Robust low cost open-ended coaxial probe for dielectric spectroscopy in laboratory and in-situ applications

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Abstract

High-frequency electromagnetic (HF-EM) measurement techniques, e.g. dielectric spectroscopy, give inside into soil physical properties which are related to soil/water interactions such as the soil water characteristics. The open-ended coaxial line technique offers a non destructive determination of dielectric spectra of fine grained soil. In this study, a commercially available Agilent high temperature open ended coaxial probe (Agilent 85070E Dielectric Probe Kit) was compared to a robust 3.8 mm open-ended coaxial probe (Sequid probe). The experimental results demonstrate that the Sequid probe is suitable for accurate determination of high resolution dielectric spectra in the Frequency range from 50 MHz to 10 GHz and thus a robust low cost alternative to the Agilent high temperature probe in laboratory and in-situ applications.

Zusammenfassung

Hochfrequente elektromagnetische (HF-EM) Messverfahren, wie die dielektrische Spektroskopie, geben Einblicke in hydraulische Eigenschaften von Böden, z.B. die Saugspannungsbeziehung, die eng verknüpft sind mit der Wechselwirkung zwischen Porenfluid und Feststoff. Die offene Koaxialsonde eröffnet die Möglichkeit einer zerstörungsfreien Erfassung dielektrischer Spektren feinkörniger Böden. Die kommerziell verfügbare Agilent Oberflächensonde (high temperature probe, Agilent 85070E Dielectric Probe Kit) wurde hierzu verglichen mit einer robusten offenen 3.8 mm Koaxialsonde (Sequid Sonde). Die Ergebnisse der experimentellen Untersuchungen zeigen die Möglichkeiten der Sequid Sonde zur Erfassung hochaufgelöster dielektrischer Spektren im Frequenzbereich von 50 MHz bis 10 GHz, die damit eine robuste preisgünstige Alternative zur kommerziellen Agilent Sonde in Labor und Feldanwendungen bietet.

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1. Introduction

High-frequency electromagnetic (HF-EM) measurement techniques, e.g. dielectric spectroscopy, give inside into soil physical properties which are related to soil/water interactions such as the soil water characteristics (see Wagner and Scheuermann 2009). The open-ended coaxial line technique offers a non destructive determination of dielectric spectra of fine grained soil (Schwing et al. 2010).

Open-ended coaxial line probes have originally been developed for broadband determination of dielectric properties of biological tissues (Stuchly and Stuchly 1980, 1982, 1982, Marsland and Evans 1987) as well as for microwave dielectric spectroscopy of liquids (Wei and Sridhar 1989, 1991, Kraszewski et al. 1983, Bao et al. 1994, 1996, Göttmann et al. 1996), furthermore for food quality determination (Kent et. al. 2004, Schimmer et al. 2009, Sheen and Woodhead 1999), agricultural (Nelson 1980, Skierucha et al. 2004, Kelleners et al. 2005), and for geotechnical or soil physical applications (Chen and Or 2006, Wagner et al. 2006, Kupfer et al. 2007, Wagner et al. 2007, 2010, Robinson et al. 2008). Recently, Kaatze and Feldman 2006 and Kaatze 2010 provided extensive reviews of dielectric spectrometric techniques including the open-ended coaxial line technique.

The electromagnetic field at the open-ended coaxial probe aperture fringes from the interface into the sample. Thus, the reflection coefficient measured by means of a vector network analyser (VNA) can be related to the complex permittivity of the sample (Stuchly and Stuchly 1980). Stuchly and Stuchly 1982 derived a lumped element parallel circuit approximation for the probe admittance. Their advantage is the simplicity of the expressions relating the measured reflection coefficient to the sample permittivity, but due to radiation losses the approach is only valid over a limited frequency range for appropriate probe dimensions (Wei and Sridhar 1991). Marsland and Evans (1987) suggested equivalent circuit models including radiation effects and

Otto and Chew (1991) further improved the calibration technique. However, due to the used probe in combination with the applied calibration procedure the overall accuracy in the frequency range around 1 GHz is considerably low. Based on the assumption of wave propagation in transverse electromagnetic mode (TEM) the open-ended coaxial probe is from a theoretical point of view extensively studied (Wei and Sridhar 1991, Göttmann et al. 1996, Blackham and Pollard 1997, Sheen and Woodhead 1999, Ellison and Moreau 2008). Furthermore, there are robust commercial available open-ended probes (Agilent 85070E Dielectric Probe Kit; high temperature, performance and slim form probe) with appropriate analysis software, which has been used extensively (Nelson 1998, 2005, 2009, Kelleners et al. 2005, Chen and Or 2006, Wagner et al. 2007, 2010, Kupfer et al. 2007, Wang and Dong 2008). Nevertheless, the probes are expensive and the software works only in combination with Agilent VNAs.

In this study, the commercially available Agilent high temperature open-ended coaxial probe (Agilent 85070E Dielectric Probe Kit, Wagner et al. 2011) was compared to a robust low-cost 3.8 mm open-ended coaxial probe (Sequid probe), commercially used in a handheld time domain reflectometer (RFQ-Scan 3.0) developed for the in-situ determination of fish quality (see Schimmer et al. 2009).

2. Basic principles

a. The open-ended coaxial line

The complex impedance $Z(j\omega)$ of the sensor/material-combination depends on the dielectric properties of the sample and the geometry of the probe. It is related to the complex reflection coefficient $\Gamma(j\omega)$ by the equation $Z(j\omega) = Z_0 \cdot [1-\Gamma(j\omega)]/[1+\Gamma(j\omega)]$. Herein Z_0 is the characteristic impedance of the open ended coaxial probe, j is the imaginary number and ω the angular frequency, given by $\omega = 2\pi f$. However, some sources of error disturb the actual determination of the true reflection coefficient $\Gamma(j\omega)$ at the aperture plane. The most important are the connector of the coaxial probe and in case of a low-cost sensor the non-ideal behaviour of the coaxial line itself. For these reasons $\Gamma(j\omega)$ is not identical with the actual measured reflection coefficient $S_{11}(j\omega)$, obtained with the VNA, since the latter includes the just described errors as well as errors introduced by the probe head (Stuchly and Stuchly 1980).

To eliminate these systematic errors, a calibration procedure must be performed prior to the determination of the dielectric spectra (Blackham and Pollard 1997). In general, two approaches are applied:

1. A two stage calibration procedure, where in a first step the true reflection coefficient $\Gamma(j\omega)$ is determined by calibration with three known reflection coefficients $S_{11}(j\omega)$ (e.g. Kraszewski et al. 1983, Otto and Chew 1991, Blackham and Pollard 1997, Popovic et al. 2005) and in a second step the

   a. permittivity is calculated by means of the theoretical or numerical formulation of the open-ended coaxial line problem with infinite ground plane and semi-infinite sample size using a quasi-analytical or numerical inversion (e.g. Blackham and Pollard 1997, Sheen and Woodhead 1999, Popovic et al. 2005), or
   b. lumped element parameters are determined (with or without considering radiation effects) in a further calibration step (Otto and Chew 1991).
2. A single-stage calibration procedure, which is based on a bilinear relationship between measured reflection coefficients $S_{11,i}(j\omega)$ and complex permittivities $\varepsilon_{r,i}^{*}(j\omega)$ of appropriate reference materials (Marsland et. al. 1987, Bao et al. 1994, 1996, Kaatze 2007, 2010, Schwing et al. 2010).

In this work a calibration procedure based on 2. is applied to avoid instabilities in the determination of the frequency dependent permittivity due to assumptions in the theoretical formulation of the inverse problem and numerical implementation of the used open-ended coaxial probe. Hence, the measured, temperature depended complex scattering parameter $S_{11}(j\omega,T)$ is related to the complex effective permittivity of the sample $\varepsilon_r^{*}(j\omega,T)$ by

$$\varepsilon_r^{*}(j\omega,T) = \frac{\mathrm{a}_1(j\omega,T)\mathrm{S}_{11}(j\omega,T)-\mathrm{a}_2(j\omega,T)}{\mathrm{a}_3(j\omega,T)-\mathrm{S}_{11}(j\omega,T)}. \quad (1)$$

$a_i(j\omega,T)$ represent three temperature and frequency-dependent complex calibration parameters. These are determined with at least three standard measurements with exactly known complex permittivities $\varepsilon_{r,i}^{*}(j\omega,T)$ in the applied frequency range. It is advantageous, that the chosen permittivities cover the expected range necessary for the appropriate application. The resultant system of equations is formulated in matrix notation $\boldsymbol{M}(j\omega,T)\cdot\boldsymbol{a}(j\omega,T) = \boldsymbol{b}(j\omega,T)$ and solved for $\boldsymbol{a}(j\omega,T) = \boldsymbol{M}^{-1}(j\omega,T)\cdot\boldsymbol{b}(j\omega,T)$ numerically in Matlab. The parameters $a_i(j\omega,T)$ compensate for systematic error and are related to the lumped element equivalent circuit parameters as shown by Marsland and Evans (1987) or Bao et al. (1994).

b. Permittivity measurements

The frequency depended permittivity of standard materials (de-ionized and tap water, ethanol, methanol) are determined under atmospheric conditions with both open-ended probes in combination with a vector network analyzer (Agilent PNA E8363B, used frequency range from 10 MHz to 20 GHz), a high-performance TDR (Tektronix 80E04, frequency range from 10 MHz to 20 GHz) and relatively low-cost TDR (Sequid STDR-65, frequency range from 50 MHz to 8 GHz), respectively. The step-like TDR curves are pre-processed (Nahmann et. al. 1981) and transformed into the frequency domain using the Fast-Fourier Transform (FFT). The complex reflection coefficients $S_{11}(j\omega,T)$ are corrected using open-short-load measurements (Agilent AN1287-3, 2002). The frequency and temperature dependent complex permittivity

$\varepsilon^*_{r,i}(j\omega,T)$ is deduced from the measured complex reflection coefficient $S_{11}(j\omega,T)$ by applying the bilinear relationship (1). Prior to the determination of $\varepsilon^*_{r,i}(j\omega,T)$ the calibration parameters $a_i(j\omega,T)$ are determined using three standard measurements: air and two well known liquids, e.g. de-ionized water and methanol; this is called open–water–liquid (OWL) calibration. The temperatures of the liquids are measured and used to calculate the frequency and temperature dependent permittivity of water (according to Kaatze 2007) and pure methanol and ethanol (according to Gregory and Clarke 2009). Additionally, a short-circuit was measured and used to check the purity of the methanol standard in the frequency range between 100 MHz and 500 MHz. In order to compare the results obtained with a complete commercially available measurement setup, all dielectric spectra have also been measured with the 85070E Dielectric Probe Kit Software. As calibration standards, the media air and water as well as a short-circuit (OWS) were used.

c. Transformation of the time-domain signal

Time-domain reflectometers mostly apply step-signals with fast rise-times. In principle, these signals are periodically repeated square-wave signals which can easily be transformed into the frequency domain by means of a FFT. However, only short segments of the complete TDR square waves contain relevant information and need to be evaluated. These parts of the curves are not periodic anymore and thus unsuited for directly applying the FFT, which implicitly assumes a periodic repetition. However, non periodic signals show an artificial ideal step at the transition from the last value to the first value. This transition causes spectral components, which are purely artificial in nature and would lead to falsified results. For circumventing these artifacts, the method from Gans et al. (1982) is used to convert the reduced signal to one being applicable to the FFT algorithm.

3. Measurement

a. Frequency domain measurements

The principle experimental setup is represented in Figure 1. In a first step the coaxial line with an PSC-2.4 to SMA adapter is fixed in a probe stand and calibrated at the SMA connector with an one port calibration with mechanical standards (open, short, load). Then the open-ended coaxial probe is carefully fixed to the SMA connector of the used adapter. In total four measurements were carried out: open, short, methanol and de-ionized water. Each calibration standard is measured using 20 averages. The temperature of the liquids was measured with an accuracy of ±0.5 K with an Ahlborn NiCr-Ni thermocouple. The measured reflection coefficients $S_{11}(j\omega)$ obtained with the Sequid probe are depicted in Figure Fig. 2. It is obvious that for materials without ionic conductivity the magnitudes as well as the phases of $S_{11}(j\omega)$ become more congruent with decreasing frequency, especially in the range below 100 MHz.

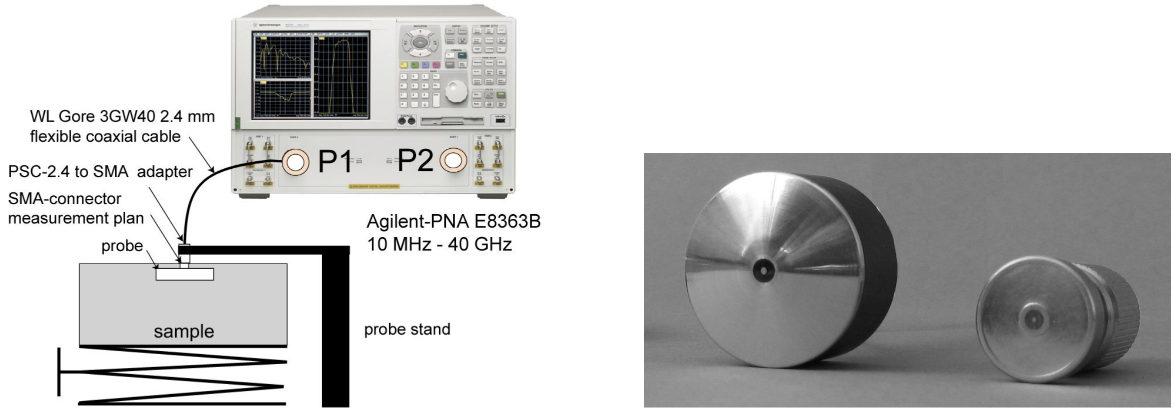

Fig. 1: (left) Measurement setup with an open-ended coaxial probe to determine dielectric spectra and (right) investigated probes (left: Sequid, right: Agilent).

Thus, the sensitivity of open-ended coaxial line sensors to determine complex permittivities below 100 MHz is low (see Figure Fig. 2) and the accuracy of the obtained permittivity decreases in dependence on the dynamic range of the instrument, the applied averaging factor, as well as the carefulness of the calibration and measurements. Moreover, as a result of this work, a lower limit of the measurement range of approximately 50 MHz with the used procedure and materials is suggested.

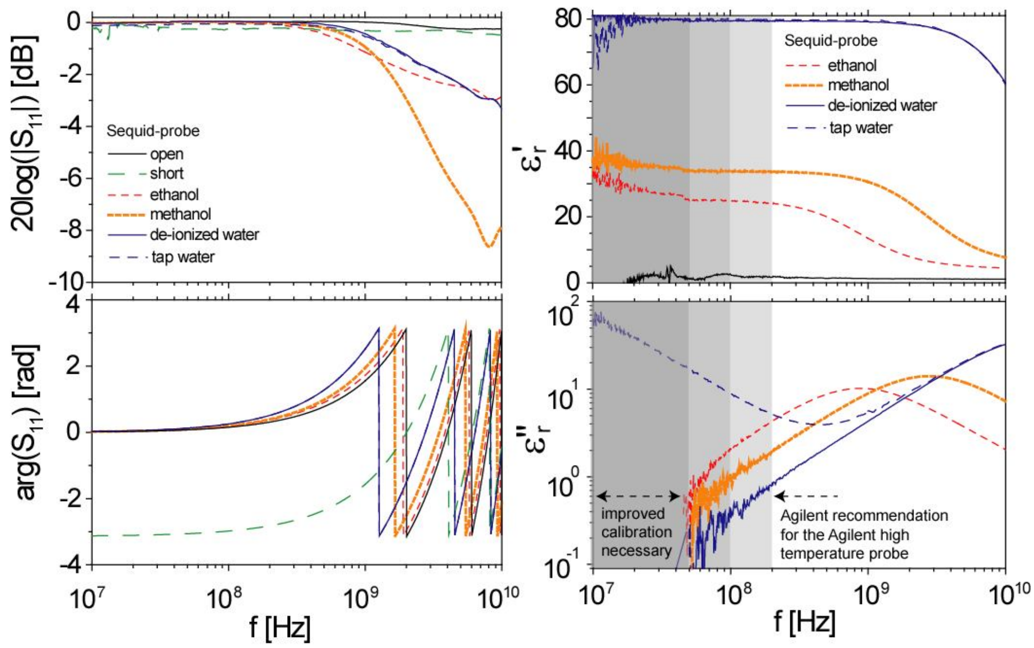

Fig. 2: (left) Magnitude and phase shift of the measured reflection coefficient $S_{11}(j\omega,T)$ obtained with the Sequid probe for the investigated liquids as well as air and short circuit measurement. (right) Complex relative permittivity $\varepsilon_r^* = \varepsilon_r' - j\varepsilon_r''$ obtained from measurements of de-ionized water, tap water, methanol and ethanol according to equation (1) after a separate calibration by means of the introduced calibration procedure.

In principle, to obtain stable permittivity results down to at least 1 MHz very precise machined probes with well-defined geometric dimensions as well as very carefully performed measurements are necessary (see Stuchly and Stuchly 1982, Sheen and Woodhead 1999). The results of the investigation of Sheen and Woodhead 1999 also point out the loss of accuracy below approximately 100 MHz. In the technical overview of the used Agilent high temperature probe 200 MHz are suggested as absolute lower limit of the measurement range (Agilent Technologies 2008). Nevertheless, the open-ended coaxial probe exhibits a high sensitivity to electrical losses. Therefore, the determination of low frequency properties, e.g. electrical conductivity or low frequency relaxation effects, is possible with an appropriate improved calibration technique (see Otto and Chew 1991).

b. Time domain measurements

As already discussed, the reflection coefficient $S_{11}(j\omega,T)$ has also been determined from TDR measurements made with the Tektronix 80E04 module and the Sequid STDR-65. The TDR signals of both devices are processed applying the same procedure as described in section 2.c. The Tektronix 80E04 utilizes a time-base resolution of 2.5 ps and a sampling interval of 10 ns. The resolution of the STDR-65 is 10 ps and the sampling interval is 50 ns. In both cases, 100 curves are acquired and averaged. The averaged TDR measurements made by means of the Sequid TDR are depicted in Figure

Fig. 3.

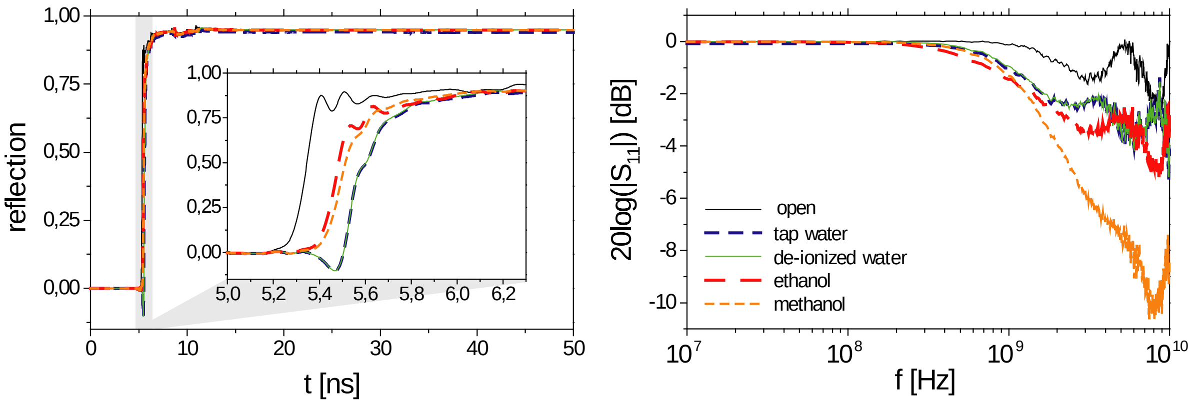

Fig. 3: (left) TDR measurements performed with Sequid's STDR-65 in combination with the Sequid-probe for the investigated liquids as well as air. (right) Magnitude of the calculated reflection coefficients in frequency domain.

In soil science applications it can be very advantageous to use a portable ruggedized TDR to determine the dielectric properties of soils directly in the field. The STDR-65, shown in Fig. 2, fulfils these requirements and is thus suitable for extensive practical in-situ measurements. The acquired curves are pre-processed in the TDR and subsequently transmitted to a laptop computer, which carries out the Fourier Transform, the calibration and the permittivity calculation.

Fig. 4: Sequid portable STDR-65 (dimensions: 54×168×208mm^3, weight: 1545g)

4. Discussion

The dielectric spectra (50 MHz to 20 GHz) of the investigated liquids obtained from the VNA measurements in combination with the OWL-calibration of both the Agilent and the Sequid probe are in close agreement with the expected theoretical spectra (see Fig. 5). In particular, it can be observed that especially in the frequency range below 1 GHz the signal-to-noise ratio of the Sequid probe is superior to the Agilent probe. Moreover, the results obtained with the Agilent Software in combination with the OWS calibration tend to show instabilities in the frequency range above 10 GHz. These instabilities were further confirmed in comparative measurements with the Agilent performance probe (500 MHz - 40 GHz).

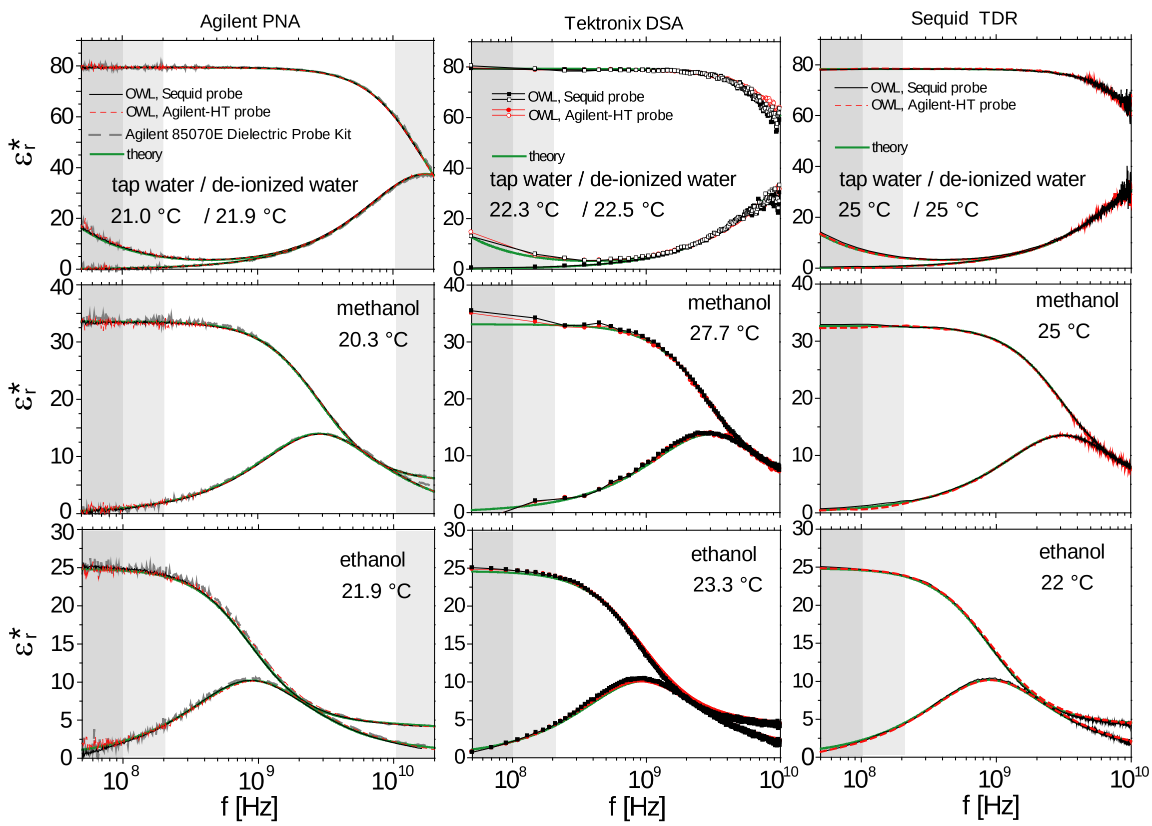

Fig. 5: Obtained dielectric spectra with the introduced procedure (OWL) in case of the different devises and the two investigated probes (Sequid probe, Agilent high temperature probe - HT) for the investigated standard liquids in comparison to the expected theoretical spectra. In case of the results determined with the Agilent PNA the appropriate spectra obtained with Agilent Material Measurement Software for the Agilent high temperature probe (Agilent 85070E Dielectric Probe Kit) are indicated.

The results obtained from both TDR-measurements (100 MHz to 10 GHz) in combination with the OWL-calibration are also in a good agreement with the theoretical results, especially in the frequency range below 5 GHz. As expected, the signal-to-noise ratios of the TDR results between 5 GHz and 10 GHz decrease with the frequency, which can be attributed to the decreasing power of the spectral components with frequency. However, this effect of noise caused by the system inherent jitter and amplitude noise can be improved by increasing the sampling rate of the used TDR and by the applied digital signal processing.

5. Conclusions and Outlook

The experimental results demonstrate that the Sequid probe is suitable for accurate determination of high resolution dielectric spectra with the used OWL calibration procedure. Hence, it offers a robust low-cost alternative to the Agilent high temperature probe for laboratory and especially for in-situ applications. The rounded shape of the aperture plane of the Sequid probe is well suited for measuring liquids, facilitating calibration measurements in the field. An integrated Pt100 sensor acquires the temperature of the calibration liquid, which is automatically considered in the calibration procedure. In further steps, the accuracy in the determination of dielectric spectra of lossy materials (e.g. soil) and the calibration procedures in the low frequency range will systematically be compared with broadband coaxial line measurement cells. In this context, appropriate standard materials for an efficient calibration of the probes have to be developed.

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