traces (modulus and phase) to filter out undesirable effects [11]. The VNA was calibrated in transmission mode with a response-type calibration between 2 and 18 GHz. All measurement sequences were computer controlled and the data collected for frequencies between 2 and 18 GHz were saved on a floppy disk.

For each sample of wheat and soybeans of given moisture content, measurement of the modulus and phase of \( S_{21} \) were taken at room temperature (about 24 °C) at three different bulk densities ranging from loosely packed to well settled. The moisture content of wheat and soybeans samples was determined with an oven-drying method according to ASAE recommendations [15]. The moisture content in percent, wet basis, was calculated as follows:

\[
M = \frac{m_w}{m_w + m_d} \times 100
\]  

(3)
where \( m_w \) is the mass of water and \( m_d \) is the mass of dry matter. For each sample, the bulk density was determined gravimetrically by dividing the total mass of the sample by the volume, \( V \), of the sample holder:

\[
\rho = \frac{m_w + m_d}{V}
\]  

(4)

2.2 Description of Near-Field Measurement System

For near-field measurements, only the horn/lens antennas shown in the measurement arrangement (Figure 1) were replaced by two single-patch microstrip antennas. The antennas were placed directly against the sample holder as shown in Fig. 2. With a Styrofoam thickness of 2.5 cm, the transmitting and receiving antennas were 2.5 cm away from the interface of any wheat or soybeans sample. The total distance between the transmitting and receiving antennas was 20.4 cm. Microstrip antennas were chosen because of their low cost, small size, and light weight. They can be easily integrated in cost-effective portable devices as well as in-line sensing systems. Microstrip antennas are characteristically narrow-band antennas [16]. The single-patch antennas used in this study were designed to resonate at 5.8 GHz [17]. Both transmitting and receiving antennas have vertical linear polarization with a return loss of 10 dB and a gain of 8.2. The measurement procedure is identical to that described for the far-field measurements. A major concern when this type of antennas are used for measurement of the dielectric properties in the near field is a shift in the frequency of resonance of the antennas that may result from changing characteristics of the medium to be tested, with possible implications on the accuracy of measured values for \( \varepsilon' \) and \( \varepsilon'' \).

3 Results and Discussion

Results of permittivity measurements in the near field with a pair of microstrip antennas on samples of wheat and soybeans of different moisture contents and different bulk densities are compared to those obtained with a pair of horn/lens antennas at 6 GHz and 24 °C in Figures 3 and 4. For purpose of comparison and to minimize the effect of bulk density, \( \varepsilon' \) and \( \varepsilon'' \) were divided by bulk density. Both the dielectric constant and loss factor, each divided by density, increase with moisture content. In general, values obtained with the microstrip antennas remain very close to those obtained with the horn/lens antennas.
In practice, dielectric properties are used as the basis for developing indirect nondestructive methods for sensing physical properties such as bulk density and moisture content in grain and seed [8]. One way to assess quantitatively the effect of near field measurements with microstrip antennas on the dielectric properties accuracy is to calculate and compare the standard errors of calibration ($SEC$) in predicting bulk density and moisture content from far-field measurements and near-field measurements. The $SEC$ is defined as:

$$SEC = \sqrt{\frac{1}{j - p - 1} \sum_{i=1}^{j} (\Delta e_i)^2}$$

where $j$ is the number of samples, $p$ is the number of variables in the regression equation with which the calibration is performed, and $\Delta e_i$ is the difference between the predicted value and that determined by a standard method for the $i$th sample.

3.1 Comparing Predicted Bulk Density

Bulk density of moist granular materials can be determined directly from measurements of their dielectric properties [8]. The complex-plane representations of $e'$ and $e''$, each divided by bulk density, for wheat and soybeans are shown in Figures 5 and 6, respectively.

![Fig. 3 Variation of the dielectric properties of wheat divided by bulk density with MC](image1)

![Fig. 4 Variation of the dielectric properties of Soybeans divided by bulk density with MC](image2)
For both wheat and soybeans, data taken with the near-field measuring system and with the far-field measuring system form a line in the complex plane and can be fitted by a linear regression of the form:

\[
\frac{\varepsilon'}{\rho} = a_f \left( \frac{\varepsilon'}{\rho} - k \right)
\]

(6)

where \( a_f \) is the slope of the line, which is dependent on frequency alone [8] and \( k \) is the x-axis intercept. Solving equation (6) for bulk density:

\[
\rho = \frac{a_f \varepsilon' - \varepsilon''}{ka_f}
\]

(7)

Table 1 provides the regression coefficients and the coefficients of determination for wheat and soybeans. Equation (7) has the advantage of providing the bulk density directly from measured dielectric properties without knowledge of moisture content or temperature of the sample. For each material and each measurement setup, the bulk-density calibration equation can be obtained from equation (7) and regression coefficients reported in Table 1. For purpose of comparison, the SEC was calculated for wheat and soybeans. Table 1 indicates that bulk density of wheat and soybeans can be better predicted from measurement of the dielectric properties in the far field. However, values of the SEC corresponding to near-field measurements are acceptable and represent a relative error of only about 2%.

Table 1  Regression coefficients and coefficients of determination for equation (7).

<table>
<thead>
<tr>
<th>Material</th>
<th>Near-field measurements</th>
<th></th>
<th>Far-field measurements</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( a_f )</td>
<td>( k )</td>
<td>( r^2 )</td>
<td>( SEC(\text{g/cm}^3) )</td>
</tr>
<tr>
<td>Wheat</td>
<td>0.512</td>
<td>2.7</td>
<td>0.953</td>
<td>0.024</td>
</tr>
<tr>
<td>Soybeans</td>
<td>0.657</td>
<td>3.17</td>
<td>0.984</td>
<td>0.017</td>
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</table>
3.2 Comparing Predicted Moisture Content

For granular materials such as cereal grain and oilseed, density-independent calibration functions were used for moisture determination from measured microwave parameters [3-5, 8]. Among these calibration functions, those expressed in terms of the dielectric properties are the most versatile since they can be applied regardless of the microwave measurement technique. A recent calibration function was tested on a variety of granular materials [8,13,19] and is expressed as follows:

\[ \psi = \frac{\epsilon''}{\sqrt{\epsilon'(a \epsilon' - \epsilon'')}} \]  

Fig. 7 and Fig. 8 show variation of \( \psi \) with moisture content. For both wheat and soybeans, \( \psi \) increases linearly with moisture content regardless of the measurement setup. However, unlike the variations observed for \( \epsilon' \) and \( \epsilon'' \), variations of \( \psi \) corresponding to dielectric measurements performed in the near field are distinct from those corresponding to measurements performed in the far field.

For each material and each measurement setup, a linear regression of the form:

\[ \psi = aM + b \]  

provides the relationship between \( \psi \) and moisture content. Table 3 shows regression coefficients and coefficients of determination corresponding to equation (9) for wheat and soybeans. Solving equation (9) for moisture content:

\[ M = \frac{\psi - b}{a} \]  

For each material and each measurement setup, the moisture-content calibration equation can be obtained from equation (10) and regression coefficients listed in Table 2. Effectiveness of each equation in predicting moisture content was compared through the computation of the SEC. Table 2 indicates that moisture content of wheat and soybeans can be better predicted from measurement of the dielectric properties in the far field. However, values of the SEC corresponding to near-field measurements remain within an acceptable range of accuracy.

Fig. 7 Variation of density-independent permittivity function \( \psi \) with MC in wheat

Fig. 8 Variation of density-independent permittivity function \( \psi \) with MC in soybeans
Table 2  Regression coefficients and coefficients of determination for equation (10).

<table>
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<td></td>
<td>a</td>
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<td>Wheat</td>
<td>0.019</td>
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</tr>
<tr>
<td>Soybeans</td>
<td>0.017</td>
<td>0.05</td>
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</table>

4 Conclusions

This study shows the feasibility of microwave characterization of moist granular materials in the near field with a pair of low-cost microstrip antennas. The dielectric properties of wheat and soybeans measured in free space with a pair of single-patch microstrip antennas are close to those measured in the far field with focused-beam horn/lens antennas. Similarly, standard errors of calibration for predicting bulk density and moisture content in wheat and soybeans from dielectric properties measured in the near field compare well with those determined from dielectric properties measured in the far field.

Both near-field measurement and use of microstrip antennas are important issues in developing cost-effective microwave sensing systems. Results presented in this paper constitute incentives for the commercialization and widespread use of this technology. The nondestructive sensing principle, shown here for wheat and soybeans, can be used for other moist granular and particulate materials.

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References


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Poster Session
Chairmen: E. Trinks; N. Wagner

P1 States of Water after the Ionising Radiation on Different Substituted Starches
S. Calvo, F. Henry, C. Aymes-Chodur, M. Serpelloni; France; L.-C. Costa; Portugal

P2 Calibration Transfer for the Unified Grain Moisture Algorithm
Z. Gillay; Budapest, Hungary; D. B. Funk; Kansas, USA

P3 Dielectric Properties of Bulk Materials and Restrictions to the Application of Two-Parameter Microwave Aquametry
I. Renhart; Minsk, Belarus; B. Tsentsiper; Houston, Texas, USA

P4 Scattering of Electromagnetic Waves by Short Thin Wire and Application to the Modeling of Composites
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P5 Temperature Corrections for a VHF Unified Grain Moisture Algorithm
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P6 Large-scale Sensing of Snow Pack Properties
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A. Brandelik, F. Königer, Ch. Kottmeier, H.-G. Mayer, R. Nüesch, A. Rohleder, S. Schlaeger, R. Schuhmann, M. Stacheder; Karlsruhe, Germany
States of Water after the Ionising Radiation on Different Substituted Starches

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ABSTRACT. The starch is the most important energy source to the human life. Its properties, particularly the biodegradability, are the base of different applications in areas like adhesives and glues, textiles, chemical industry, agriculture and civil engineering. It is present in the corn, potatoes and rice, it is observed as insoluble grain in water. Its shapes are an indication of the provenance, and the size range is between 1 and 100 µm.

Two macromolecules exist in the starch, the amylose (linear polymer) and the amylopectine (ramified polymer). The size and crystallinity of the grains of starch are responsible for their insolubility. In order to decrease this degree of insolubility we can make different treatments.

In this work, we have submitted to the action of ionising radiation (electron beam) of 25, 75 and 150 kGy, different starches i) native, ii) anionic with a substitution degree of 0.056% with sulphates and carboxylates groups and iii) cationic, with a substitution degree of 0.6% with amines groups.

With this ionisation, the goals are i) to increase the solubility in water and the film-forming of the starches and ii) to understand the interactions with water.

The study includes different measurements techniques and methods. The molecular masses are measured by Steric Exclusion Chromatography (SEC) with polystyrene as standard, and the interactions with water by FTIR. The low frequency impedance spectroscopy were used to determinate the critical concentration of interpenetration. The β dielectric relaxation was studied by dielectric measurements, which is particularly sensible to the water presence. Finally, the transition temperature Tg was determinate by DSC.
ABSTRACT. This research was performed to support the development of the Unified Grain Moisture Algorithm (UGMA). The main goals of this research were to develop appropriate mathematical models for parallel-plate transmission line test cells and to test the transferability of grain moisture calibrations (based on the UGMA) among different types of test cells. One of the goals of the UGMA was to permit calibration transfer among different moisture meter models with minimal effort. This paper reports results of calibration transfer tests between a large “master” test cell and a smaller, practical test cell design.

Keywords: dielectric, moisture, grain, standardization

1 Introduction

1.1 Unified Grain Moisture Algorithm

The United States Department of Agriculture—Grain Inspection, Packers and Stockyards Administration has developed a Unified Grain Moisture Algorithm (UGMA) that has the potential to provide improved accuracy, better calibration stability, reduced calibration development effort, and calibrations that are transferable among different moisture meter models that are built to conform to the basic algorithm. Briefly, the algorithm involves measuring the dielectric constant at 149 MHz, applying an effective density correction, applying three grain-group-specific “unifying parameters” to the density-corrected dielectric constant values, and then applying one polynomial equation to predict moisture content for all grain types. The objectives and mathematics of the UGMA are discussed further in two other papers [1,2] in these proceedings.

The original research upon which the UGMA was based used a large “Master” test cell that was far too large to be practical for commercial grain moisture meters. Therefore, it was necessary to “transfer” the UGMA calibrations to other much smaller test cells. This research involved 1) developing mathematical models to describe the relationships between (measured) complex reflection coefficient and “actual” dielectric constant for transmission line test cells of different dimensions, and 2) analyzing the transferability of calibrations from the Master test cell to a smaller test cell.

Direct comparison was performed for 400 samples of several different grain types. A simple standardization process that used only one grain type yielded fairly close overall agreement for all of the grain types. The standard deviations of differences for individual grain types were low; however, there were significant biases and slope differences between the two test cells for some grain types—particularly sunflower seed and rough rice. Small grain-type-specific adjustments were successful in achieving excellent agreement between test cells for all grain types.
2 Materials and Methods

2.1 Materials

The calibration transfer comparisons involved 400 samples representing 8 grain groups and a total of 15 grain types from the USDA-GIPSA Annual Moisture Study [3]. The grain groups and moisture ranges are listed in Table 2.1. Note that some grain groups include multiple grain types; the wheat group, for instance, includes five classes of wheat.

Table 2.1 Grain groups, numbers of the samples, and minimum and maximum moisture levels

<table>
<thead>
<tr>
<th>Grain group</th>
<th>Number of samples</th>
<th>Minimum moisture</th>
<th>Maximum moisture</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soybeans</td>
<td>75</td>
<td>8</td>
<td>24</td>
</tr>
<tr>
<td>Sorghum</td>
<td>12</td>
<td>13</td>
<td>26</td>
</tr>
<tr>
<td>Sunflower</td>
<td>36</td>
<td>5</td>
<td>36</td>
</tr>
<tr>
<td>Corn</td>
<td>93</td>
<td>9</td>
<td>44</td>
</tr>
<tr>
<td>Oats</td>
<td>6</td>
<td>9</td>
<td>19</td>
</tr>
<tr>
<td>Wheat</td>
<td>63</td>
<td>9</td>
<td>24</td>
</tr>
<tr>
<td>Barley</td>
<td>24</td>
<td>10</td>
<td>17</td>
</tr>
<tr>
<td>Rough rice</td>
<td>91</td>
<td>9</td>
<td>25</td>
</tr>
</tbody>
</table>

2.2 Instrumentation

2.2.1 Test Cells

The “Master” test cell (Fig. 1) was constructed as a 50-ohm transmission line. It consisted of three parallel aluminium plates. The ends of the plates were connected to endplates from a Hewlett-Packard 805A Slotted Line. Each endplate contained a machined conical 50-ohm transition from an N-type coaxial connector to a threaded stud that was connected directly to the centre plate. The centre plate was 12 mm shorter than the two outer plates, so there was a 6 mm air gap between each end of the centre plate and the endplate of the test cell.

Fig. 1 HP-4291A RF Material/Impedance Analyzer, loading funnel, and the Master test cell

A sliding gate, consisting of a 6 mm thick PVC plate, was positioned under the centre grain-filled section of the transmission line. The test cell was mounted on aluminium end supports
that were bolted to an aluminium plate (approx. 300 mm x 740 mm x 0.76 mm thick) as shown in Figure 1. The centres of the connectors on either end of the test cell were located 145 mm above the aluminium plate. Lawrence [4] provides more mechanical details of the Master test cell design.

Figure 2 shows two views of the Prototype VI test cell that was used in this research to test calibration transferability. This test cell was dimensioned to be more practical for commercial implementation than the Master test cell. The air-filled and grain-filled sections were both shortened substantially. The height of the test cell plates was reduced, and the spacing of the plates was reduced proportionately to maintain 50-ohm characteristic impedance. The parallel plates were made from 3.2 mm double-sided copper-clad epoxy-glass circuit board material because of ease of fabrication and mounting for the centre plate. At each end (both sides) of the centre plate, the copper cladding was removed from a six mm strip to provide an insulating gap similar to the air gap on the Master test cell. Two 10 mm tabs were machined on each end of the centre plate to fit tightly in matching slots in the endplates of the test cell. (See test cell end view in Fig. 2.) At each end of the test cell, N-type chassis connectors were bolted to the endplates and the centre contacts were connected to both sides of the centre plate through a 6 mm section of 4 mm diameter brass tubing. Pieces of Owens-Corning Pink® extruded polystyrene (dielectric constant of approximately 1.08) were machined to fill the “air-filled” sections of the test cell and provide boundaries for the grain-filled section. Another piece of extruded polystyrene was used for the removable gate beneath the grain-filled section. The test cell was mounted on aluminium end supports that were bolted to an aluminium plate (approx. 102 mm x 330 mm x 0.76 mm thick). The test cells were terminated with a precision 50-ohm load (HP-909C) during grain tests. Table 2.2 gives additional dimensions for the two test cells.

![Fig. 2 Prototype VI test cell](image)

![Table 2.2 Test cell parameters](image)

<table>
<thead>
<tr>
<th>Test Cell</th>
<th>Plate Height (mm)</th>
<th>Air section Length (mm)</th>
<th>Grain section Length (mm)</th>
<th>Spacing (mm)</th>
<th>Volume (ml)</th>
<th>Plate Thickness (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Master (Fig.1)</td>
<td>90.0</td>
<td>223.8</td>
<td>152.4</td>
<td>31.4</td>
<td>861</td>
<td>4.8</td>
</tr>
<tr>
<td>Prototype VI (Fig.2)</td>
<td>76.2</td>
<td>63.5</td>
<td>101.5</td>
<td>26.8</td>
<td>415</td>
<td>3.2</td>
</tr>
</tbody>
</table>
2.2.2 Measurement System

The HP-4291A RF Material/Impedance Analyzer (Fig.1) is a single-port RF instrument designed to measure and complex reflection coefficients (and other derived parameters) with high precision from 1 to 1800 MHz. It includes software to calculate correction parameters to calibrate the instrument with standard networks (open, short, and 50-ohm load) at the instrument’s test port and to extend that calibration to other reference planes. The instrument is based on Hewlett-Packard’s “RF – I/V” method rather than a classical vector network analyzer approach in order to provide high accuracy over a wide range of impedance values [5].

2.3 Grain Test Method

Before each day’s tests, the HP-4291A system was calibrated according to the manufacturer’s recommendations by means of open, short, and 50-ohm load tests. Each day, tests were performed for all available samples first with the Master test cell and afterwards with the Prototype VI test cell. Immediately prior to loading each sample into the loading funnel, the temperature of the sample was determined with a thermocouple-type digital thermometer (0.1 °C resolution). Samples were loaded into the test cell through a funnel (Seedburo Model 151) that was positioned over the centre of the grain-filled section. After the sample overflowed the test cell, the excess grain was removed by striking off the grain with a wooden straightedge. The excess was removed from the vicinity of the test cell before initiating the complex reflection coefficient measurement (a reading every 2 MHz from 1 to 501 MHz). After the reflection coefficient measurements, the sample was emptied from the test cell and weighed (0.1 gram resolution). The actual moisture content of each sample was determined by the applicable official USDA air oven method [6].

2.4 Data Analysis Methods

The data (sample number, air oven moisture, sample mass, temperature, and complex reflection coefficient) were collected into separate files by grain type for processing. The reflection coefficient data were converted to effective dielectric constant by means of two types of models (for comparison). All mathematical algorithms were coded with Mathcad Version 2001i [7].

2.4.1 Signal Flow Graph Model

The signal flow graph model of the test cells describes two transmission line sections and two interfaces: the air-filled section nearest the signal source, the interface between the first air-filled section and the grain filled section; the grain-filled section, and the interface between the grain filled section and the second air-filled section nearest the precision 50-ohm termination. Because the characteristic impedance of the transmission line test cell is assumed to be 50 ohms—matched to the termination—the length of the second air-filled section does not appear in the resulting mathematical model (Eq. 1).
\[
F_m = e^{-j \cdot \frac{2 \cdot \omega}{c} \cdot d_0} \cdot \frac{\left( 1 - \frac{1}{1 + \sqrt{\varepsilon}} \right)}{\left( 1 - \frac{1}{1 + \sqrt{\varepsilon}} \right)^2 - j \cdot \frac{2 \cdot \omega}{c} \cdot d_g \cdot \sqrt{\varepsilon}}
\]

where \( F_m \) = measured reflection coefficient, \( \omega \) = radian frequency, \( d_0 \) = air-filled section length, \( d_g \) = grain-filled section length, \( c \) = velocity of light, and \( \varepsilon \) = grain complex permittivity.

2.4.2 ABCD Matrix

More flexible models were created based on the ABCD matrix approach [8]. Fig. 3 shows the block diagram of the elements that were included in the ABCD matrix model. Each of the transmission line sections was defined by its length, characteristic impedance (Z), and propagation constant (\( \gamma \)). Furthermore, the transitions at either end of the test cell were modelled by lumped parameters (L and C) and the termination resistance could be varied from the nominal value of 50 ohms. Space limitations prevent showing the details of the ABCD matrix model.

The signal flow graph model and the ABCD matrix model calculate complex reflection coefficient values from complex permittivity values, but the calculation that is needed here it to go the other direction—from complex reflection coefficient to complex permittivity. An iterative non-linear solver (based on the Mathcad FIND function) was used with both model types to determine the complex (effective) permittivity from the measured complex reflection coefficient. The model parameters for the Master test cell were determined based on measurements of several types of high-purity alcohols by Kurt C. Lawrence. The extra modelling flexibility afforded by the ABCD matrix approach was not needed to characterize the Master test cell; the simpler signal flow graph model yielded equivalent results.

2.4.3 Unified Grain Moisture Algorithm

The equations that were used to calculate moisture content from the complex permittivity are presented in another paper in these proceedings [1].
3 Results and Discussion

3.1 Wheat (Grain-Type-Independent) Standardization

We chose wheat (63 samples representing 5 classes of wheat) as the “transfer” standard for a grain-type-independent standardization. The test cell model parameters (air-filled length, empty cell correction factor, and filling fraction) were adjusted to achieve minimum differences between the density-corrected dielectric constant values (and, therefore, the predicted moisture values) for the two test cells. The unifying parameters and polynomial regression coefficients that were developed for 1998 to 2003 data were used to predict moisture contents. Figure 3A shows the moisture prediction errors (with respect to the air oven moisture values) for the Master test cell for all grain groups. (The wheat data are marked by filled circles in Figs. 3A, B, and D.) Figure 3B shows the prediction errors for the Prototype VI test cell. Figure 3D shows the standardization errors (predicted moisture for the Prototype VI test cell minus the predicted moisture for the Master test cell). The solid “funnel” lines in the plots represent the moisture accuracy tolerances (with respect to the air oven method) for corn, rice, oats, and sunflower that have been established by the US National Conference on Weights and Measures [9]. The lines in Figs. 3D and E are dashed because those tolerances do not apply to differences between instruments.

With the simple standardization based on wheat, the agreement between the two test cells was reasonably good for all grain groups in the low moisture range (below about 20 percent moisture), but the agreement was progressively poorer at higher moisture levels. Corn and sunflower seed, which had the widest moisture ranges, were most affected. Figure 4 presents the results for each grain group individually. Mean differences are marked by circles and the error bars represent plus and minus one standard deviation of the differences. Figure 4D shows small but potentially significant mean differences between the two test cells for different grain groups. Also, residual plots for individual grain groups showed significant slope differences—especially between rice and sunflower seed.

Many different adjustments to the test cell model parameters in the ABCD matrix were attempted to eliminate the slope differences and mean differences among grain groups. As yet, no grain-independent approach has been entirely successful. For example, adjusting the characteristic impedance of the transmission line in the model was capable of minimizing the differences between rice and sunflower seed—but at the expense of greater differences for corn. Several hypotheses regarding the cause of the differences are yet to be tested.

3.2 Grain-Specific Standardization

A second level of standardization was applied to eliminate the differences among grain groups. The slope and offset unifying parameters (defined by Funk [1]) for each grain group were adjusted as needed to eliminate the mean differences and slope differences among grain groups. Figs. 3C and E and Figs. 4 C and E show the moisture accuracy and standardization agreement that were achieved after grain-specific standardization.
Fig. 3 Moisture prediction errors with respect to the air oven method (A, B, C) and standardization errors (differences between Prototype VI and Master test cells) (D, E) for grain-independent standardization (B, D) and grain-specific standardization (C, E).

Fig. 4 Moisture prediction errors (mean (o) and plus and minus one standard deviation (bars)) for seven grain groups. Sy: soybeans, Sg: sorghum, Sf: sunflower seed, Cn: corn, Oa: oats, Wh: wheat, Ba: barley, and Ri: rice.
3.3 Discussion

From Figs. 4A, B, and D, it can be seen that the standard deviations of differences between results for the two test cells (with only the simple standardization) are very small relative to the standard deviations of the errors with respect to the air oven for either test cell (except for corn and sunflower seed where slope differences inflated the values). Therefore, the intent of achieving close agreement among instruments appears to be achievable, though some adjustments in the unifying parameters may be needed to align results for different moisture meter models with very different test cells. The accuracy (with respect to the air oven moisture) of the Prototype VI test cell after grain-specific standardization (Fig. 4C) was virtually indistinguishable from that of the Master test cell (Fig. 4A).

4 Conclusions

The predicted moisture values for the two test cells tracked closely (as shown by the low standard deviations of differences) with simple or grain-specific standardization. A simple standardization based on one grain type did not completely eliminate offset and slope errors between the two test cells’ results for all grain types. For high accuracy applications, additional grain-specific standardization may be required. The same basic polynomial regression equation gave essentially equal accuracy for both test cells after grain-specific standardization. With grain-specific standardization adjustments, the agreement between the results for the two test cells was very small compared to the agreement to the air oven. We are testing other hypotheses that may eliminate the need for grain-dependent standardization adjustments.

References

3. USDA-GIPSA, Moisture Handbook. USDA-GIPSA, 1999

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Dielectric Properties of Bulk Materials and Restrictions to the Application of Two-Parameter Microwave Aquametry

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ABSTRACT. The application of two-parametrical methods for density-independent measurements of moisture content has been considered. Theoretical estimation of the accuracy of determination of moisture in the scope of linear model of mixture was discussed. The changes of dielectric properties with the increase of moisture content were studied for the substances containing free or bound water using resonator and waveguide methods. The theoretical and experimental investigation shows that the problems of moisture determination cannot be solved by the increase of the accuracy of measurements for some substances. The restrictions of the application of two-parametrical methods are discussed for different materials and sensors.

Keywords: moisture measurement, complex permittivity, two-parametrical method.

1 Introduction

Microwave moisture meters are an essential part of many industrial processes at present. In a number of these processes material moisture contents W are measured under the conditions of variable density ρ. In this case for W determination the measurements of density are necessary. The systems containing microwave moisture meters and isotopic densimeters are used for this purpose. However the problem of radioactive safety arises on their application. The methods invariant to density fluctuations are developed for moisture measurements under such conditions [1].

Two-parametrical methods of moisture determination based on measurements of complex permittivity $\varepsilon = \varepsilon' - i\varepsilon''$ or other two values connected with $\varepsilon'$ and $\varepsilon''$ using only microwave technique are widely discussed [2-4]. Unfortunately, measured parameters are not always independent in experimental conditions. The relative contribution of permittivity of dry materials to one of “material – water” mixture diminishes with the increase of moisture content and can reach the limit at which this contribution can be negligible in the frame of experimental error. In this case the mixture formulae defining $\varepsilon'$ and $\varepsilon''$ as functions of mixture component properties transform to the functions of $W\rho$ product and the determination of W and ρ from measured dielectric properties is impossible. This situation restricts the application of two-parametrical microwave methods. The aim of our study is the determination of boundaries of sensitivity of microwave two-parametrical methods for resonator, transmission and combined devices under moisture measurements for materials containing free and bound waters.

2 Theoretical

Within the scope of linear model [5] the complex permittivity for a 3-component mixture (material, water, air) is
\[ \varepsilon^n = \frac{\varepsilon^n_w - \varepsilon^n_d}{\sqrt{\varepsilon^n_w} - \sqrt{\varepsilon^n_d}}, \]

or

\[ \sqrt{\varepsilon'} = W \frac{\rho}{\rho_w} (\sqrt{\varepsilon'_w} - 1) + (\sqrt{\varepsilon'_d} - 1) \frac{\rho}{\rho_d} (1 - W) + 1, \]

where indices \( d \) and \( w \) correspond to dry material and water parameters. With the moisture increase, \( \sqrt{\varepsilon'} = f(W\rho) \). This situation is realized when

\[ W >> \left( \frac{\sqrt{\varepsilon'_d} - 1}{\rho_d} \right) / \left( \frac{\sqrt{\varepsilon'_w} - 1}{\rho_w} + \frac{\sqrt{\varepsilon'_d} - 1}{\rho_d} \right). \]

In the typical conditions \( \varepsilon'_d \approx 3 \), \( \rho_d = 1 + 2g/cm^3 \) and \( \varepsilon'_w \approx 80 \) we obtained \( W >> 4\% \). At these values of moisture within the scope of linear model the simultaneous determination of moisture and density using complex permittivity data is impossible.

We propose to carry out the hypothetical experiment. Assume that we conducted \( i \) measurements at different values of \( W_i \). Complex permittivity \( \varepsilon'_j \) was defined at different values of density \( \rho_j \) for each moisture. So we have a set of data (Tab.1). We can obtain \( \varepsilon''_{ij} = \varepsilon''_{j} \) in \( k \) and \( m \) measurements for diverse values of moisture and density. In this case according to (1) variation of imaginary part of dielectric functions \( \Delta \varepsilon'' \) can be determined.

### Table 1 Data obtained as the result of \( i \) measurements in hypothetical experiment

<table>
<thead>
<tr>
<th>1-th measurement</th>
<th>( W_1 )</th>
<th>( \rho_{11} )</th>
<th>( \varepsilon'_{11} )</th>
<th>( \varepsilon''_{11} )</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( \rho_{12} )</td>
<td>( \varepsilon'_{12} )</td>
<td>( \varepsilon''_{12} )</td>
<td></td>
</tr>
<tr>
<td></td>
<td>\vdots</td>
<td>\vdots</td>
<td>\vdots</td>
<td></td>
</tr>
<tr>
<td></td>
<td>( \rho_{ij} )</td>
<td>( \varepsilon'_{ij} )</td>
<td>( \varepsilon''_{ij} )</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>2-th measurement</th>
<th>( W_2 )</th>
<th>( \rho_{21} )</th>
<th>( \varepsilon'_{21} )</th>
<th>( \varepsilon''_{21} )</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( \rho_{2j} )</td>
<td>( \varepsilon'_{2j} )</td>
<td>( \varepsilon''_{2j} )</td>
<td></td>
</tr>
<tr>
<td></td>
<td>\vdots</td>
<td>\vdots</td>
<td>\vdots</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>i-th measurement</th>
<th>( W_i )</th>
<th>( \rho_{i1} )</th>
<th>( \varepsilon'_{i1} )</th>
<th>( \varepsilon''_{i1} )</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( \rho_{ij} )</td>
<td>( \varepsilon'_{ij} )</td>
<td>( \varepsilon''_{ij} )</td>
<td></td>
</tr>
</tbody>
</table>

\[ \Delta \varepsilon'' = \frac{\varepsilon''_w}{\sqrt{\varepsilon''_w}} \Delta W \frac{\rho}{\rho_w} \sqrt{\varepsilon'} + \frac{\varepsilon''_w}{\sqrt{\varepsilon''_w}} W \frac{\Delta \rho}{\rho_w} \sqrt{\varepsilon'}, \]

or

\[ \frac{\Delta \varepsilon''}{\varepsilon''} = \frac{\Delta W}{W} + \frac{\Delta \rho}{\rho}. \]
The variation of real part of dielectric function is

\[
\Delta \varepsilon' = 2\sqrt{\varepsilon} \left( \frac{\rho}{\rho_w} (\sqrt{\varepsilon_w} - 1) - (\sqrt{\varepsilon_d'} - 1) \frac{\rho}{\rho_d} \right) \Delta W + \\
2\sqrt{\varepsilon} (W \frac{\sqrt{\varepsilon_w'} - 1}{\rho_w} + (\sqrt{\varepsilon_d'} - 1)(1 - W)) \Delta \rho
\]  

(5)

Since \( \varepsilon' \) does not change under the conditions of our experiment, variation of density can be written in next kind

\[
\Delta \rho = -\Delta W \frac{(\sqrt{\varepsilon_w} - 1) \frac{\rho}{\rho_w} - (\sqrt{\varepsilon_d'} - 1) \frac{\rho}{\rho_d}}{W(\sqrt{\varepsilon_w'} - 1) + (\sqrt{\varepsilon_d'} - 1)(1 - W)}. 
\]  

(6)

In this case the variations of \( \varepsilon'' \) depend on moisture changes in the following way:

\[
\frac{\Delta \varepsilon''}{\varepsilon''} = \frac{\Delta W}{W} K(W),
\]  

(7)

where

\[
K(W) = \frac{\sqrt{\varepsilon_d'} - 1}{W(\sqrt{\varepsilon_w'} - 1) + (\sqrt{\varepsilon_d'} - 1)(1 - W)}. 
\]

The coefficient \( K(W) \) shows the extent of influence of moisture variations on the changes of imaginary part of mixture permittivity on the assumption that \( \varepsilon' = \text{const} \). The coefficient \( K \) depending on moisture was calculated for typical parameters of moist materials (Fig. 1). \( K \) sharply diminishes with moisture increase for all considered parameters.

This result points out that the changes in \( \varepsilon'' \) diminish with \( W \) growth but the problem of \( W \) measurements can be solved by the increase of the accuracy of measuring system. We obtained the theoretical estimation (within the scope of linear theory for mixtures) of the accuracy with which we should carry out measurements for determination of moisture. To confirm this outcome we carried out experimental investigation for materials containing free or bound water.
We studied the possibilities of the measurements of moisture by two-parametrical method for different materials and for different measuring systems. We used quartz sand as material containing practically free water in moist state and wheat flour containing up to $W=17\%$ exceptionally bound water. Samples of moist sand were prepared by mixing of water and dry sand, the samples of flour were obtained by drying flour with $W\approx 17\%$ up to required moisture. The moisture content of the prepared samples of sand and flour are determined by drying to constant weight at temperature $103\, ^\circ\mathrm{C}$ in a drying oven. To decrease the moisture fluctuations the samples were kept in sealed jars for 72 hours at room temperature. We also carried out measurements with potato starch and kaolin powder. The samples were prepared similarly to the experiment with flour.

The measurements were carried out by resonator and waveguide methods. In our experiments we defined the following couples of parameters: 1) shift of resonance frequency $\Delta f = f - F$ ($f$ and $F$ is a resonance frequency of filled with matter or empty resonator) and Q-factor $Q$; 2) $\Delta f$ and transient attenuation $A$ for resonator methods; or 3) $A$ and phase incursion $\beta$ for waveguide methods. All these parameters are the functions of dielectric properties of materials studied.

The samples of moist sands are located in a bulk cylindrical resonator. The measurements were carried out at the resonator frequencies of $F_1=1.5$ and $F_2=2.5$ GHz. In each measurements with fixed $W=W_a$ we determined the values of $f_{nk}$ and $Q_{nk}$ for different densities $\rho_{nk}$ (Tab. 2). Using our experimental data and regression analysis we obtained functions $Q_n = Z_n(f_n)$ Then we calculated the values of $Q$ for different moisture contents at the fixed value $f_c=1595\, MHz$ for $F_1$ and at $f_c=2595\, MHz$ for $F_2$. Note that resonance frequency shift and Q–factor is the functions of complex permittivity and to some extend the situation with fixed frequency in real experiment is the analogue of our hypothetical experiment, where the fixed value was $\varepsilon'$. We also carried out the measurements of these parameters for different moisture at fixed densities $\rho_c=1.45\, g/cm^3$. Dependence of Q-factor on moisture in cases of fixed density or fixed shift of resonance frequency are shown in Fig. 2.

![Fig. 1 Coefficient K in dependence on moisture for different material parameters.](image)
Table 2 Experimental data and data analysis

<table>
<thead>
<tr>
<th></th>
<th>( \rho_{11} )</th>
<th>( \Delta f_{11} )</th>
<th>( Q_{11} )</th>
<th>( Q_{i} = Z_{i}(f_{i}) )</th>
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<td>1-th measurement ( W_{1} )</td>
<td>( \rho_{12} )</td>
<td>( \Delta f_{12} )</td>
<td>( Q_{12} )</td>
<td>( Q_{i} = Z_{i}(f_{i}) )</td>
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<td>( \rho_{1j} )</td>
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<td>( Q_{1j} )</td>
<td>( Q_{i} = Z_{i}(f_{i}) )</td>
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<tr>
<th></th>
<th>( \rho_{21} )</th>
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<th>( Q_{21} )</th>
<th>( Q_{i} = Z_{i}(f_{i}) )</th>
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<tbody>
<tr>
<td>2-th measurement ( W_{2} )</td>
<td>( \rho_{22} )</td>
<td>( \Delta f_{22} )</td>
<td>( Q_{22} )</td>
<td>( Q_{i} = Z_{i}(f_{i}) )</td>
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<td>( \rho_{2j} )</td>
<td>( \Delta f_{2j} )</td>
<td>( Q_{2j} )</td>
<td>( Q_{i} = Z_{i}(f_{i}) )</td>
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<th>( \Delta f_{i1} )</th>
<th>( Q_{i1} )</th>
<th>( Q_{i} = Z_{i}(f_{i}) )</th>
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<tbody>
<tr>
<td>i-th measurement ( W_{i} )</td>
<td>( \rho_{ij} )</td>
<td>( \Delta f_{ij} )</td>
<td>( Q_{ij} )</td>
<td>( Q_{i} = Z_{i}(f_{i}) )</td>
</tr>
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The similar measurements were carried out for wheat flour by the use of a bulk cylindrical resonator. But in this case we determined \( \Delta f \) and transient attenuation \( A \). Measurements were carried out at the resonator frequencies of \( F_{1}=1.1 \) and \( F_{2}=2.5 \) GHz. The procedure described above was used for determination of dependences of transient attenuation on moisture in the conditions of fixed shift of resonance frequency or fixed density (Fig. 3). We also studied the dependence of \( A \) and \( f \) on moisture increase in the experiments at \( F=2.5 \) GHz with potato starch by the same way (Fig. 4).

The waveguide method was used in experiments with moist kaolin powder. The measurements were carried out at \( F=10 \) GHz. Attenuation \( A \) and phase incursion \( \beta \) in the layer of moist matter was used as measured parameters. For each moisture function \( A_{i} = \Psi_{i}(\beta_{i}) \) was determined using experimental data on \( A \) and \( \beta \) and regression analysis. Results of investigation are shown in Fig.5.

4 Results and Discussion

The determination of sensitivity of measurements to moisture and density variations is very important task in development of microwave moisture meters. As follows from our theoretical consideration in the approximation of linear model of mixtures that with \( W \) increase, moisture variations result in very small changes in \( \varepsilon'' \) at fixed \( \varepsilon' \). This points out on very high requirements to accuracy of microwave moisture meters and maybe, on impossibility of distinctive determination of moisture and density by use of exclusively microwave measurements. Our experimental study confirms this outcome.

According to our measurements with quartz sand by resonator method, Q-factor monotonically decreases with increase of moisture at fixed density (Fig. 2a,b). This behavior is typical for the most of large disperse materials. But Q-factor diminishes with \( W \) increase when \( f_{c} = \text{const} \) at first and then \( Q \) does not change. It takes place at \( W>4\% \). We can conclude that this moisture magnitude is the boundary value of \( W \) up to which we can use microwave methods. The sensitivity of measurements at \( W = 4\% \), which follows for quartz sand from
expression (7), gives the value of ~0.3%. Thus our measurements for moist quartz sand confirm conclusion obtained within the scope of linear theory of mixture.

We observed the same situation under measurements with wheat flour. We have typical dependence for \( A(W) \) at fixed densities. The modulus of \( A \) grows with \( W \) increase at fixed \( \rho \). When \( f_c = \text{const} \), for both resonant frequencies \(|A|\) increases with moisture growth up to \( W \sim 15\% \) and with further \( W \) growth, attenuation does not change (Fig. 3a,b). But in contrast to large disperse matter (quartz sand) this is observed for moisture values greater than 15+20%. So we again obtained that there is the restriction of application of two-parametric method or microwave techniques but in this case this restriction is reached at the greater values of the moisture in comparison with quartz sand. It means also that for moisture measurements in wheat flour the sensitivity requirements for measuring devices are not so strong like in the case of large disperse materials. This advantage of small disperse matter in respect to large disperse matters can be explained by presence of bound water in small disperse matters.

It should be noted that we observe monotonically lessening \( Q \) and \( A \) dependence on moisture for both large disperse or small disperse materials. This behavior is typical for a number of materials.
However, our study shows that in the experiments with potato starch or kaoline powder the dependences $A(W)$ have an extremum or the dependence curves have inflexion (Fig. 4 and Fig. 5). In this situation the equal values of attenuation correspond to different values of moisture. It is evident that the measurements of moisture are impossible in this case. For potato starch we can carry out these measurements up to values of $W \sim 15\%$, where $A(W)$ monotonically lessens at fixed frequency shift. As for kaolin powder, the measurements are not already possible at $W \geq 4\%$. This change in behavior of $A(W)$ (we mean the appearance of an extremum) can be explained by formation of free water. As is known both these materials (flour and starch) contain bound water. With moisture increase along with bound water free water appears. This results in faster growth of $\varepsilon'$ in comparison with the imaginary part and leads to change in behavior of dependence of measured parameters on moisture.

5 Conclusion

Our theoretical study within the scope of linear theory of mixture shows that the application of two-parametrical microwave methods for moisture determination has essential restrictions. The experiments carried out with materials containing free and bound water confirm this conclusion. We obtained that the application of this method for materials containing free water (quartz sand) is restricted by the values of moisture less than 4%. In the case of materials containing bound water (wheat flour and potato starch) the measurements of $W$ by two-parametrical method are possible up to $W \sim 15+20\%$. Our theoretical and experimental results show that the simple increase of accuracy of measuring system can solve the problem of moisture measurements not always. The dependences of measured parameters on moisture have inflexion for some materials (potato starch and kaoline powder). In this case different values of moisture correspond to equal measured parameters and the use of two-parametrical methods for moisture measurements are impossible.
Acknowledgments

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References


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Scattering of Electromagnetic Waves by Short Thin Wire and Application to the Modeling of Composites

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ABSTRACT. A numerical technique of polarizability tensors calculation of thin-wire elements is developed. This method is based on the numerical solution of the Poklington integral equation. The opportunity to define of effective constitutive parameters for composite materials on the basis of conducting fibres is shown. The property of these composites to transform polarization of an incident field is considered.

Keywords: susceptibility coefficients, chiral materials

The progress in the field of designing artificial composite materials has determined their wide application in different areas of microwave engineering: in frequency and polarizing filters, in polarization transformers and for making non-reflective coating. One of perspective directions in the field of composites development is application of dielectric materials, which contain metal fibres inclusion of various geometry. Analysis of such structures presumes calculation of composite macroscopic parameters with respect to separate inclusions geometry and to their arrangement in a dielectric.

In this work the method of susceptibility coefficients (SC) calculation for conducting fibres of arbitrary configuration is offered. As the conductor has an arbitrary shape there is an opportunity to analyze particles having the chiral properties. Later on known SC of single scatterer the effective electrodynamics parameters (EEP) (such as permittivity \( \varepsilon_{\text{eff}} \), permeability \( \mu_{\text{eff}} \) and for chiral medium the chiral parameter \( \kappa_{\text{eff}} \)) of composite on their basis are determined.

The technique of SC calculation is based on the interaction analysis of an electromagnetic field with a separate wire inclusion. Under external influence, current is induced along the particle. Its calculation is carried out by numerical solution of Pocklington integral equation (IE) for current in thin conducting fibres. The solution of IE is carried out by collocation method with step basis function application [1]. Since this IE is easily generalized for the case of impedance conductors [2], it enables to take into account the phenomenon of skin-effect in metal fibres and to investigate the influence of particle conductivity on EEP of a composite in general.

On known current distribution the values of electrical \( \tilde{p} \) and magnetic \( \tilde{m} \) moments for a wire element are computed [3]:

\[
\tilde{p} = \frac{1}{i\omega} \int \tilde{I}(s) ds ,
\]

\[
\tilde{m} = \frac{\mu_0}{2} \int \left[ \tilde{r}(s) \times \tilde{I}(s) \right] ds .
\]

The integration in formulas (1), (2) is held along the wire.
If linear dimensions of a particle are small in comparison with the wavelength \( L_r \ll \lambda \), the relation of dipole moments \( \vec{p}, \vec{m} \) and SC is determined by the following matrix equation [3]:

\[
\begin{bmatrix}
\vec{p} \\
\vec{m}
\end{bmatrix} =
\begin{bmatrix}
\vec{a}_{ee} & \vec{a}_{eh} \\
\vec{a}_{he} & \vec{a}_{hh}
\end{bmatrix}
\begin{bmatrix}
\vec{E}_0 \\
\vec{H}_0
\end{bmatrix},
\]

(3)

where \( \vec{E}_0, \vec{H}_0 \) – intensity amplitude of the incident electromagnetic field, \( \vec{a}_{ee}, \vec{a}_{eh}, \vec{a}_{he}, \vec{a}_{hh} \) – SC of particles.

The matrix expression (3) represents the system of \( N \) linear algebraic equations (\( N=6 \)), in which the unknown magnitudes are tensors of SC (the quantity of unknown values makes \( N^2 \)). To define all the SC it is necessary to consider \( N \) cases of particle excitation \( \vec{E}_i, \vec{H}_i \) and its corresponding responses \( \vec{p}_i, \vec{m}_i, \ i=1,6 \). Since SC do not depend on the way of particle excitation, it is possible to write down:

\[
\begin{bmatrix}
\vec{p}_1 \\
\vec{m}_1
\end{bmatrix} + \begin{bmatrix}
\vec{p}_2 \\
\vec{m}_2
\end{bmatrix} + \ldots + \begin{bmatrix}
\vec{p}_6 \\
\vec{m}_6
\end{bmatrix} =
\begin{bmatrix}
\vec{a}_{ee} & \vec{a}_{em} \\
\vec{a}_{me} & \vec{a}_{mm}
\end{bmatrix}
\begin{bmatrix}
\vec{E}_1 \\
\vec{H}_1
\end{bmatrix} + \begin{bmatrix}
\vec{E}_2 \\
\vec{H}_2
\end{bmatrix} + \ldots + \begin{bmatrix}
\vec{E}_6 \\
\vec{H}_6
\end{bmatrix},
\]

(4)

or

\[
[P] = [A][E],
\]

where \([P]\) – the matrix of particle dipole response to external excitation \([E]\), \([A]\) – unknown square matrix of SC with dimensions \( N \times N \).

If there is an inverse matrix \([E]^{-1}\), the decision is found as \([A]=[P][E]^{-1}\), where elements of \([P]\) are determined after IE solving using the formulas (1), (3). For \([E]^{-1}\) to exist it is necessary to choose the vectors of external impact \( \vec{E}_i, \vec{H}_i, \ i=1,6 \) so that the columns of \([E]\) would be linearly independent, i.e. determinant \( \det[E] \neq 0 \). To get a stable solution the matrix \([E]\) should be well-conditioned.

![Graph of SC dependence on length](image.png)

**Fig. 1** The dependence of dipole SC from its length

On fig. 1, as a test, the dependences of wire dipole SC from its length are given (1 – numerical solution; 2 – asymptotic approximation where the vibrator is represented as an oblong ellipsoid [4]; 3 – the electric circuit approximation calculation in the assumption that the current along the particle is constant [3]). The dipole is supposed to be ideally conducting,
with wire radius \( r_0=0.1 \text{ mm} \) and located along \( Z \) axis. In this case, the only non-zero SC will be \( a_{zz}^{(e)} \). Apparently, the received numerical results are in good agreement with existing approximations. It is possible to explain some divergences in results by neglect in analytical expressions of interaction of the closely located particle elements and in case of [3] – the boundary conditions on conductor ends were not taken into account.

As distinct from work [5], where the SC calculation for spiral particles was carried out, the offered method of wire particle SC definition may be used for arbitrary geometry scatterers. In particular, this method was used for SC analysis of helix conductors, dipoles, \( \Omega \) – shape particles, helix with attached rectilinear conductors. Calculation result comparison of SC for some of the analyzed particles was carried out with the data acquired on the basis of other numerical methods [6] and various semianalytic approximations [7]. In most cases a good result agreement is observed in a wide range of frequencies, except for areas, in which the frequency of the incident field is close to resonant frequency of a particle. In this case analytical computational models become unacceptable and the necessary decision accuracy can be obtained using more precise numerical techniques.

After solving the system (5) and determining SC tensors for a particle, the calculation of composite EEP is carried out. The EEP definition for artificial medium consisting of a large number of randomly oriented particles in homogeneous dielectric is of great interest for practical application. In most general case medium can possess distinct chiral properties and can be described by the equations [3]:

\[
\vec{D} = \epsilon_{\text{eff}} \vec{E} - i \kappa_{\text{eff}} \sqrt{\mu_0 \epsilon_0} \vec{H}, \quad \vec{B} = i \kappa_{\text{eff}} \sqrt{\mu_0 \epsilon_0} \vec{E} + \mu_{\text{eff}} \vec{H}.
\] (6)

The calculation of EEP for such a material may be carried out by Maxwell-Garnett method, generalized for the case of a chiral medium. If dimensions of a single scatterer are small in comparison with wavelength and the volumetric filling factor \( Q/nV \) \((n – \text{concentration of particles}, V – \text{volume of scatterer})\) is small, EEP of the medium are determined as follows [6]:

\[
\begin{align*}
\epsilon_{\text{eff}} &= \frac{f(2b_{ee}, 2b_{eh}, b_{he}, b_{hh})}{f(-b_{ee}, -b_{eh}, b_{he}, b_{hh})}, & \kappa_{\text{eff}} &= \frac{inb_{eh}}{f(-b_{ee}, -b_{eh}, b_{he}, b_{hh})}, \\
\mu_{\text{eff}} &= \frac{f(-b_{ee}, 2b_{eh}, b_{he}, -2b_{hh})}{f(-b_{ee}, -b_{eh}, b_{he}, b_{hh})}, & f(x, y, z, t) &= \left(1 + nx \frac{3}{3}\right) \left(1 + nt \frac{3}{3}\right) + n^2 \gamma z, \\
\mu_c &= \frac{\text{tr} \left( \vec{a}_{ee} \right)}{\epsilon_c}, & b_{ee} &= \frac{\text{tr} \left( \vec{a}_{hh} \right)}{\mu_c}, & b_{eh} &= -i \frac{\text{tr} \left( \vec{a}_{eh} \right)}{\sqrt{\epsilon_c \mu_c}}, & b_{he} &= -i \frac{\text{tr} \left( \vec{a}_{he} \right)}{\epsilon_c \mu_c},
\end{align*}
\] (7)

where \( \epsilon_c, \mu_c – \text{permittivity and permeability of a dielectric, in which the particles are located, the coefficients } b_{ee}, b_{eh}, b_{he}, b_{hh} – \text{are normalized SC of inclusions. Symbol “tr” in (8) means the sum of diagonal elements for corresponding matrices.}

The above mentioned method of wire particles SC definition and EEP calculation also allows to take into account the influence of the closely located from each other particles. It enables to investigate and to create materials with non-trivial properties. Let’s show it on an example of EEP calculation for a medium consisting of randomly oriented double helices with small distance \( D \) between them. The coils of helices are twisted in opposite directions, as shown on fig. 2 a.
The particles have two coils, they are perfectly conductive, with radius and pitch of the helix $a=0.74 \text{ mm}$, $h=0.75 \text{ mm}$ correspondingly, wire thickness $2r_0=0.2 \text{ mm}$ and the distance between particles $D=1.8 \text{ mm}$. The wire inclusions are randomly oriented in dielectric with permittivity and permeability $\varepsilon_c=1$, $\mu_c=1$ and volumetric filling factor $Q=0.18\%$.

On fig. 3 frequency dependences of effective permittivity $\varepsilon_{\text{eff}}=\varepsilon'-i\varepsilon''$ and effective permeability $\mu_{\text{eff}}=\mu'-i\mu''$ for the investigated composite are given. Apparently, the curves are similar to the dependences in Lorentz’s dispersion model close to abnormal dispersion range. Since the considered geometry of inclusions has a centre of inversion, system does not exhibit chiral properties, which are typical of separate particles. The conducted calculations show that $\kappa_{\text{eff}}=0$ in the whole range of investigated frequencies.

Apparently, the system has two resonant frequencies at which it shows either electrical or magnetic properties. The closer particles are located to each other, the further these resonances are locate from each other (in the limit, when $D=0$, the areas of a resonance will be located as far away from each other as possible). This fact is in good agreement with the conclusions of other authors [3], and its demonstration may serve as additional proof of the offered calculation model adequacy.

The same calculations for $\varepsilon_{\text{eff}}$, $\mu_{\text{eff}}$, $\kappa_{\text{eff}}$ definition were done also for composites with inclusions as separate helix, $\Omega$– structures, dipole systems, helixes with attached conductors, serially located between coils. The estimation of EEP dependence on thickness of wire
particles, their resistance, general length of a conductor, longitudinal and cross dimensions of particles, distance between coupled particles and inclusion concentration in volume of a composite was carried out. The influence of particle geometry peculiarities on their resonant properties is also estimated. It is known [7], that at interaction of an electromagnetic field with wire particles the current resonance is observed for \( L \approx \lambda / 2 \) \( (L_r \ll \lambda) \). At the same time at certain geometry of particles great deviations (more than 20%) are possible, both in larger and smaller wavelength regions. For example, similar effects are observed for a helix when its pitch and radius are changed while the wire length \( L \) remains constant. The dispersion characteristics of a composite made of such particles varies similarly. The resonance frequency of particle current can be controlled in a wide range by changing the reactive component of conductor surface resistance. It should be noted, that the most perspective particles for creating materials with distinct dispersion properties could become the scatterers on the basis of impedance fibres.

Polarization properties of the considered composites were also investigated. Knowing EEP, the calculation of ellipticity \( P \) and polarization plane rotation angle \( \alpha_{sc} \) for electromagnetic wave, that passed through a layer of a composite, was carried out. Typical results for an example of wire structure with chiral properties (fig. 2 b) are shown. The layer of a composite loaded with such particles also acquires chiral properties and it is able to change the polarizing characteristics of an incident electromagnetic wave. The incident wave is defined as parallel to surface layer normal and its polarization is linear. On fig. 4 the frequency dependences of ellipticity \( P \) (curve 1) and polarization plane rotation angle \( \alpha_{sc} \) (curve 2) for the considered chiral layer with thickness \( d = 9 \text{ mm} \) are given. Geometrical dimensions of the particles presented on fig. 2, a and fig. 2, b are the same. Apparently, material demonstrates polarizing properties mainly on frequencies that are resonant for particles. In resonance range the passed wave becomes in general case elliptically polarized, \( P > 0 \). The angle \( \alpha_{sc} \) depends on frequency and can reach the value \( \alpha_{sc} = 30^\circ \).

![Fig. 4 The frequency dependences of ellipticity \( P \) (1) and polarization plane rotation angle \( \alpha_{sc} \) (2) for chiral layer loaded with randomly oriented helix particles](image.png)

According to numerical experiments it is established, that \( P \) and \( \alpha_{sc} \) can be controlled in a wide range by changing geometry of a particle, conductor resistance, varying distance between structure elements and changing layer thickness.

Thus, on the basis of IE method the way of the SC analysis for thin-wire particles of arbitrary geometry that also allows to take into account the influence of the closely located elements of
a system is offered. EEP calculation for a composite on the basis of similar particles is carried out. The dependence of EEP from geometrical parameters of the particles is established. The possibility to create composite materials with controlled properties on the basis of conducting fibres is demonstrated.

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**Temperature Corrections for a VHF Unified Grain Moisture Algorithm**

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**ABSTRACT.** Successful dielectric grain moisture measurement methods require precise temperature correction. Typically, a fixed correction is added to the uncorrected moisture prediction. Dielectric characteristics at 149 MHz (for seven grain types over wide moisture and temperature ranges) were measured by a prototype measurement system. A temperature correction that was a linear function of both temperature and moisture content was found to be more effective than a moisture-independent correction. A quadratic (temperature) term improved the correction further if very low temperatures (< 0 °C) were considered.

*Keywords: grain, moisture, VHF, temperature*

1 **Introduction**

Research at the USDA-Grain Inspection, Packers and Stockyards Administration (GIPSA) has resulted in an improved RF dielectric method, the Unified Grain Moisture Algorithm (UGMA), that effectively combines many diverse grain types into a single unified calibration. This method appears to be capable of moisture measurement accuracy that is equal to or better than what is achievable for grain-specific calibrations with current RF dielectric-type instruments. The method predicts moisture content from the sample dielectric constant (149 MHz), sample density, and sample temperature. The method is described in detail in a prior publication [1] and summarized elsewhere in these proceedings. [2] The purpose of the research presented here was to facilitate the development of commercial instrumentation using the Unified Grain Moisture Algorithm by optimizing the functional form and specific coefficients for temperature correction.

Based on the limited data available the original research [1] suggested the use of a simple moisture-independent correction function (Eq. 1) for temperature correction—but stated that more research was needed to optimize the function.

\[
\%M_{TC} = \%M - KTC \cdot (T - 25)
\]  

(1)

where  
\( \%M_{TC} \) is the temperature-corrected predicted moisture content  
\( \%M \) is the predicted moisture content without temperature correction  
\( KTC \) is the temperature correction coefficient (% moisture per degree C.)  
\( T \) is the measured sample temperature in degrees Celsius

Some prior research indicated that the relationships between dielectric constant and temperature are dependent on sample moisture content and measurement frequency. Lawrence et al [3] examined the dielectric constant of wheat as a function of temperature (-5 to 50 degrees Celsius) for several different frequencies (0.1 MHz to 100 MHz). They found that the change in dielectric characteristics with temperature was highly dependent on sample moisture content at lower frequencies but nearly independent of moisture content at 100 MHz. Other research [4] (200 kHz to 20 MHz) indicated that Eq. 1 provides reasonably
accurate temperature correction at 20 MHz but is less satisfactory at lower measurement frequencies. Another study [5] showed that the dielectric method (with Eq. 1) has the potential for accurate measurements below zero Celsius if the sample moisture content is below certain limits.

2 Materials and Methods

2.1 Grain Samples

For this study, bulk samples were obtained from grain receiving stations in Hungary (Herceghalom and Szombathely). The grain types tested included: soybeans (*Glycine soja*), soft wheat (*Triticum aestivum*), sunflower (*Helianthus annuus*), rapeseed (*Brassica napus*), autumn barley (*Hordeum vulgare*), and oats (*Avena sativa*). The moisture contents of the samples were adjusted as necessary by adding distilled water to the samples, mixing the samples thoroughly in sealed containers, and allowing them to equilibrate under refrigeration for at least one week prior to testing.

2.2 Instrumentation

Figure 1 shows the block diagram of a prototype reflectometer-type sensor that was used to measure dielectric characteristics at 149 MHz. An Analog Devices AD-8302 Gain/Phase Detector provided 1) an output that was proportional to the logarithm of the ratios of the reflected and incident signal magnitudes at the test cell and 2) an output that was proportional to the phase difference between the incident and reflected signals. An Analog Devices type AD-590 temperature sensor (1 µA/K) was immersed in the grain in the test cell.

![VHF prototype sensor block diagram](image_url)

Fig. 1 VHF prototype sensor block diagram

The test cell (Fig. 2) was constructed as a parallel-plate transmission line consisting of two outer copper plates (76 mm x 229 mm x 0.8 mm) and a centre plate (also 76 mm x 229 mm) made of 0.8 mm thick double-sided copper-clad epoxy-glass circuit board material. At each
end of the centre plate, a 1-cm wide strip of copper cladding (both sides) was removed except for where the copper was soldered to a SMA-type coaxial connector. The spacing between electrodes was set at 25.4 mm to achieve a characteristic impedance of very nearly 50 ohms. The test cell was terminated with a precision 50-ohm load. Pieces of rigid extruded polystyrene foam insulation (Owens-Corning PINK®, $\varepsilon_r = $ approx. 1.1) were used as spacers between the plates and to define the grain-filled section (101.6 mm length). Other pieces of extruded polystyrene insulation were machined to provide an insulating jacket that completely surrounded the test cell.

The dc voltages from the level detector and the gain/phase detector and the current from the AD-590 temperature sensor were measured by an HP-3457A digital multimeter, which was controlled by a PC-compatible computer through a GPIB card. The software checked the temperature every 30 seconds and recorded a set of readings each time the sample temperature had changed by more than 0.5 °C from the temperature of the previously recorded data. This permitted unattended operation during the lengthy (several hours) sample equilibration periods.

The gain ($V_G$) and phase ($V_P$) signals from the AD-8302 detector system were converted to complex reflection coefficient ($\Gamma$) by means of Eq. 2.

$$\Gamma = \left[ 10^{V_G \cdot 0.45 \over 0.6} \cdot e^{j(51.033 + 87.609 \cdot V_P)} \right]$$

2.3 Test Methods

Grain was poured slowly into the test cell while the test cell was shaken to settle the sample. After loading, the top of the test cell was sealed with wide plastic adhesive tape to minimize moisture loss. The test cell was placed in a laboratory freezer (approx. –25 °C) and allowed to equilibrate for at least 12 hours. Then, the insulated test cell assembly was removed from the freezer, covered with a piece of polystyrene insulation, transported to the laboratory area (approx. 22 °C), connected to the measuring system, and allowed to warm (very slowly) to room temperature. When the grain had warmed to near room temperature, the automatic data collection sequence was terminated. After that, the test cell was placed in a laboratory oven (approx. 50 °C) and allowed to equilibrate for at least 6 hours. Then the test cell was removed from the oven and was reconnected to the measuring system. When the grain had cooled to near room temperature, the automatic data collection sequence was terminated. The grain was
emptied from the test cell and weighed so that the sample density could be determined. The reference moisture value for each test sample was determined by the appropriate Hungarian Ministry of Agriculture standard air oven method (Hungarian Standard 6367/3-83).

The mathematical steps involved in converting the complex reflection coefficient values to sample moisture contents are described elsewhere in these proceedings. [2]

3 Results and Discussion

We compared the effectiveness of three alternative temperature correction functions. Eq. 1 was the simplest—independent of sample moisture content and linearly proportional to sample temperature. Eq. 3 was linear with sample temperature and linearly dependent on sample (predicted) moisture. Eq. 4 added a term that was quadratic in temperature.

\[
\%M_{TC} = \%M - (KTC0 + KTCS \cdot \%M) \cdot (T - 25) \quad (3)
\]

\[
\%M_{TC} = \%M - (KTC01 + KTCS1 \cdot \%M) \cdot (T - 25) - KTCQ \cdot (T - 25)^2 \quad (4)
\]

Fig. 3 shows typical results for one of the grain types that were tested (wheat). Each curve represents the data for one sample. For some samples, only the low temperature part of the test was done. Curves are labeled with the corresponding samples’ air oven moisture values.
Figure 3a presents the predicted moisture values without temperature correction. The predicted values increased with increasing temperature for all samples. The wheat samples at moisture levels above 20 percent showed slope discontinuities at zero Celsius. Those discontinuities are believed to be due to the phase change of the free water in the samples. The average slope (% moisture per degree Celsius) for the 0 to 50 C temperature range was calculated for each sample. The average slope for all samples (of the grain type) was used as the $KTC$ value in the correction defined by Eq. 1. Figure 3b shows the effects of that temperature correction approach. The average slope for temperatures above zero Celsius was forced to zero, but individual samples exhibit significant residual slope errors.

Linear regression was used to determine the optimum $KTCO$ and $KTCS$ coefficients (for each grain type) for Eq. 3. Applying those coefficients in Eq. 3 to the predicted moisture data gave the results that are shown in Figure 3c for wheat. The slopes of the curves for all the samples are near zero in the 0 to 20 C temperature range, but some curvature is evident at temperature extremes.

Coefficients for Eq. 4 were calculated by polynomial regression for each sample. The quadratic temperature term provided by Eq. 4 reduced the error at temperature extremes, as shown (for wheat) in Fig. 3d. With the temperature correction provided by Eq. 4, the predicted moisture errors (relative to predicted moisture content at 25 C) were less than 0.6% moisture for all grain samples at temperatures above zero degrees Celsius. For samples with moisture levels below the "free" water limit (approx. 20 % for wheat), the useable temperature range extended to -20 °C or lower. The coefficients ($KTCQ$) of the quadratic term that were calculated for each sample were not highly correlated to sample moisture content, so an average value was selected for each grain type. A constant, moisture-independent $KTCQ$ (per grain type) avoided a major increase in model complexity. The optimum values (based on the available samples) for the coefficients for Eqns. 1 and 4 are given in Table 3.1.

Figure 4 shows the calculated optimum linear temperature corrections (% moisture per degree Celsius) as a function of grain moisture content for the seven grain types. It can be seen that there is a general decreasing linear relationship between the linear temperature correction needed and the sample moisture content. That is, predicted moisture values for higher moisture samples vary less with sample temperature than predicted values for lower moisture samples. It cannot be determined from the available data whether or not that trend continues for extremely low moisture and extremely high moisture samples.

Table 3.1 Temperature correction coefficients for Eq. 1 and Eq. 4 for seven grain types

<table>
<thead>
<tr>
<th>Grain</th>
<th>$KTC$</th>
<th>$KTCO$</th>
<th>$KTCS$</th>
<th>$KTCQ$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Barley</td>
<td>0.113</td>
<td>0.1774</td>
<td>-0.0050</td>
<td>-0.0014</td>
</tr>
<tr>
<td>Corn</td>
<td>0.093</td>
<td>0.1969</td>
<td>-0.0075</td>
<td>-0.0012</td>
</tr>
<tr>
<td>Oats</td>
<td>0.143</td>
<td>0.1844</td>
<td>-0.0051</td>
<td>-0.0014</td>
</tr>
<tr>
<td>Rapeseed</td>
<td>0.081</td>
<td>0.1745</td>
<td>-0.0098</td>
<td>-0.0013</td>
</tr>
<tr>
<td>Soybean</td>
<td>0.107</td>
<td>0.1366</td>
<td>-0.0072</td>
<td>-0.0022</td>
</tr>
<tr>
<td>Sunflower</td>
<td>0.054</td>
<td>0.0868</td>
<td>-0.0026</td>
<td>-0.0004</td>
</tr>
<tr>
<td>Wheat</td>
<td>0.088</td>
<td>0.1201</td>
<td>-0.0044</td>
<td>-0.0021</td>
</tr>
</tbody>
</table>
Fig. 4 Optimum (linear term) temperature coefficients for the quadratic temperature function (Eq. 4) versus moisture content for seven grain types. For each grain, the y-axis intercept value is $KTCO1$ and the slope is $KTCSI$ (see Table 3.1).

The measure of success of a temperature correction algorithm is how closely the corrected moisture values at other temperatures compare to the predicted moisture values at the reference temperature (typically 25 °C). One way of quantifying that agreement is to calculate the residual slope after temperature correction (slope of the corrected moisture predictions versus temperature). Fig. 5 presents the residual slope values for each of the samples for each of seven grain types for temperature corrections based on Eq. 1, Eq. 3, and Eq. 4. Zero residual slope, of course, is the goal. The residual slopes that are shown were calculated for sample temperatures between 0 and 40 degrees Celsius. Note the two different sizes of symbols. Smaller symbols indicate tests that included only the low (< 22 °C) temperature range.
Fig. 5 Residual (uncorrected) slope of the predicted moisture curve (from 0 to +40 Celsius) versus air oven moisture content for seven grain types. (*) with constant temperature correction (Eq. 1); (X) with linear moisture-dependent temperature correction (Eq. 3); (□) with linear moisture-dependent temperature correction plus a quadratic temperature term (Eq. 4). Large symbols signify full temperature range tests. Small symbols signify only low temperature tests were performed.
Figure 5 shows that, in general, the residual slope values are closer to zero for the moisture-dependent corrections than for the moisture-independent corrections. The advantage provided by the extra quadratic temperature term (Eq. 4) is not as apparent in Fig. 5 as in Figure 3d because the temperature range for the slope calculations did not extend below zero Celsius. Space limitations preclude showing those results.

4 Conclusions

Three temperature correction functions were evaluated for use with the 149 MHz Unified Grain Moisture Algorithm. Optimum temperature correction coefficients were tabulated for seven grain types. Linear and quadratic moisture-dependent temperature correction functions were found to be more effective than a simple moisture-independent function—especially below zero degrees Celsius. More temperature measurements are needed at low moisture levels and high moisture levels to confirm that a linear moisture-dependent and quadratic temperature-dependent correction is the optimum temperature correction function and to establish the correction coefficients more precisely. Similar tests are needed to determine correction coefficients for additional grain types.

5 Acknowledgements

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Large-Scale Sensing of Snow Pack Properties

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ABSTRACT. A new in-situ sensor for the simultaneous measurement of snow water equivalent (SWE), snow density and liquid water content is presented. The system consists of radio frequency transmission lines of up to 25 m length cast in a flat PVC-band. The dielectric coefficient along the flat-band cable is measured with a time domain reflectometer (TDR) at high frequencies, and with a low frequency impedance analyzer. The performance of the sensor system has been tested at a high alpine test site in Switzerland. The cable suspension and set-up of the sloping cable showed to be critical with regard to stability and formation of unwanted air-gaps along the cable. Overall, the sensing system proved to be quite robust and produced results in agreement with manual snow pack observations.

Keywords: TDR, Frequency domain, snow water equivalent

1 Introduction

Avalanche and flood warning authorities are in dire need for precise snow property data. Hydro-power stations in alpine or Nordic countries depend on snow parameters of their catchments in order to predict the filling stage of their water storage reservoirs. Also, the distribution of liquid water flow paths in a snow pack is important to know for solute transport and wet-snow avalanche formation.

Radar remote sensing has potential to provide this information if accurate ground reference measurements are available for calibration. For this purpose, we developed and tested a new in-situ sensor system for snow moisture and density determination in a snow pack. The system consists of up to 25-m long radio frequency transmission lines cast in a flat PVC-band. The dielectric coefficient of the snow along the flat-band cable is measured with a time domain reflectometer (TDR) at high frequencies and with a low frequency impedance analyzer. Ice shows significant differences in dielectric properties at high and low frequencies, which enables the determination of the volumetric ice, air, and water content, as well as of the snow pack density using measurements at different frequencies. The cable may either be mounted sloping from a mast to the ground to measure vertical snow profile characteristics or be placed on the snow surface prior to a snowfall to measure the horizontal variations of the dielectric coefficient in the adjacent snow layer.

The objective of this paper is to present the basic principles of the sensor and to show its performance at a high-alpine test site in Switzerland (Weissfluhjoch, Davos) during two winter seasons.
2 Materials and Method

2.1 The Snow Cable Sensor

The sensor consists of a three-wire flat band cable with a plastic insulation as shown in Fig. 1.

![Insulated flat band cable](image)

Fig. 1 The insulated flat band cable (short section with uncovered conductor to show connection and geometry).

The total capacitance $C$ of the equivalent circuit is a non-linear function of the dielectric properties $\varepsilon_m$ of the surrounding material [1]:

$$C = C_1 + \frac{C_2 \varepsilon_m C_3}{C_2 + \varepsilon_m C_3}$$

(1)

The parameters $C_1$, $C_2$ and $C_3$ are measured (travel time and wave impedance) and calculated with electromagnetic field simulations.

2.2 Air Gap Correction

One of the challenging problems for continuous measurements in snow is the disturbing interaction of the sensor with the surrounding material. An air gap may form around the sensor due to mechanical influences (induced by wind) or partial melting. In order to detect and correct the effect of an air gap the following procedure is applied. The three-wire sensor cable is measured in two modes, with small and with large spacing between the conductors. In the large spacing mode, the air gap has less influence on the measurement of the dielectric coefficient than in the small spacing mode. This influence can be calculated and a correction algorithm can be derived [2].

In addition to the air gap correction algorithm, a snow profile reconstruction code has been developed [3] which allows the spatial determination of the liquid water content or the snow layer density (for situations without liquid water) along the cable sensor.

2.3 Field Test

The performance of the sensor system was tested during two winter seasons (2001/02, 2002/03) at the experimental site Weissfluhjoch, Davos (Switzerland), located at an altitude of 2550 m (Fig. 2). The site is snow-covered from late October to June, and the winter climate is harsh with minimum air temperatures of down to -25°C and high wind speeds. In both winters, four flat-band cables were set up: one of them sloping from a mast to the soil surface to measure vertical properties of the snow pack (snow depth, density profile, SWE), and three of them horizontally at different depths of the snow pack to capture the spatial variability of
liquid water content and snow density. These horizontal cables were placed on the snow surface at different dates of the winter and followed the natural settling of the snow pack.

Fig. 2 The Weissfluhjoch test site in January 2002.

The electronic measurement devices of the system (TDR cable tester ‘Tektronix 1502B’, impedance analyzer ‘HP-VNWA 8712’, PC) were placed in a shelter and connected with 50-m (first winter), and 35-m (second winter) coaxial cables to the flat-band sensor. For the validation of the snow cable measurements, snow profiles were excavated every 15 days in close vicinity of the sensor where the snow density was determined layer-wise. Also, an ultra-sonic snow depth sensor recorded the snow depth continuously and melt-water outflow from the snow pack was measured in a 2 x 2 m lysimeter next to the sloping cable sensor. Standard meteorological variables were monitored continuously throughout both winter seasons.

### 3 Results

#### 3.1 Suspension and Set-Up of the Sloping Cable

The original suspension, consisting of a wooden fastener attached with ropes to two iron springs, failed twice during the first winter: after a heavy wind storm in the second half of January, and towards the end of the winter season, when the snow load (maximum snow water equivalent: 740 mm) became too heavy and a spring broke. Consequently, an improved suspension was designed for the second winter consisting of a cylinder with a mechanical brake (Fig. 3), which kept a constant pressure on the cable. The suspension adjusted continuously when the snow load increased, by releasing the cable.

Fig. 3 Suspension of the snow cable sensor for winter 2001/02 (left) and winter 2002/03 (right).
The slope angle of the cable sensor is one of the key-factors for the formation of an air gap around the flat-band cable, as well as for the bending of the cable inside the snow pack.

In the first winter we set up the cable at an angle of 30° to the ground surface and tried to keep it in its original position by fixing it on 3 vertical poles. Such poles, however, proved to have negative impacts during the snowmelt by preferentially melting the snow and generating air gaps around the pole. Also a sharp bend of the cable formed at the head of the mid-pole after snow pack settling during the melting period (Fig. 4).

In addition, the poles were not stable enough to withstand the heavy snow load and bent considerably towards the end of the season. Consequently, for the second winter we decided to set up the cable at a steeper angle (45°) and to omit the supporting poles. The steeper installation reduced the snow load on the cable, and the cable was kept in its original position during the whole winter. However, we noted increased flickering of the cable in moderate to high wind speeds in the absence of supporting poles. These movements of the cable most likely generated the large air gaps observed at the surface, and may have caused a cable failure during a snow storm in January 2003. A further complication was a 90° twist of the cable around its own axes, which may have increased the water flow along the cable (Fig 4).

Fig. 4 Preferential melting around the supporting poles and formation of a sharp bend at the head of the pole in winter 2001/02 (left). Large air gap at the air-snow-interface of the twisted sloping cable in winter 2002/03 (right).

Apart from these problems we believe that the new suspension at a steeper angle has improved the installation, and could be further improved by the combination with springs to reduce the lateral movements of the cable.

3.2 Air Gap

Air gaps along the flat-band cable are highly undesirable since they not only falsify the electronic signal, but may also transfer melting water preferentially along the cable. The influence of an air gap on the measurement results can be partially corrected with a correction method [1], but with respect to a potential preferential water transport along the cable it was crucial to thoroughly inspect the size of the air gap and evaluate its importance as water flow path.

At both sites the air gap showed up in different sizes and shapes, depending on wind speed, wind direction and melting intensity (Fig. 5). The assumption of a symmetric air gap, which is made in the correction algorithm, was not appropriate for many situations.
Fig. 5 Observations of air gaps at the snow surface in the vicinity of the snow cable sensor.

There were dates (27 Feb, 2003; Alptal) where an up to 5 cm opening formed on one side of the band-cable, whereas the other side was in close contact with the snow. At the Weissfluhjoch site the air gap was mostly generated by wind. Hence, the compass orientation of the flat band cable was decisive for the shape of the air gap. We also observed that air gaps were able to fill up again when the wind conditions had changed. Our overall conclusion was that the shape of the air gap at the snow surface was very dynamic and unpredictable. Inside the snow pack, we didn't find evidence for significant voids around the cable when we excavated the flat-band cable at the sub-alpine site, Alptal, on 1 March, 2003. This was at a stage when the snow pack had settled to a density of 290 kg m$^{-3}$ and liquid melt water had penetrated to the bottom of the snow pack (Fig. 6).

Fig. 6 Dye tracer indicating the water flow paths during a melt event at the Alptal-site. (left) Front view at the cable end showing no dye tracer in close vicinity of the cable. (right) Vertical cross-section along the flat-band cable. Terrain slope-direction is indicated.

Close inspection of the flat-band cable was also carried out to examine whether the snow-band had conducted melt-water preferentially during the snowmelt. A dye tracer had been applied 15 days earlier (prior to the snowmelt) on the snow surface (area indicated in Fig. 6). A vertical profile was excavated along the cable and perpendicularly at the lower end of the cable. No stained water was detected at the lower end of the cable, whereas to the left and right of the cable end considerable stained areas were detected originating from lateral water transport along snow layers. There is evidence that some dye did follow the cable for some distance before finding alternative flow paths to the side of and below the cable, but we did not find indications of extensive lateral water flow expressed in terms of intensely stained
areas following the direction of the cable. We think that we can conclude in general that water flow along the cable is not greater than the natural lateral water flow in the snow pack. Concerning thermal effects of the sensor on the surrounding snow, we found some thermal radiation effects of a horizontal cable only once, shortly after it was laid out on the snow surface and the coverage by fallen snow was only very shallow. But as soon as the cable was covered with more than 10 cm of snow, this effect was not found anymore.

3.3 The Electronic System

With the exception of some weeks in December 2001, when an erroneous setting prevented the impedance analyzer from running, and in April 2002, when a break down of an internal electronic component forced us to replace the impedance analyzer, the electronic system ran more or less failure-free throughout the first winter season. The system uptime was high also in the second winter (80%). The automatic start of the measurements occasionally failed at the interface between the controlling PC unit and the Tektronix. Since this happened mainly at very low temperatures, we think this is a temperature problem of the electronic components of the Tektronix device, which was not built to operate under such low temperatures.

3.4 Comparison of Snow Sensor Measurements with Manual Measurements

Dielectric constant, bulk snow density, and liquid water content of snow pack (mean value along the cable) were calculated from the raw signals. And the moisture distribution along one of the horizontal cables was reconstructed for different stages of the winter. The natural settling of the snow cover was reflected nicely in the horizontal cable measurements (Fig. 7).

Fig. 7 Measured density along the two horizontal cables during winter 2001/2002. Dots show manually determined density of the corresponding snowpack layers. Due to a break down of the impedance analyser no data were available from late March to early May.

The snow density increased from initially 100-200 kg m\(^{-3}\) to approximately 500 kg m\(^{-3}\) at the end of the winter season, which was in accordance with manual snow density measurements of the corresponding snow layers. The two cables located at two different depths of the snow pack also reproduced correctly a higher density in the lower part of the profile in February
(cable 1) and a faster compaction in the upper part during the snowmelt (cable 2). However, cable 2 seems to have systematically underestimated density, which we think may be explained by spatial differences in the measurement spots of cable and manual density measurements.

![Graph showing liquid water content and accumulated lysimeter outflow.](image1.png)

**Fig. 8** Liquid water content (average 2 horizontal cables) during melting period of spring 2002. Comparison with accumulated melt-water outflow from the lysimeter.

No liquid water was detected with the horizontal cables until the end of April, after that the snow pack had reached its maximum depth (Fig. 8). This is supported by the automated snow temperature measurements, indicating dry snow conditions and temperatures below zero before. Once the snowmelt set in, liquid water content steadily increased indicating the downwards penetration of the wetting-front. The upper cable (no. 2) not only reflected an earlier and faster increase in liquid water content than the lower cable, it also reacted more clearly to the diurnal variation caused by melting during the day and refreezing during the night.

![Graph showing spatial variability.](image2.png)

**Fig. 9** Spatial variability of liquid water content along horizontal flat-band cable 2 on 13 and 17 May 2002.
With regard to the spatial variation of liquid water, the horizontal flat-band cable nicely demonstrated the formation of preferential water flow paths adjacent to the cable (Fig. 9), which is the natural process of water transport in a melting snow pack [4]. The horizontal cable at about 1 m below the snow surface indicated in mid May an emerging water conducting zone at 14 m from the cable end, as well as a newly developing flow finger at 8 m from the cable end.

4 Discussion and conclusions

The measurement results yielded good correspondence of the snow pack density with manual reference measurements taken twice a month at the measurement site. Also, the determination of liquid water content from measurements of the dielectric constants according to [5] gave plausible results, both compared to lysimeter data taken on the test field and with regard to the spatial variation of flow fingers that we normally experience in a natural snow pack.

The device’s suitability for these measurements has been demonstrated, but there is a dire need for improvement of the instrumentation set-up to withstand the harsh conditions of alpine winter seasons. This will be a task for future improvements.

We learned that the suspension of the cable sensors is a crucial point in the measurement. For successful measurements it is indispensable to reduce the following sources of error:

- Air gaps between cable sensor and surrounding snow must be minimized, although it is possible to correct for minor effects.
- The suspension of the cable sensors must allow the cable to settle with the snow pack, but at the same time must be robust enough to withstand heavy winds and snow loads.

References


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Comparing Time Domain Reflectometry and Electrical Resistivity Tomography Measurements for Estimating Soil Water Distribution

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ABSTRACT. Two different methods to determine the spatial distribution of soil water were tested and compared on a full-scale dike model. TDR-measurements of the spatially distributed water content along transmission lines were compared with electrical resistivity tomography (ERT) measurements along the same cross-section. In contrast to the TDR method, ERT is a non-invasive method and does not require the perturbation of the dike’s interior for the measurements. However, determining the soil water content from the resulting electrical resistivity values is more difficult than in case of the dielectric coefficients using TDR. Field data were obtained during a controlled simulation of an extreme precipitation event in the context of water balance investigations. A comparison of both methods shows similar results, except for differences in spatial resolution.

Keywords: spatial TDR, geoelectric, dike model, comparison of methods

1 Introduction

Geophysical measurement methods are essential elements for monitoring systems. Especially storage buildings like earth dams have to be observed, because of their high exposure. For this purpose, pressure gauges are used normally. Increasingly fibre optical temperature measurements are implemented for example to localise temperature anomalies indicating leakages in sealing systems [1]. However, in many cases the geotechnical engineer is interested in quantitative water content distributions characterising the hydraulic situation within earthen structures. Based on this information it is possible to predict the hydraulic behaviour of an earth dam depending on hydraulic loads. Such continuous moisture measurements have been realised on a full-scaled dike model using Time Domain Reflectometry (TDR) for the first time [2]. In this experiment, the spatially distributed water content along expanded transmission lines was interpolated between the measurement profiles. However, the electrical field triggered by TDR-signals is limited to a small range around the transmission line, why local anomalies in some distance stay undetected.

One possibility to determine the fully two-dimensional spatial distribution of moisture in earthen structures is the electrical resistivity tomography (ERT) method, which is often used in combination with exploration drillings in order to determine the structural composition of dikes and subsoil [3]. Apart from many further applications in environmental, engineering and archaeological studies ERT has been successfully used to monitor the seepage in an earth embankment dam [4].

The following contribution shows a comparison of both systems (TDR and ERT) during a sprinkler irrigation test on a full scaled dike model. The aim of the combination of both systems is to improve the interpretation of 2-dimensional water content distribution.
2 Methods

2.1 Properties of Soils

Soil is a typical porous medium consisting of the three phases pore air, grain and pore water (cf. Fig. 1). The fractions of the soil phases vary both in space – due to composition and density of soil – and time – due to changing water content. For the determination of the water content of the soil, one utilise the fact that total permittivity and total resistivity are dependent on the fractions of the soil phases. A straightforward but laborious way to find the relation between water content and total permittivity or resistivity is to perform a specific laboratory calibration with gravimetric sampling. This is often impractical for operational use. Therefore, several empirical, semi-empirical and theoretical mixing rules with different degrees of experimental justification have been developed and applied. One of the commonly used empirical equations for the determination of the permittivity was given by [5]. A mixing rule depending on the bond form of water was developed by [6]. The most popular mixing rule for the determination of the resistivity was given by Archie [8].

Fig. 1 Electric (resistivity $\rho$ [Ωm]) and dielectric (permittivity $\varepsilon_m$ [-]) properties of soil phases

2.2 Time Domain Reflectometry (TDR)

For TDR measurements a fast time voltage step is launched into a transmission line and the reflections are recorded (e.g. with an oscilloscope). The length of standard, non-insulated metallic forks as transmission lines are usually restricted to 30 cm because of high damping losses. For longer transmission lines, a new insulated flat band cable was developed [7, 9]. It shows much less pulse attenuation than non-insulated metallic forks in the same media. The flat band cable is shown in Fig. 2 together with its electrical field distribution in the cross-section for excitation in air. The cable consists of three copper wires covered with polyethylene insulation. The electrical field concentrates around the conductors and defines a sensitive area of 3
to 5 cm around the cable. For the calculation of the permittivity from the determined capacitance of the TDR measurement, a special capacitance model was developed [7].

Fig. 2 Flat band cable and electrical field distribution in the cross-section for symmetric excitation in air

The standard TDR measurement procedure based on travel time analysis delivers only the mean water content along the transmission line. As the TDR response contains far more than the travel time of the reflected electromagnetic signal, a three-step algorithm has been developed to reconstruct the soil moisture profile from the full signal response along the transmission line [10]. This new TDR measurement system consisting of recorded step pulse with spatial analysis has already proved its effectiveness in different applications [2, 11, 12] and is introduced as spatial TDR.

2.3 Electrical Resistivity Tomography (ERT)

The ERT technique is based on electrical resistivity differences between different subsurface materials. With the development of fast, commercially available 2-dimensional inversion schemes for tomographic data sets, the DC resistivity method has been increasingly applied, especially for environmental and engineering problems [e.g. 4, 13, 14].

In DC resistivity surveys electrical current is injected into the ground via two current electrodes. The resistance of the ground is then determined by measuring the electric potential between two other electrodes and dividing by the current. By multiplying the resistance with a geometric factor depending on the distance between the electrodes and choosing different electrode spacings and locations, the so-called apparent electrical resistivity is determined on a 2-dimensional grid. By using a tomographic inversion scheme (RES2DINV) these apparent resistivities can be inverted to yield a 2-dimensional specific resistivity model of the ground [15]. For monitoring purposes, these measurements are repeated at certain time intervals using a permanently installed electrode array. The permanent electrode array effectively filters resistivity variations due to variable electrode contacts or geological background variations. By this, temporal resistivity changes can be related to changes in the subsurface water content on a 2-dimensional or even 3-dimensional grid.

An estimate of the water content of the ground through repeated resistivity measurements can be obtained by using a simple approach based on a well-known relation between the resistivity of a material and its pore fluid called Archie’s law:
\[ \rho = a \rho_w \Phi^{-m} S_w^{-n} \] (1)

where \( \rho \) is the resistivity of the material, \( \rho_w \) is the resistivity of the water in the pore spaces, \( \Phi \) is the porosity, \( S_w \) is the fraction of the pore space occupied by liquid water (i.e. the saturation) and \( a, m \) and \( n \) are empirically determined parameters [8]. By taking the ratio of the observed resistivities of subsequent measurements \( t_1 \) and \( t_0 \) the resistivity of the pore water \( \rho_w \) and the material parameter \( \Phi, a \) and \( m \) can be eliminated, giving

\[ \rho(t_1)/\rho(t_0) = S_w^{-n}(t_1)/S_w^{-n}(t_0). \] (2)

The evolution of the saturation \( S_w \) at a later time \( t_1 \) can now be calculated using the initial saturation \( S_w(t_0) \) and the ratio of the observed resistivities

\[ S_w(t_1) = S_w(t_0) \cdot (\rho(t_0)/\rho(t_1))^{1/n}. \] (3)

King et al. estimated the so-called saturation exponent \( n \) between 2-3 (for sands) and 5-8 (for clays) [16]. The water content as determined by the TDR cables can then be calculated by multiplying the saturation \( S_w \) with the porosity \( \Phi \) of the material.

3 Dike Model and Instrumentation

3.1 Dike Model

The transient progression of the seepage within a dike body during a temporary flood event depends among others on the initial soil water content of the earth material. In this connexion, the initial soil water content is the result of meteorological and hydrological water balance processes affecting the dike body. For the investigation of the hydraulic behaviour of dikes as earthen structures, a full-scale dike model is available (cf. Fig. 3).

Fig. 3 Full-scale dike model at the Federal Waterways and Research Institute in Karlsruhe during a flood simulation test in December 2000
3.2 Instrumentation

The dike model is permanently equipped with 12 vertically installed flat band cables from 1 to 3 m in length, connected from both sides with coaxial cables to a multiplexer and TDR device in a box on the crest of the dike. The data collection and controlling equipment (PC) of the system is located in a measuring container at the toe of the landside slope (cf. Fig. 4). ERT measurements were conducted manually from the crest of the dike using a conventional resistivity meter (ABEM Terrameter). For this, 23 resistivity electrodes were distributed along the top ground surface (cf. Fig. 4).

Fig. 4 Location of the flat band cables and the resistivity electrodes in the cross-section of the dike and schematic description of the TDR measuring system.

4 Results

Fig. 5 shows the results of TDR measurements as distribution of saturation (ratio of volumetric water content and porosity) from two time instances before and after the end of an irrigation experiment. The results after the irrigation show saturation values of around 30% at the surface, which is decreasing with depth to values below 20% in the dike interior. Maximal values are found at the surface about halfway down the dike’s flank, where the water input during the irrigation experiment was maximal.

In contrast to the TDR results, the ERT results of Fig. 6 depict not directly the water content or saturation but the electrical resistivity (in $\Omega$ m). In homogeneous material low resistivities (dark shading) can be associated with high saturation and high resistivities (light shading) with low saturation. As the ERT method is surface-based, measurements had to be conducted across a small stone path on top of the dike crest (cf. Fig. 3), which is clearly seen by the anomalously high resistivity in this region.

In dry state resistivities are lowest along the surface and increasing to greater depths. Maximal resistivities are found at the bottom in the central part of the dike, which is in good agreement with the corresponding low water contents ($< 20\%$) in the TDR results. After the irrigation resistivities are markedly lower throughout the whole dike’s interior and lowest (corresponding to maximal water contents) around halfway down the flank, where the water input during the experiment was maximal. Again, this is in good agreement with the corresponding high water contents visible in the TDR results.
Fig. 5 Result of TDR measurements presented as saturation (top) in dry state and (bottom) after an irrigation experiment.

Fig. 6 Result of ERT measurement represented as resistivity in $\Omega\text{m}$ (top) in dry state and (bottom) after an irrigation experiment.
For a quantitative comparison between the two methods, the distribution of saturation at a later time instance (5 hours after the irrigation) is calculated from the resistivity results using Equation (3). Fig. 7 shows a comparison between the calculated ERT results (left) and TDR (right). Apart from the crest of the dike, where the presence of the stone path leads to differences between the in-situ method (TDR) and the surface-based method (ERT), both measurements are in good quantitative agreement.

5 Summary and Conclusion

We presented a comparative study using spatial TDR and electrical resistivity tomography (ERT) to determine the spatial and temporal variability of water content distribution in a full-scale dike model. Whereas spatial TDR determines the in-situ water content distribution along up to 3 m long flat band cables by analysing the full signal response and relating it to the dielectric properties, the ERT system determines the 2-dimensional electric resistivity distribution from surface-based geoelectric measurements and subsequent data inversion. TDR and ERT results from experiments before and after a sprinkler-irrigation test were shown.

Both methods show good agreement, both, concerning the spatial variability before, as well as concerning the temporal evolution during and after the irrigation experiment. The advantages of the spatial TDR system are the high spatial resolution near the sensor cable and the well-established relation between dielectric parameters and water content. The advantages of the ERT approach are the full 2-dimensional representation of the parameters in the dike's interior and the non-invasive character of the measurements. A combination of both methods, e.g. a
calibration of the ERT approach using spatial TDR and large-scale measurements using the surface-based ERT, seems promising for future earth dam monitoring systems.

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Detecting and Monitoring Frozen Ground and Unfrozen Water Content Using Electric and Electromagnetic Techniques

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ABSTRACT. A series of different electric and electromagnetic techniques were applied in high mountain and polar regions within a number of projects since 1998. The aim was to determine suitable methods to detect, characterise, map and monitor permafrost in the context of possible slope instabilities resulting from climate induced thawing of permafrost bodies. The applied techniques include electrical resistivity tomography, frequency-domain and time-domain electromagnetic induction methods and ground-penetrating radar. Various inversion algorithms and model approaches were used to relate the observed data set (the electrical resistivity or its reciprocal, the electrical conductivity) to the subsurface properties of interest, namely the ice content or the unfrozen water content. Resistivity changes were related to borehole temperature changes and the evolution of the unfrozen water content could be determined at different depths. As the unfrozen water content is a key parameter concerning slope instabilities induced by thawing permafrost, this approach is considered a promising contribution to future monitoring studies.

Keywords: geoelectric methods, electromagnetic methods, permafrost, ground ice, monitoring

1 Introduction

In the Earth’s climate system, the cryosphere plays a special role, due to its sensitivity to even small climate changes. Within the cryosphere permanently frozen ground (permafrost) in mountainous regions is specifically vulnerable, as temperatures are often close to the melting point of ice. In view of a warming climate and the recent retreat of most Alpine glaciers, the need for a continuous monitoring of the permafrost evolution in mountainous regions has been identified [1]. This includes long-term temperature monitoring programmes (such as the EU-funded PACE (Permafrost and Climate in Europe) project, 1998-2001), as well as improved process understanding and impact assessment of permafrost thawing in the mountains of Europe. Specifically important are thawing permafrost slopes, which may induce natural hazards such as rock falls and debris flows [2].

Contrary to glaciers, ground ice occurrences in permafrost regions are invisible from the surface. In order to detect permafrost, as well as understand the processes associated with freezing and thawing of the subsurface, suitable methods have to be determined to characterise and monitor ground ice occurrences [3]. Permafrost occurrences in mountain regions are highly variable and depend strongly on altitude, incoming radiation, local climate and geology. The heterogeneous permafrost distribution calls for methods, which are able to resolve the shallow subsurface at horizontal scales between a few metres and 1 km and vertical scales of 1-50 m. Drilling in mountainous areas is costly and often logistically difficult or impossible, which excludes the application of most in-situ methods on larger scales.

Between 1998 and present a large number of geophysical surveys were conducted on mountain permafrost terrain in order to evaluate the applicability of the various methods for permafrost related problems. Some examples are presented in the following.
2 Methods

A large variety of electric and electromagnetic techniques is based on changes of subsurface resistivity $\rho$, its reciprocal, the electrical conductivity $\sigma$ or the permittivity $\varepsilon$. A marked increase in $\rho$ and $\varepsilon$ at the freezing point was shown in several field and laboratory studies [e.g. 4]. For many soils, the resistivity increases exponentially until most of the pore water is frozen. The resistivity is reduced for saline pore waters, as the freezing point is depressed and the unfrozen water content at subzero temperatures is increased. Generally, the resistivity values depend mainly on the material type and the unfrozen water content in the sample [5]. Due to the presence of four phases within the frozen material (rock/soil matrix, unfrozen water, ice, air), the respective volumetric fractions of the phases in the subsurface cannot be quantified by using one method alone. Using a combination of electric and seismic methods and suitable mixing rules, the respective volumetric fractions of each phase (e.g. the ice content) can be approximated [6].

2.1 Electrical Resistivity Tomography (ERT)

The ERT technique is based on electrical resistivity differences between different subsurface materials. In contrast to most applications of ERT in environmental and engineering studies (see e.g. Hauck & Scheuermann, this volume), mountain permafrost targets can be highly resistive (> 5-10 k$\Omega$m to several M$\Omega$m). In spite of the resulting difficulties in getting sufficient electrical current into the ground, the method was successfully applied to map and characterise different permafrost structures in mountains terrain [7, 8, 9, 10, 11, 12].

In ERT surveys electrical current is injected into the ground via two current electrodes. The resistance of the ground is then determined by measuring the electric potential between two other electrodes and dividing by the current. In choosing different electrode spacings and locations and applying a tomographic inversion scheme (RES2DINV, [13]), the observed apparent resistivities can be inverted to yield a 2-dimensional specific resistivity model of the ground. For monitoring purposes, these measurements are repeated at certain time intervals using a permanently installed electrode array, which allows for measurements independent of the snow cover thickness. Furthermore, the fixed-electrode array effectively filters resistivity variations due to variable electrode contacts or geological background variations, as mainly temporal resistivity changes are determined [10].

Fig. 1 shows an example from the Italian Alps, where isolated permafrost occurrences are presumed at an altitude of about 2700m a.s.l. [14]. The left panel shows the inversion results of a 2-dimensional ERT survey along a 200 m line. Two regions of higher resistivity (ca. 10 K$\Omega$m) can be identified on the upper and lower ends of the profile. Due to the fine-grained characteristics of the surface cover (which excludes the presence of high-resistive rock outcrops), and additional seismic evidence the causes of these high-resistive anomalies are believed to be subsurface ice occurrences. The vertical extent of the anomalies is between 10 and 20 m. Below and throughout the centre of the profile, resistivities of less than 2 K$\Omega$m are found indicating unfrozen material.
2.2 Electromagnetic Induction Mapping

Electromagnetic (EM) induction techniques measure the electrical conductivity, with the important difference to ERT, that no direct (galvanic) contact with the ground is needed. This is a great advantage in high mountain environments, as getting sufficient electrical current into the ground is one of the largest problems in ERT surveys. Furthermore, ERT surveys in wintertime are usually impossible to conduct, as a dry snow cover acts as an electrical insulator. EM induction methods employ a magnetic field to induce the electrical current in the subsurface. Therefore, there is no need for direct electrical contact between the transmitter and the ground.

EM induction is based on the principle that circular, concentric magnetic field lines surround each current-carrying wire. If bent into a small loop, the wire produces a primary magnetic dipole field. In EM induction this dipole field is varied, either by alternating the current (operating in the frequency-domain (FEM method)) or by terminating it (transient methods), operating in the time-domain (TEM, see next section). This time-varying magnetic field induces very small eddy currents in the Earth. The eddy currents generate a secondary magnetic field, which may be sensed at a receiver loop at the surface. The more conductive the subsurface, the larger are the eddy currents and the larger is the measured secondary field, which in turn allows the ground conductivity to be determined by a simple proportional relation. No further data processing is required.

Due to the fast and simple application, FEM systems are often used for conductivity mapping of larger areas. Hereby, only the spatial variability of the bulk conductivity of the uppermost subsurface layer is determined without any depth information [e.g. 3, 14].

The right panel in Fig. 1 shows an example of FEM conductivity mapping with the one-person portable EM-31 (Geonics). The survey was conducted within the area of the ERT survey (black line) to determine the spatial representativeness of the resistive anomalies found in the ERT results. The results indicate that the distribution of the anomalies shown in the left panel is almost parallel to the altitudinal lines. In the upper part (in the southern part in Fig. 1 (left) ERT survey results of a partly frozen slope near Stelvio pass, Italian Alps. White shading marks areas with high electrical resistivity corresponding to possible ground ice occurrences. The root-mean-square error (RMS) of the inversion results is given in the left corner. (right) FEM conductivity mapping of the same area. The black line marks the location of the ERT survey line. White shading marks low relative electrical conductivities corresponding to possible ground ice occurrences.
1), above the DC resistivity line, relative conductivities decrease even more, which may indicate a larger ground ice content. Note, that the Geonics EM-31 measures the bulk conductivity of the uppermost 6m, so that no depth information can be obtained.

2.3 Electromagnetic Induction Sounding

EM induction sounding, that is the determination of vertical conductivity variations, is commonly conducted with transient electromagnetic systems (TEM). Contrary to FEM, TEM instruments measure the induced secondary magnetic field in the transmitter-off periods, as the primary magnetic field is not alternated, as in FEM, but terminated. The response of the subsurface in terms of the decaying amplitude of the secondary magnetic field can then be measured as a function of time and therefore of depth, because later responses originate at greater depths.

In TEM surveys, different measurement configurations are utilised. Central loop soundings, where the receiver coil is located in the centre of a large transmitter coil (usually 40m x 40m or 100m x 100m) or outside configurations, where the receiver is placed outside the transmitter loop to reduce the noise level through primary field effects have been employed [15, 16]. On mountain slopes or rock glaciers, such large transmitter loops are often impossible to use due to the blocky and irregular terrain. Alternatively, a flexible eight-turn 5m x 5m transmitter loop can be applied, which allows for reasonably easy handling with a still sufficient penetration depth [14].

Fig. 2 shows an example from rock glacier Muragl, Swiss Alps, where extensive glaciological, geophysical and geotechnical studies have been performed to assess the stability of degrading mountain permafrost slopes [e.g. 17]. The left panel shows the measured (squares) apparent resistivities as a function of measurement time (in ms). The solid line shows the modelled apparent resistivities corresponding to the 1-dimensional resistivity model shown in the right panel. The results indicate an approximately 10m thick unfrozen surface layer ($\rho < 1000\Omega m$) and a 20m thick frozen layer ($\rho > 50000\Omega m$) above the bedrock layer ($\rho < 10\Omega m$), which is in good agreement with complementary geophysical and borehole data [17].

2.4 Ground-penetrating Radar (GPR)

GPR has been successfully used to study the permafrost distribution and structure in the Arctic and Antarctica during the last few years [e.g. 18]. Similar to the reflection seismic method, an energy pulse is directed into the ground and the arrival times of reflections from subsurface interfaces are recorded. Data interpretation is simplest when the ground consists of vertically stacked layers of homogeneous, non-dispersive dielectric materials. Attenuation is highest (and therefore penetration lowest) in low-resistive materials (e.g. materials with a high liquid water content) and particularly in fine-grained sediments (even when frozen), where penetration depths can be less than 1 m. Because of this, and the high dielectric contrast at the frozen surface, GPR is best suited for obtaining detailed information on the geometry of the unfrozen top layer in summer and on ice-rich permafrost occurrences. Data processing can be rather complex, however as the method works analogous to the well-known seismic reflection technique, commercial processing software is readily available.

Due to the heterogeneous subsurface and surface conditions in mountainous terrain, GPR surveys are less suited for mountain permafrost occurrences. A low signal-to-noise ratio arises
from complex reflections within blocky layers, which masks the true layer horizons. Nevertheless, the depth of mountain permafrost structures could be detected in a few cases [19, 20].

Fig. 2 TEM sounding data (Geonics PROTEM) recorded across Muragl rock glacier, Swiss Alps and the corresponding 1D resistivity model. The left panel shows measured (squares) and modelled (line) apparent resistivity values. Right panel shows derived resistivity model.

3 Resistivity Monitoring Schilthorn/Swiss Alps

The Schilthorn (2970 m) is located in the Bernese Oberland in the Northern Swiss Alps. The investigation site is located on a small plateau on the north facing slope with a surface consisting of small to medium size debris originating from weathered bedrock with no vegetation cover. Due to a high amount of precipitation and additional snow input through wind transport, the snow cover persists usually from October to June. Presence of permafrost has been found within three boreholes (14m in 1998, 100m and 100m at an angle of 30° in 2000) drilled within the PACE project [2]. The observed permafrost temperatures are comparatively warm, reaching -0.7°C at 14 m depth. Consequently, the ice content is low and the unfrozen water content is high leading to low resistivity values compared to typical mountain permafrost occurrences.

A fixed-electrode array allowing repeatable resistivity tomography measurements along a 58 m survey line throughout the year was permanently installed at Schilthorn in September 1999 [10]. The 30 stainless steel electrodes were buried 1 m into the ground. Cables were connected to a manual switchbox, which is accessible throughout the winter, and were buried in case of avalanches. Resistivity surveys were made by connecting an OYO McOhm resistivity meter to the switchbox for each of the selected electrode configurations.

Between September 1999 and present, a number of monitoring studies on daily, seasonal and annual time scales have been conducted. Fig. 3 shows an example from 1999-2000, where the seasonal variation of the resistivity in the uppermost 10 m was determined. Instead of analysing the resulting resistivity tomograms in terms of absolute values, the cumulative resistivity differences per day based on the first September measurement (Fig. 3a) are shown. Largest resistivity increases (white colours) were observed in October, when the snow cover
was not yet established and cold temperatures could penetrate into the subsurface (Fig. 3b). Freezing extends along the whole survey line and reaches a depth of 2 m. From October 1999 to April 2000, resistivities increased only slowly due to the insulating snow cover, which arrived in October and effectively decoupled the subsurface thermal regime from the atmosphere. During the phase transition the temperatures remain close at 0°C (the so-called zero-curtain effect), while the resistivities increase, as the unfrozen water content is diminished.

Fig. 3 (a) Resistivity model for the measurement on 15.9.1999 as determined by the inversion. (b)-(i) Resistivity difference per day based on the September measurement (a). White and dark shading denote resistivity increase and decrease, respectively.

At the time of the first resistivity measurement after the snow melt (June 2000), most of the frozen water in the uppermost 2-3 m had already melted which led to a strong resistivity decrease near the surface (grey colours in Fig. 3g). This decrease continued until end of August 2000, hereby almost totally equalising the resistivity increase of the winter months (Fig. 3i). A more detailed analysis of the resistivity results can be found in [10].

Laboratory experiments were conducted to relate resistivity variations to changes in the unfrozen water content. Fig. 4a shows two curves for unsaturated and saturated material from the Schilthorn site. For temperatures above the freezing point, resistivity increases linearly but small with decreasing temperatures due to the diminished mobility of the ions in the pore fluid. For temperatures below the freezing point, the resistivity increases exponentially with decreasing temperature. From the form of the curves, exponential relationships between resistivity and temperature can be determined. The exponential increase is larger in the saturated experiment than for the unsaturated material, which is in good agreement with previous studies [21, 22].

Fig. 4b shows the evolution of the unfrozen water content, which was calculated using the resistivity results of Fig. 3 and the relationships determined by the laboratory experiments using an approach by [10]. In the uppermost layer (0.5 m) the unfrozen water content starts to
decrease at the end of October. The minimum is reached in February and subsequently later at greater depth (beginning of June at 8.7 m depth). At larger depths, the evolution is nearly sinusoidal, corresponding to the seasonal variation of ground temperature.

Fig. 4 (a) Resistivity-temperature relationship determined in the laboratory for two different saturated and unsaturated Schilthorn samples. (b) Evolution of the unfrozen water content at different depths using the relationships determined by the laboratory experiments as shown in [10].

4 Summary and Conclusion

An overview of electrical and electromagnetic methods used in the study of permafrost is presented. The aim was to find suitable methods to detect, map, characterise and monitor ground ice occurrences in the context of climate induced permafrost degradation and its impact on slope instability.

The methods include electrical resistivity tomography (ERT), frequency- (FEM) and time-domain (TEM) electromagnetic induction methods as well as ground-penetrating radar (GPR). ERT turned out to be a suitable method for most permafrost related questions, such as detection and monitoring of ground-ice occurrences. Measurements with electromagnetic induction methods included permafrost mapping of larger areas (FEM) as well as vertical sounding to determine the permafrost base (TEM). GPR is best for determining the internal structure of layered permafrost bodies, as well as for detecting the spatial extent of individual layers. However, in case of a conductive overburden, penetration depth can be shallow. If possible, several geophysical techniques should be used together (including seismics), as results from only one method may yield ambiguities concerning the interpretation of subsurface anomalies. Finally, resistivity monitoring in combination with calibration curves can be used to determine the evolution of the ground ice unfrozen water content, respectively. This approach seems promising for future monitoring systems to assess the potential danger from thawing permafrost slopes in mountainous regions.

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Improved Process Control of the Water Content in Biological Filtration Plants

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ABSTRACT. This paper presents an improved system of supervising and control the content of water in biological filtration plants by using strategies of computational intelligence (CI). Biological filtration plants are used to realize a biological waste gas detoxification. Optimal waste gas decomposition can only be realized by optimal living conditions of the micro-organisms in the filter plant. Improvements are achieved by application of methods from neural network, sensor fusion in combination of a microwave moist meter and a pressure sensor to provide an enhanced measurement. This kind of data mining allows measurements of process data’s and states that would otherwise not be observable.

Keywords: biofilter, moist, control, neural network

1 Introduction and Background

The waste gas detoxification with biological filter plants (Fig. 1) is state of the art in most fields, especially when cleaning bigger volume streams at low costs. The advantage of biological waste gas detoxification is the complete removal of harmful substances without using chemical components. Based of microbial metabolism the micro-organisms convert the pollutant substances of the waste gas into the harmless decomposition products water (H$_2$O) and carbon dioxide (CO$_2$). The process is safe, natural and non-hazardous. The living conditions of micro-organisms must be optimized in biological filters. Especially concerning the temperature in the filter plants and the content of water. So it is very important to lead this process optimally by the determination of the water content in the plant. The biological system is optimized, if the water content in the filter is kept within a threshold sector between 40 and 70\%. For this purpose, most filter plants use humidifiers to moisturize the substratum. This substratum is an organic and very heterogeneous material. So it is very difficult to measure the moisture content of this substratum.

Fig. 1 Block scheme of a Biofilter plant

The measurements were performed with microwave probes, MOUIST BIO, (hf sensor GmbH Leipzig). The measurement equipment represent a new type of sensors for the test and non-destructive measurement of water content in bulks, with a good volume expansion, permitting
a continuous measurement without taking samples. Microwave moisture measurement systems
typically measure specific points of water content. In order to control the water content for the
biological filter it is necessary to measure the moisture of the complete substratum. This value
is called “Middle Water Content at the Complete Volume” (MWC). This value depends on many factors, for instance the relative atmospheric humidity and
temperature of the waste gas, air current of the waste air and bigger time constants. These
factors do not have linear interrelationships. That is why it is not possible to use mathematical
models or statistic methods. In the past few years computational intelligence procedures –
besides classical methods or to complement such – have gained increasing significance in
modelling applications. Fuzzy control (FC) systems or applications of artificial neural
networks (KNN) are the state-of-the-art. CI methods have become especially indispensable in
finding solutions in bioprocess engineering-related process classification, modelling or
control, due to grossly non-linear interrelationships of process variables often in connection
with no or hardly existing process expertise.

2 Objectives

The Objectives are: (i) to measure online of the moisture content in a complex inhomogeneous
biological substratum; (ii) to develop a model for the determination of the volumetric water
content based on an artificial neural network (iii) to implement the model in a microcontroller
board as a run-time-C-code and (iv) to realize a cost efficient and robust microcontroller
based process control system.

3 Experimental Set-up and Model Development

3.1 Microwave Instrumentation

The basic construction of such an applicator is shown in Fig. 2 and Fig. 3. The sensing head
consists of three main parts: an applicator case designed as a grip - comprising the analogue
and a part of the digital electronic -, a stainless steel tube with diameter 20 mm and the active
microwave structure. The microwave structure is surrounded by a dielectric cover shaped as a
tip, which protects the structure against mechanical stress and pollution. The material of the
sensing head consisted of TK-Peek and has to be resistant to chemical and mechanical stress
and must work electrically stable.

The possible sample materials have various consistencies: they can appear as shred, chips,
fibres or fibre bundles, mats, bulk goods of various particle size, etc. Corresponding to the
structure of the inhomogeneous materials is inevitable and the sensing head won’t be each
time in good contact to the material. Therefore the interaction of the electromagnetic wave
with the organic material must comprise a large sample volume and has to happen omni
directional over an extended aperture in comparison to the mean particle size in the medium.
The dielectric cover matches the antenna impedance to the impedance of the surrounding
medium and makes the electromagnetic interaction tunable in a way that a good average of
the whole moisture range of interest is possible.

For the microwave measurement of dielectric material parameters or water content
transmission and reflection methods can be used.
A good possibility to enlarge the interaction volume is given by the use of radiating resonators or antenna structures [1]. From these structures in addition to the near field interaction, which is the same as for open resonators, electromagnetic energy is transported into the medium and transformed into heat by dielectric losses. This raises the measuring effect. A limiting condition to the use of these kind applicators is the large penetration depth of the electromagnetic wave. The sample volume has to be large enough to avoid reflections from the samples edges. For the analysed microwave structures the minimum sample volume was in the order of 30...100 litres. This requirement has to be kept in mind for calibration of the applicator on a special material.

A dipole has an almost ideal field distribution for the use in a penetration applicator, but the mechanical fixing and the feed at the high impedance termination were considered to be problematical. Therefore first structures investigated were monopoles, but problems in reducing the necessary ground area to the diameter of the applicator made it impossible to use this antenna type. Experiments with radial symmetrical arranged \( \lambda \)-resonators (Reuse) made of metal rods fed at their ends and coupled by short tube segments also were not satisfying.

A solution for the problem of mechanical fixing was found in the combination of a half wave dipole and a quarter wave Sperrtopf. This arrangement is known as Sperrtopf antenna [1] and allows by shifting the feed point at the inner conductor matching of the feed point impedance to the feeding coaxial cable. To reach a resonant frequency of 2.45 GHz the dielectric cover of the applicator has to be taken into consideration for the design.
Test measurements were made in various biological substances, e.g. biofilter-material, compost, grain.

Measured data in Fig. 4 showed that microwave moisture measurement in difficult materials such as compost or biofilter-material is possible with the designed applicators and gives a sufficient accuracy for process control. The designed penetration applicators can also be used for long term on line measurements.

3.2 Data Generation

The measuring hardware is realized on a PC with a measuring board (ADC) to measure data of sensors (one sensor to measure the pressure and three (or more) sensors to measure the water content of many places). It is also possible to use sensors with a serial interface (for instance RS232) and connect they to the COM interface of the PC.

The measuring process is realized on the PC by the software DIAdem® (National Instruments). This software covers the measured values and evaluates the data over a long time. The collection of a large number of representative data is very important for a successful design of the artificial neural net. The measurement software DIAdem® allows programming an automatic evaluation of the measuring values to get prepared data with following focal points:

- calibration the sensors with approximation
- automatic erasure of non-relevant data
- percolation of signal spikes
- more smoothness of the measuring values
- data transfer in normalized values

Preliminary examinations, which are realized for a long time (1 year), indicate that there is sufficient correlation between pressure (pressure sensor DIGMA premo, SI-special instruments Nördlingen (Germany)) and water content in the filter plant. Moreover special correlations were found, proved by measuring and discussions with experts:

- if the water content increases in normal filter operation, the pressure in the filter will increase too
- low water content and high pressure indicate a water tailback in the filter plant
- high water content and low pressure indicate local drying-out of the filter (both cases might have been generated by an error in the sensors (“Error A” “Error B”) as well)
3.3 Design of a Classification and Control Model

The model design based on the software tool EmCoSoft (Embedded Softcomputing) developed by our team [2, 3]. It consists of the sub-tools „Model Builder“ and „Application Builder“ and enables the realization of the complete algorithm. Fig. 5 shows the structure of this software system. All sub-functions are displayed for the user block-oriented via a graphical user interface. Its integrates also all relevant sub-aspects from process signals via data mining to process model and control algorithm with signal output. A further key issue is the export of the monitoring or control algorithm, developed on a PC by using the described software tools and under consideration of the design steps, into a Run-time (RT) C code for the target hardware.

![Diagram](image)

**ADU** = Analog Digital Converter  
**DOUT** = Digital Out  
**FKCN** = Fuzzy Kohonen Clustering Network  
**MLP** = Multi Layer Perceptron  
**SOM** = Self Organizing Map

In order to control the humidification arrangements it is necessary, to assess the water content in the filter. Therefore the process values must be classified. This classification is effected by a self-organizing neural net. The results of this classification are assigned to the following five output classes:


Self-organising artificial neural networks (self-organising maps = SOM) are capable of automatically recognising neighbourhood properties in input data patterns. Hence, SOM are ideally suited for data classification. Provided that there is a suitable network algorithm and a matching weight matrix allocation, a certain coherent neighbourhood area for each input data pattern is activated in the output configuration while the other properties remain inactive. Unsupervised learning in the training phase results in self-organization of weights as a
function of frequency distribution of the provided input patterns and their neighbourhood properties [4]. In order to be usable as a classification each neuron has to be assigned a meaning in a labelling phase. The SOM algorithm in the Model Builder combines the training and labelling phases into one step and, in a further step, can apply the trained SOM to new data.

In the training phase the SOM learns to estimate the “MWC” (Middle Water Content at the complete volume) in these five classes. That is why it is necessary to use extended measuring equipment. This measuring process, which was realized on a pilot filter plant, uses three sensors to measure the water content and one sensor to measure the pressure (Fig. 2). The three sensors to measure the water content are located in different areas and lie at different depths in the substratum.

The aim is to estimate the “MWC” with reduced measuring equipment (Fig. 6), this minimizes the number of sensors and reduces the costs of the measuring equipment.

Classification in groups of pattern

For the two groups of data (MWC and pressure) threshold values must be defined (upper and lower water content and upper and lower pressure). By comparing the measuring values with the threshold values algebraically, it is possible to assign these measuring values to the 9 classes of process data. Fig. 6 shows the diagram of clusters of classes with the normalized measuring values (\{0<\text{x}<1\}). The classes 4, 7 and 8 as well as the classes 2, 3 and 6 are grouped in each class corresponding to the physical correlation and technical properties.

Fig. 6 Classification diagram water content/pressure

These classes may be interpreted as:

- Class 1 : "too dry" ⇒ "moisten the substratum ON"
- Class 4,7,8 : "error A" ⇒ "substratum in a water tailback" or "sensor defective"
- Class 2,3,6 : "error B" ⇒ "substratum locally too dry" or "sensor defective"
- Class 5 : "correct" ⇒ "water content in the filter in an optimal area"
- Class 9 : "too wet" ⇒ "substrate too wet"

This classification supplies the same results when using one sensor for water content and one for the pressure only (substitute (MWC \rightarrow Data of sensor 1). The resulting matrix is the basis for the training process of a neural net and the labelling of the SOM.

The dependence of pressure and the water content is shown in Fig. 7. This proves our approach to reproducing two non-existing sensors with one existing sensor and the measuring value of pressure in the filter plant.

Fig. 7 Classification diagram after substitution
4 Results and Conclusion

Finally, the complete algorithm is translated into a RT-C code regarding the relevant user specifications. The generated C code contains also programme blocks for on-chip periphery control of the target hardware, mainly ADC, digital-port output and timer functions. Moreover, an interrupt system is initiated and used in the algorithm. The C code is optimized for the target hardware and, due to the special characteristics of every target hardware system, can only run on the indicated system after compilation with a suitable C compiler. Presently the Application Builder supports two target hardware systems with µC 80C167.

The capability of the described application is represented exemplary in Fig. 8. The upper diagram in Fig. 8 shows the course of the process sizes of the biofilter over a time period of approximately 10 days. The input values for the SOM are the prepared measurement data of the pressure sensor and a microwave moister sensor (H2O content_1).

Additionally the diagram shows the time course of the other moisture sensors (H2O content_2 and H2O content_3). These delivered together with the H2O content_1 the basis for the computation of the middle waters content, which was input data for training SOM with the target of data classification.

The wished assignment of the measurements of the 5 characteristic classes represents the low diagram in Fig. 8 (split up 1/0).

Measured data showed that microwave moisture measurement in difficult materials such as biofilter-material is possible with the designed applicators and gives a sufficient accuracy in combination with a artificial neural network for process control. The designed penetration applicators can also be used for long term on line measurements.

We have used our software tool EmCoSoft for design und testing a moister measurement and control system for biological filtration plants by using strategies of computational intelligence. All design steps are comprised in a design process with direct interface to the process.
This simulation proves that the algorithm is suitable for supervising and classification of the water content in biological filter plants with reduced number of sensors (minimized measuring equipment) (Fig. 9).

Fig. 9 Generalized block scheme of our project (Model development and implementation in µ-Controller board)

The conviction of the trained SOM to a microcontroller/DSP system with a useful run time C-code is a demand of the plant operators, for a robust and cheap control system in moisturization arrangements for biological filter plants. The RT C code formed as a result of the complete algorithm constitutes an application of its own in the target hardware system with µC or DSP which can be employed for process monitoring or plant control independently of a PC.

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Experience with Detectors for Infrared Moisture Measuring

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ABSTRACT. The wheat with the known quantity of moisture is treated by the pulse energy from near infrared region (NIR). Wavelength of the pulses is 1800 nm and 1940 nm. The reflected signals of the wheat are directed to PbS detector, which is cooled to the temperature ranging from +20°C to -15°C. The change of detector temperature leads to the change of its conductivity. This paper shows the change of detector sensibility depending on various detector temperatures.

Keywords: near infrared, moisture, wheat

1 Introduction

Near-Infrared (NIR) spectroscopy and the chemometric approach of multivariate data analysis have been combined. The combination of these two very different disciplines has led to numerous applications in various fields of science and industry. Near infrared radiation are the electromagnetic waves in the wavelength region longer than the visible light wavelength, lying from 780 nm to 2500 nm. This radiation is invisible to human eyes. It is equal to vibrational or rotational energy of molecules. This phenomenon makes it possible to identify molecules. In NIR spectrum part the stretching vibrations of methyl C-H, methyl C-H, aromatic C-H and O-H bonds. Moreover, the great influence in this region is found in the stretching met oxide vibrations C-H, carbonyl C-H, N-H groups of the primary, secondary and tertiary amides of this N-H group of the ammoniac salt which can’t be considered as dominant. Thus NIR region contains the chemical information CH, OH and NH of the functional groups because they have the strongest absorption. The quantitative measurements of the chemical concentration in a material can be possible in this way. NIR spectrum also gives information about the physical properties of samples and it can be employed for the average magnitude particle estimation or e.g. for the determining of the biomass percent in the fermentation. In NIR part very complex motions occur but only with the mentioned groups like C-H, N-H and O-H. For that reason NIR is inconvenient for the qualitative spectrometry, but it is very acceptable for the quantitative analytics of some individual substance kinds which contain the mentioned groups. The water is also included to these groups. On the other hand, the more complex motion in the water molecules results with the more ambient substance influence, and it makes the water content measuring in some substances to be somewhat more complex. It has been established that the water molecules particularly absorb electromagnetic radiation with the wavelength of 1200, 1450, 1940 and 2950nm, and explicitly with the wavelength of 1450 and 1940nm. Thus those ones for water are called absorbing and on the basis of absorption with these wavelengths the water content can be determined in thin foliages and granular substance structure.

The Near-Infrared spectrum can be divided into two separate regions. These are: the wavelength range from 780-1,200 nm, also referred to as the Herschel region, where silicon detectors are used; and the range between 1,100 and 2,500 nm, where lead sulphide (PbS) is used as the material in the detectors.
2 Moisture Meter and Measurement

2.1 Working Principle

The water is a very good absorber in NIR region and it is very convenient for the quantitative analyses. The three-atomic nonlinearly molecules (H₂O) in any substances can perform very complex motions under the influence of NIR radiation for what they need energy taken from radiation. This absorption has been established with the wavelengths of 1200, 1450, 1940 and 2950nm, and especially it can be seen with 1450 and 1940nm. Those are the absorbing wavelengths of the water. The absorbing spectrometer capable for the work in NIR is the basis for the moisture meter, and the goal is to determine the ratio between the absorbed and all together emitted radiation by the source at the absorbing wavelength, 1940nm for example. Some NIR spectrums of the textile pattern with the different water quantity are measured, figure 1. On the ordinate is marked by A the ratio between the absorbed radiation and the overall emitted radiation by the source on the any given wavelength in the relative units.

![Fig. 1 Some NIR spectrums of the textile pattern with the different water quantity](image)

By considering this ratio, it is necessary to notice the relative moisture in the measuring substance i.e. in the sample. The material sample in which a part of moisture has to be determined can place between the source of IR radiation and detector. The absorption in the sample decreases the radiation that detector receive. The source can be dispersive or non-dispersive. If the radiation from the source is polychrome, it is into the narrow wave bands divided. The two different wavelengths are important for example. One is absorbing (1940nm) and the other reference wavelength (1800nm). The transmission ratio for these different wavelengths is measured.

2.2 Optoelectronic Configuration

The solution of a spectrometer with one ray and two rays are possible. Both are in figure 2 shown. The numbers 1-6 marks as follows: 1 light source, 2 lens, 3 filter wheel, 4a absorption ray, 4b reference ray, 5 sample, 6 detector.
The absorption rays and the reference rays come over the sample in the spectrometer with one ray in detector against the solution with two rays where the reference ray is not contact with the sample. Hence, the solution with two rays is some expensively but it can eliminate some negative influence factors like the sample surface influence, the particle size and the sample colour. Thus by means of the spectrometer with two rays can enlarge measuring accuracy unlike the spectrometer with one ray which can instable work in the certain working terms and conditions even. Furthermore, two of several analysed optoelectronic configurations are interesting to show, Fig. 3. The numbers 1-6 equally on the Fig. 2, 7 mirrors, 8 electromotor, and 9 infrared transmission filter specifies the appropriate elements.

The advantage of the optoelectronic configurations of spectrometers shown on the figure 3 lies in a possibility of the elimination some negative influence factors coupled with the size and ruggedness of the pattern. However, the complexity and the expense for its realization are by those configurations increased.

2.3 Detector and Filter Properties

The appropriate detector choice is very complex. The photoconductive and the photovoltaic detectors in NIR region sensitive are considered, the detectors based on PbS, InSb and InAs for example. The resistance of the photoconductive detectors decreases with the input of IR light. The PbS photoconductive detector can be used for measuring of radiation wavelengths from 1000nm to 3200nm while by the InSb photoconductive or photovoltaic detectors can
detect the IR radiation over the long range of wavelengths even to 6500nm. InAs photovoltaic detectors covers the spectral response range close to PbS but offers higher response speed.

The major characteristics indicating IR detector performance are the photosensitivity, the noise equivalent power (NEP) and $D^*$. The photosensitivity (in V/W) is the output voltage per input radiation power when is noise excluded. NEP is the quantity of input radiation power when the signal to noise ratio is 1. Some noise may come from the infrared detector itself, from its operating circuits or from background fluctuation. However, under the assumption of the noise from an infrared detector and its circuits can be ignored in comparison with the noise caused by background fluctuation define NEP. In the many detectors, NEP is proportional to the square root of the detector active area. $D^*$ in cm·Hz$^{-0.5}$/W or susceptibility is the photosensitivity per unit active area of a detector and makes it easier to compare the characteristics of different detectors.

The small energy of the IR radiation is a special problem in comparison with visible and UV rays, for example 1.24eV at 1000nm and 0.12eV at 10000nm. However, the IR detection efficiency can increase by the detector cooling. The spectral response curves of PbS photoconductive detectors are shift to the longer wavelength side at the same time. The spectral responses one PbS detector is on the Fig. 4.a shown. For example, its highest susceptibility at temperature of 25°C lies at 2200nm and it can increase at -20°C to 2500nm.

![Fig. 4a Spectral response of a PbS detector](image1)

![Fig. 4b Band pass filter for wavelength 1.94 μm](image2)

The time response of PbS photoconductive detectors becomes slower when cooling these but it is not essential for this moisture detector. The output signal from a detector is generally quite small and has to amplify. The preamplifier impedance has to be appropriate in consideration of the detector, low noise and bandwidth. So, the incident light is modulated by the luminous chopper, it is need to use a tuned amplifier in reducing the noise. In addition, it is practical to cool amplifier with the detector together. The mark T on the ordinate in Fig. 4.b represents the omitted rays transmission in %, and the abscissa contains the wave number data the magnitude, which is particularly introduced in IR region. It is equal to reciprocal of one wavelength and it is given in cm$^{-1}$. With of the omitted band along one of the water absorption wavelengths can be seen in that figure.
2.4 Result of Measurement

We used Peltier effect to cool the detector elements. Detector temperature has been changed from +25 ºC to – 20 ºC. When an electric current pass by through a type semiconductor, one end of the semiconductor is cooled and the other end is heated. Photoconductive detectors are operated using a constant current power supply. This PbS detector is quantum type and it must be cooled for accurate measurement. Value resistance from detector compare with reference and the difference controls the current circuit. The current circuit supply PbS detector with constant current. See Fig. 5.

![Block diagram for thermoelectric cooling](image)

Fig. 5 Block diagram for thermoelectric cooling

From the wheat reflected signals have been measured, see Fig. 6. The results analysis shows a significant increase of detector sensibility. The detector sensibility is 1.8 times higher at the temperature of -20 ºC comparing to + 25 ºC. The increased sensibility of detector at low temperatures produces more accurate measured signals.

![Reflections vs. element temperature](image)

Fig. 6 Reflections vs. element temperature
3 Conclusion

The thickness of the sample, which is convenient for a successful moisture measurement, depends on the sample structure and on the wavelength band that is by the source emitted. E.g. for the moisture measurement in NIR spectrum region between 1800nm and 2500nm, the sample thickness has to be about 1mm. In the wavelength measurement region less than 1300nm thanks to less absorption, the sample thickness can be about 5cm. The stronger source of NIR radiation also enables the longer path of the measuring electromagnetic rays so the maximal sample thickness depends on the instrument design. The less transmission paths are for the solid substances moisture measurement needed and it is more convenient to apply the reflection procedure from the surface. Then the moisture is by the calculation of the ratio between the radiations around the two different wavelengths determined. One that is absorbing for water and the other, which is not absorbing. However, the solution of a spectrometer with two rays is also possible, one that is to the sample directed and the reference, which is to the detector directed. The surface of the sample must be characteristic for the observed material, and the system has to calibrate separately for each material. The moisture concentrations from 0.02% to 100% can be in that way measured. In the case of the mirroring reflection, this procedure cannot use. The following effects emerge in the applications for the measuring of the granulated samples: the mirroring reflection, the total absorption and the diffuse reflection/absorption. The contribution of these effects separately depends on the structure and granules size of the sample. With the large granules, the internal reflections occur and it can particularly complicate the measurement.

The determining of the functional dependence of Moisture content % = f (Absorbance)% which is typical for every substance is included into the experimental phase along with the corresponding application of the moisture meter working by using the gravimetric procedure for the calibration. It has to establish before and must be stored in NIR moisture meter memory.

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TAUPE Sealing Monitoring System (SMS) for Landfills

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System Functioning

TAUPE sensor cables that are arranged in the ground of a landfill allow to measure the moisture of the surrounding material. For this purpose, a short, steep-flank impulse is fed into the cable. It runs until the end of the cable and from there, it is reflected towards the start. On its way along the cable, the impulse is influenced by the dielectric properties of the surrounding material. Both the shape and runtime of the impulse are changed. These values may then be related to the moisture of the material, into which the TAUPE sensor cable is embedded.

Moisture Monitoring

If water flows through a leak in a landfill sealing, it propagates in all directions from there. By means of the TAUPE sensor cables, propagation of moisture is measured and the corresponding data are stored. From the moisture distribution measured along the individual sensor cables, a three-dimensional representation of water inflow is obtained.

The chart shows the moisture measured. The bluish color indicates a higher water content.

Setup of the Sealing

Usually, the sealed dump consists of three layers (from bottom to top):
- The waste,
- the sealing layer with TAUPE sensor cables integrated in a monitoring layer, and the surface filter with the above vegetation layer.
**Measurement Principle**

The measurement principle is clearly obvious from the landfill sealing model. In case the sensor cable is arranged in air, as shown in the top figure, the signal is nearly horizontal. If the sensor cable is touched by a hand, the signal collapses, as is shown in the bottom figure. The moisture of the hand changes the signal behavior.

**Setup and Arrangement**

Emissions from landfills into the atmosphere are prevented above all by qualified surface sealing systems. These sealing systems consist of several components that complement each other. If a component fails, its function is taken over by another one. Sealing systems have to be checked regularly for their functioning and efficiency.

Monitoring takes place by sensors that are integrated in a monitoring layer of the sealing system. A system is tight, if no precipitation water enters the waste through the sealing system. Penetrating precipitation water would increase the
water content in the monitoring layer. This increased water content can be detected by means of the TAUPE system. As a result, leaks may be detected and the landfill operator is enabled to repair this leak with a reasonable economic expenditure.

The principal arrangement of the sensors is shown in the figure below. The sensors are installed parallel to the waste slope.

**Test Field in Rastatt-Oberweier**

The sensors are arranged on a test field of about 2 ha. They are installed in a monitoring layer of about 15 cm thickness and distributed homogeneously over the test field.

For this purpose, ditches are dug manually into the slope. This work may be integrated flexibly in the construction process. The sensors are put into these ditches in a flat position and covered by ground material. Immediately afterwards, the first measurement is made.

By arranging the sensors at small distances from each other, measurement of ground moisture of the complete area is ensured.

**Advantages of the TAUPE System**

- Significant reduction of construction costs when using alternative sealings.
- Long-term prognosis for a possible shortening of the post-monitoring phase.
- Development of the sealing (setting, compression) is represented continuously.

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Soil Moisture Group Karlsruhe
Current Work and Future Prospects of a New Research Group

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ABSTRACT. Water content and moisture plays an important role almost everywhere in applied sciences. In many applications, such as renewable energies, resource management, soil science, building materials and natural disaster forecast, water content is a key issue for the characterization and prediction of the system behaviour. These applications will be linked with fundamental research and new measurement technique developed by the Soil Moisture Group (SMG), a team of researchers from different disciplines working together towards a common goal. This contribution introduces to the work of the SMG.

Keywords: Soil Moisture Group, moisture, water content measurement

1 Introduction

Developing moisture measurement methods requires expertise in many scientific disciplines, e.g. material science, hydrology, electrical engineering and so on. Therefore it is advised to form a group of experts in the relevant fields to start a joint effort. A successful example of such a collaboration is the Soil Moisture Group (SMG) which will be presented in this paper. The Soil Moisture Group is a fusion of different institutions open for the scientific community. Up to now the involved institutions are from the University of Karlsruhe, the Research Centre Karlsruhe and the University of Applied Sciences of Mannheim and Karlsruhe. Seven Institutes of three faculties of the University of Karlsruhe are members of the Soil Moisture Group.

The Soil Moisture Group started in 2001 with 5 scientists. At present 25 scientists are doing common research in 32 projects. Nowadays 17 co-operations with industry and research exist. Since 2001 33 papers and 4 PhD-thesis were published.

At the workshop Innovative Moisture Measurements in Research and Practice (http://www.smg.uni-karlsruhe.de) organized by the Soil Moisture Group in 2003 more than 80 participants with 25 presentations attended and 6 exhibitors presented their latest developments in technology for the measurement of water content.

2 Motivation and Goal

A materials moisture is decisive for many physical, chemical, and biological processes, such as hydraulic conductivity of porous media, the ageing of concrete constructions and the composting of biological substances. Until now there is still a need for integration of basic research and practical implementation. To fill this gap the SMG researchers are developing moisture measuring techniques by connecting all allied working units such as application, technology of measurement, and data analysis.

The research group focuses on the common interest of scientific analysis of moisture measurement. It aims to create a nationwide focal point of moisture measurement, in which the knowledge and experience is bundled to establish an enduring center of knowledge in the range of moisture measurement. Furthermore the implementation of new measurement techniques in practice as well as applications are envisaged together with industry.
3 A glimpse at the Current Research Activities of the SMG

Fig. 1 shows that the SMG activities are covering several fields of applications in which natural and multiphase mixtures play an essential role.

3.1 Fundamental Research

The fundamental research covers the influence of moisture on the properties of natural and artificial multi-phase mixtures. The four main topics are:

- Simulation of electromagnetic fields for verification of mixing rules [1], investigation of moisture probes, improvement of calibration procedures [2]
- Determination of the influence of water content on clays e.g. bentonite, organic materials, durability of constructions and materials [3]
- Investigation of the influence of soil moisture on stability of flood water dikes [4], prediction of critical flood water drainage, optimization of hydrological and climate models
- Finally linking the physical properties of partially-saturated media with soil mechanics (slope stability), hydraulics (water movement, sealing), small-scale variability (solute transport) [5]
3.2 Techniques for Moisture Determination

There is no simple and non-destructive measurement technique for water content determination. Therefore the approach is the efficient use of existing technologies by the combination of techniques, methods and experiences for different objectives. This will include:

- Soil physical methods: gravimetric method and tensiometry
- Time domain techniques [6, 7]
- Frequency domain techniques [7, 8, 9]
- Radiometry methods: neutron probe, gamma probe
- Radar techniques: ground penetrating radar, airborne and satellite radar systems

Two examples for different scales of moisture measuring systems developed by the SMG are the TAUPE sensor cable (length up to 100 m) and a robust and cost-effective simple soil moisture probe (Sisomop) which can be embedded in soil or other material of interest (see Fig. 2). The latter is especially suited for sensor arrays to determine water content distribution in heterogeneous media. It consists of a transmission line in the feedback loop of a ring oscillator. For sensor design, construction, calibration and first experimental results see [9].

![Fig. 2: Examples for moisture measuring systems on different scales A) TAUPE sensor cable and B) a new simple and cost-effective soil moisture probe with a size of 185mm x 30mm](image)

3.3 Renewable Energies

Moisture is a key issue for renewable energies e.g. processing of biomass and hydro power generation. For better prognoses in avalanche and flood warning as well as improvements in predicting the filling stages of Nordic and Alpine hydro-power reservoirs, it is necessary to improve the determination of snow pack properties such as liquid water content, density and snow water equivalent [10, 11]. The sensor cables (TAUPE, see Fig. 2) can have lengths of approx. 100 m, thus, in a cross laid pattern, the covered area can be as large as a radar pixel size and can be used for calibrating remote sensing images used for snow water equivalent determination.

Precondition for the sustainable application is the online and continuous moisture monitoring of raw materials involved in these processes. Water content measuring techniques can support the sustainable use of energy and the exploitation of renewable energy sources. The work in the SMG concentrate on the following three topics:
- Monitoring of power generation constructions, monitoring and improvement of energy savings and life expectancy
- Renewable resources improvement of calorific value, monitoring of drying and filtering
- Effective use of energy, optimize the hydro power generation („Snowpower“) [10, 11], monitoring of fossil fuels (calorific value), CO₂-storage

Fig 3: Renewable Energies with A) Test site at the Weissfluhjoch (Davos, Switzerland) for long term measurements of snow moisture profiles (EU-project Snowpower). B) Modern combustor running with wood chip, where water content is a limiting factor for energy balance and emissions.

FREE LINE SENSING of soil moisture and saturation of embankment in a large scale is a new challenge. Free lines over land can transmit electric signals for measuring purposes. The signal propagation depends on the earth surface constitution, mainly humidity content (see TDR).

The reversal process, the measurement of the propagation parameters, provides the surface constitution on distances of several km, which enables continuous large-surface information of socio-economic relevance, e.g. assessment of the safety of embankment.

Fig. 4: FREE LINE SENSING of soil moisture in a large scale
3.4 Management of Resources

High food production rate, the storage and transport of food, as well as the availability and quality are key tasks of the 21st century. All kind of processes in food and medical production and their quality strongly depend on moisture control. Agriculture needs most water for irrigation in dry regions. Therefore the development of high-sophisticated strategies for irrigation is crucial for the effective use of water resources. The strategies based on soil humidity measurement are suitable for the optimized use of energy for drying of raw materials and subsequently humidity may improve storage and transportation capabilities. Recapitulating monitoring of humidity improve the management of resources, food quality and finally the health of people.

Fig. 5: Management of resources with A) typical conventional irrigation for agriculture in dry region. B) Humidity control should help to avoid formation of toxic mould in food and to secure health quality.

3.5 Soil Science

Water and transport of solutions as well as their storage in soils are mainly a function of their water content in unsaturated soils and can be roughly described with a “nano to macro” approach. The adsorption of water on mineral surfaces act on the nano-scale of mineral surfaces. Mainly soil minerals with larger surface areas, such as clay minerals adsorbe several percentage of water and may promote for chemical reaction. The characterization of the state of soil water such as properties and effects of pore water operate on micro-scale. On macro-scale the relationship between matrix potential and water content, hydraulic conductivity, degree of saturation and water flux will be determined.

The physical properties of soil such as water content or hydraulic conductivity are very important in many applications in soil mechanics and other geosciences. Due to a lack of adequate measurement methods the SMG has developed a new soil moisture measurement technology [5]. This measurement system consists of moisture sensitive transmission lines which are buried in the soil. Electromagnetic pulses are applied to the sensor and the reflections are recorded with a time domain reflectometer (TDR).
3.6 Sensor Technology for Buildings

From an economical point of view it is indispensable to achieve a sustainable quality assurance of building materials and to monitor the life cycle of the buildings. Humidity monitoring is a key issue for durability and enhanced performance during the life cycle of building materials and constructions. Mainly infrastructural buildings need monitoring of conditions to prevent early damage and to achieve optimized service life.

In practice buildings such as concrete structures are often exposed to various environmental impacts which strongly determine the service life. Most of all damage mechanisms which lead to the deterioration of building materials are based on moisture transport or the capillary uptake of aggressive chemical compounds dissolved in water. Therefore, monitoring of the water content (QA) or water transport in building materials, for example during the production of concrete, bricks and other building materials, for the characterization of the performance of preventive surface protection measures or climate control in historical buildings are decisive for the performance and durability of structures.

Barriers and sealing, e.g. for toxic waste in waste disposals, underground repositories become more safe with online-monitoring of the technical barriers. The use of simple and cost-effective capillary barrier as an alternative surface covering system of landfill instead of a combination sealing needs a water control system.

Using the TAUPE Sealing monitoring system leaks have to be detected with an accuracy of $<100m^2$ and variation of the water content in the sealing with an accuracy of 10%. Functioning of the TAUPE system has already been tested successfully on a smaller test fields in a landfill and in a lysimeter (see e.g. [6]).

![Figure 6: Water content monitoring in construction (A) and surface covering system of a landfill (B).](image)

A new type of technical barriers of subsurface storage facilities for hazardous waste integrating equipotential segments to improve homogeneous moistening and long-term stability. Hazardous waste materials have to be deposited e.g. in salt formations and encapsulated to protect the biosphere. The isolation itself is a complicated mine shaft closure construction, often made of clayey materials, such as bentonite. In contact with water bentonite tends to swell and if encapsulated in a container, it produces very high pressures that limit further water movement and thus seal the shaft closure.

In order to validate the movement of modeled wetting fronts, measurements are needed, which detect the progress of the front. We build moisture sensing cables that can be integrated in the bentonite layer and are capable of measuring the proceeding wetness. For more information see [http://www.untertageverschluss.de](http://www.untertageverschluss.de)
3.7 Natural Disasters

The early detection of water content can be used for preventive measures, e.g. loss in harvest yield due to heavy rain and drought, damage due to hail and flood events. Mass movement such as landslides, avalanches and mud slides, collapsing dams starts often when the soil is saturated with water. The goal of water control is to get an advanced warning system to prevent collapsing of artificial dams as well as failure of technical barriers.

The flood events at the river Elbe 2002 in Germany and in Czech Republic, but also at the river Danube in Germany and Austria have repeatedly shown the dangers of flooding for the population involved [4]. In spite of all the provisions and optimal flood event forecasting, dikes will remain an essential instrument for flood prevention. However, especially long term flood events pose a particular threat to the security of dikes.

The aim of a current research is the quantitative description of the influence of an initial soil water distribution within a dike body on the progression of the seepage during a flood event. So far TDR technology as a well established moisture measurement technology has been limited to integral or very coarsely resolved water content determination along the transmission line [3]. High spatial and temporal resolution is achieved by applying inverse modelling techniques (so-called reconstruction algorithms) in combination with conventional TDR systems.

![Natural disaster after a flood event](image)

4 Objectives of the Research Group

Summarizing the aspects presented in this contribution the main goals for SMG are:

- To be a center of competence for “water content” of many materials
- To use synergy of multidisciplinarity
- To organize workshops and symposia and to be a part of academia
- To promote young scientists
- To cooperate with other research groups and industrial partners
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Exhibitors

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