Broadband dielectric spectroscopy open ended probe for the characterization of dispersive materials

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ABSTRACT

We propose a large open ended wideband probe for the characterization of the hydromechanic properties of heterogeneous materials. The probe, which is coaxial and features a cone shape, is due to be put in contact with the material to characterize and to enable a reflectometry measurement over a microwave band. The measured signal is a function of the dielectric permittivity of the material which depends on its hydromechanic properties (water saturation, density…). In this paper, we describe the proposed probe concept and provide simulations based on finite elements modeling which demonstrate that such probes are sensitive to materials such as dispersive clays featuring different water saturations.

Keywords: Dielectric spectroscopy, structural health monitoring, bentonite

1 INTRODUCTION

The study of soils, the health monitoring of civil engineering works and that of nuclear waste repository systems require the monitoring of the hydro-mechanical properties of the materials they are made up of, such as concrete and clay. Such monitoring implies the development of dedicated instrumentation methods. We propose a broadband approach which lies in the field of dielectric spectroscopy, which represents a suitable alternative [1] to time domain reflectometry (TDR) methods [2] as far as dispersive materials are concerned. Indeed, materials such as clay may feature significant dielectric permittivity variations as a function of the frequency [3, 4], over ranges that can spread from 1 MHz to several GHz. Such dispersion is imputable to different relaxation phenomena depending on the hydro-mechanical characteristics of the materials (water content, density) [5, 6]. Moreover, it has been demonstrated that the localized sensing area and the air gap surrounding the probes such as those implemented in TDR methods could affect the accuracy of measurements [7]. The characterization method we report on relies on the use of a large open-ended probe. It can be suitable for laboratory studies and also to structural health monitoring thanks to the reflectometry principle it relies on.

This paper is organized as follows: section 2 highlights the instrumentation issues regarding the structural health monitoring (SHM) of dispersive structures such as the bentonite closures due to be built in nuclear waste repositories. Section 3 presents the concept of a broadband reflectometry probe which could be used in a SHM context with the hydro-mechanical characterization of dispersive materials in view. Section 4 presents simulations of such a probe based on finite elements modeling. Finally, conclusions are provided in section 5.

2 STAKES OF THE STRUCTURAL HEALTH MONITORING OF BENTONITE CLOSURES

For safety reasons, the nuclear industry draws important structural health monitoring stakes, some of which lie in the field of the characterization of the saturation degree of materials like concrete or clay. Among the concerned applications are those related to radioactive waste repositories. Indeed, using underground callovo-oxfordian clay-rock layers is envisaged for radioactive long-lived high and intermediate level wastes disposal, since these geological layers feature a very low permeability to radionuclide. Several experiments of such industrial repositories are conducted around the world, the Cigéo project being an emblematic example set in France [8-10]. Within the framework of such geological repositories, a monitoring system must fulfil the knowledge required to run the disposal and its reversible management (Cigéo must be reversible for at least a century-long period), structured according to steps of progressive construction, emplacement and closure of disposal cells. Monitoring should provide with the information required to confirm the knowledge implied by the evaluation of the long term safety and improve the models, on the basis of data obtained in situ, within the framework of periodical re-evaluations of the structures. Current understanding and predictions are based on experimental results obtained in surface and underground laboratories, as well as on data from modelling and simulations. This knowledge is due to be verified, confirmed and enhanced based on data obtained in situ, that is in a repository at scale 1, and/or in the Pilot phase of Cigéo.
Various closure elements are planned for repository backfilling and sealing. More precisely, specific closure backfills and seals are foreseen at the head of each repository cell, and for the entire infrastructure (drifts, shafts and ramps). Fig. 1 illustrates a cell seal, including supporting concrete structures. Most of the seals are made up of clay material (swelling clay or clay stones), excepting concrete plugs providing the seals with mechanical support.

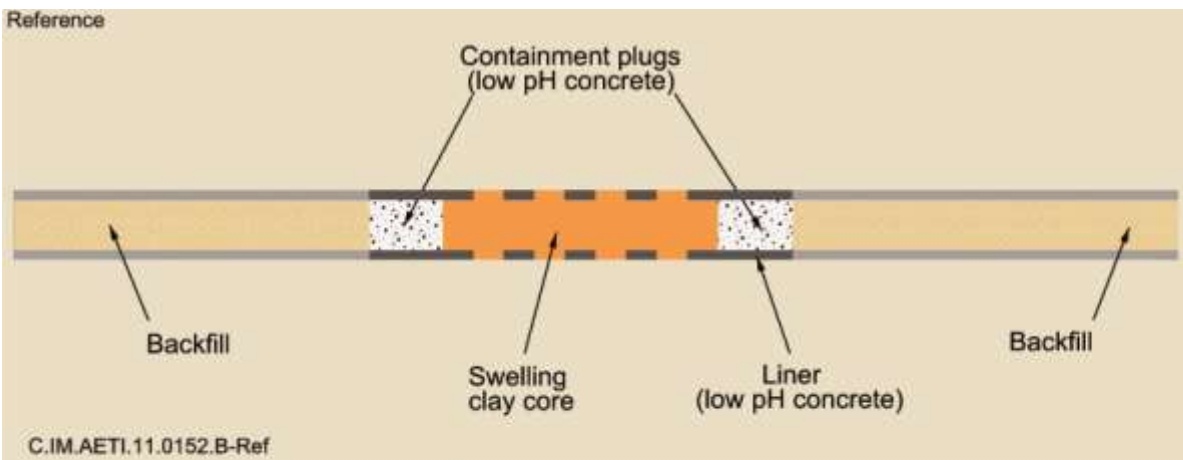

Fig. 1: *Scheme of a seal of a repository cell for radioactive wastes © Andra (French National Agency for radioactive waste management, in charge of the Cigéo project).*

The bentonite is a clay material of utmost importance in the repository seals. The primary function of a bentonite barrier is to ensure that the transfer of various substances through it is dominated by diffusion. At emplacement, bentonite seals may consist of a mixture of pellets, powder and engineering voids, as shown in Fig. 2a. Water uptake after deposition will lead to swelling. This will cause all the gaps to disappear and the material to be homogenized (Fig. 2b). However, some inhomogeneity will remain due to friction in the bentonite. This residual inhomogeneity is of importance for the design premises and the configuration (pellets, powder and voids) in which the seal is deposited.

The overall objective of the study reported in the present paper is the development of a monitoring technique suitable to characterize the mechanical evolution of an inhomogeneous bentonite barrier while the water content is increasing and therefore to evaluate the sealing performance with time. Whether regarding the processes in the early evolution of the repository system, or with a view to evaluate the impact of the processes on the long-term safety functions.

(a) ***(b)***

Fig. 2: *(a)Bentonite mixture made up of pellets and powder, representative of the material at emplacement of nuclear waste repository seals ; (b) bentonite during the hydration phase.*

Dielectric characterization methods usually are good candidates to moisture measurements. Indeed, as show in Table 1, the relative dielectric permittivity ε_r of a medium depends on its composition and the water content may be of great influence.

Table 1: *Relative dielectric permittivity of different materials.*

Material	Real part of the relative dielectric permittivity ε_r'
Water	80
Solid particles	$5 < \varepsilon_r' < 15$
Gas	1

TDR (time domain reflectometry) probes are commonly applied to moisture measurements of soils with agriculture watering optimization in view. However, as far as clay materials (such as bentonite) are concerned, the choice of the characterization technique requires several particularities to be taken into consideration: (i) the ranges of variation of signals are smaller, (ii) empirical calibration equations suitable for soils such as Topp law [11] fail, (iii) clay is a dispersive material (the dielectric permeability depends on the frequency). These are among the reasons for which the use of frequency domain reflectometry techniques shall be envisaged [12] — not to mention that the insertion of TDR probes into clay often creates an air gap which can compromise the robustness of the measurements.
In order to both cope with the dispersive and heterogeneous features of bentonite materials, we propose to develop a spectroscopy method implementing a broadband reflectometry probe featuring a large sensing area (see below).

3 BROADBAND REFLECTOMETRY PROBE CONCEPT

The choice of a large open-ended [13-16] topology which we consider aims at combining two main features: on the one hand broadband operation which is suitable for characterizing dispersive material and on the other hand a large probe aperture so as to radiate an electromagnetic wave towards a representative elementary volume (REV) of material that may be in the order of several cm (cubed), depending on the heterogeneity of the material. Now, the sounded volume increases with the probe aperture [15].
The proposed probe (Fig. 3) consists in a cone-shaped coaxial waveguide destined to be placed in contact or embedded within the material to be characterized. It is due to be connected to a vector network analyzer via a coaxial line, used to measure the reflexion coefficient Γ in the input plane of the probe. Γ is a function of both the scattering parameters S_{ij} ($i,j \in \{1,2\}$) of the latter and the reflection coefficient ρ in the output plane of the probe, which depends on the dielectric characteristics of the material under test:

$$\Gamma = S_{11} + \frac{S_{21}S_{12}\rho}{1-S_{22}\rho} \quad (1)$$

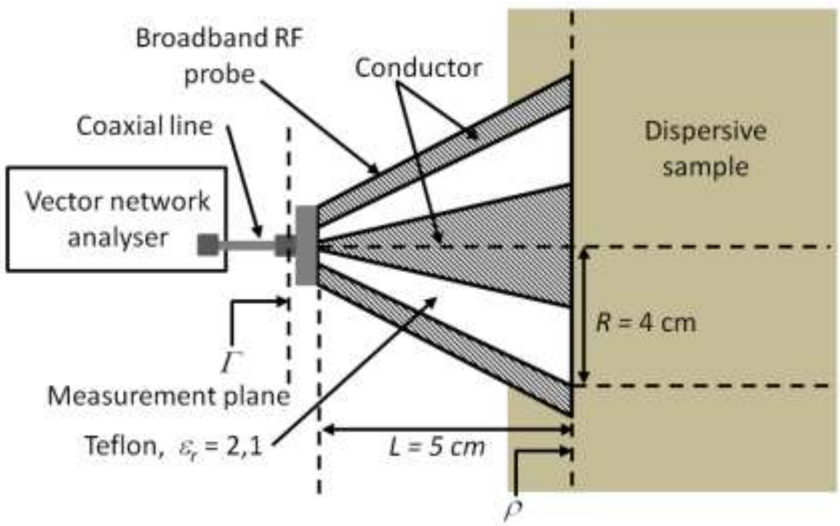

Fig. 3: *Large open-ended cone-shaped coaxial probe (cross-sectional view) for the characterization of dispersive dielectric materials.*

It is therefore interesting to sense Γ and subsequently determine ρ, with a view to access the hydro-mechanical characteristics of the materials which the dielectric permittivity is dependent on. To do so, one shall first determine the scattering parameters of the probe by means of a calibration on reference sample materials featuring known dielectric permittivity and ρ [17].

4 BROADBAND REFLECTOMETRY PROBE SIMULATIONS

A study based on finite elements (FE) simulation (using HFSS software) of open ended probes such as that described above and depicted in Fig. 3 was performed. All simulations reported here have been performed considering probes featuring a length L = 5 cm and an output aperture R = 4 cm (Fig. 3). The dielectric of the probes was Teflon (of relative dielectric permittivity ε_r = 2.1) and they were designed so as to feature a Z_c = 50 Ω characteristic impedance their whole length along. Note that this probe features a cut-off frequency in the order of 1.3 GHz (i.e. above that limit the waveguide is no longer single mode). Nevertheless the simulations were done over the [30 MHz 3 GHz] band. The dispersive materials under test which were used were clay rocks for which we did dispose of dielectric permittivity characteristics as a function of the frequency for different water saturations (*Sw*) [18].

4.1 Study of the sounded volume

Among the points examined was the ability of the probes to sound a volume of material sufficient for envisaging the characterization of heterogeneous materials like those mentioned above. Such an ability can be qualitatively observed on the example depicted in Fig. 4, which shows the electric field distribution in a clay rock featuring a *Sw* of 45%.

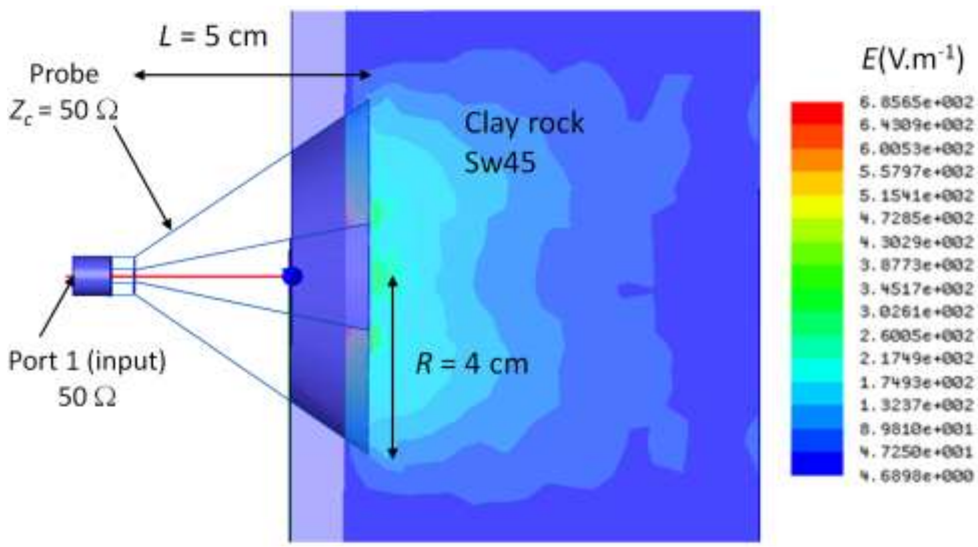

Fig. 4: *FE simulation of the cartography of the electric field radiated by an open ended probe featuring L = 5 cm, R = 4 cm and* Z_c *= 50 Ω within a clay rock featuring a Sw of 45 %.*

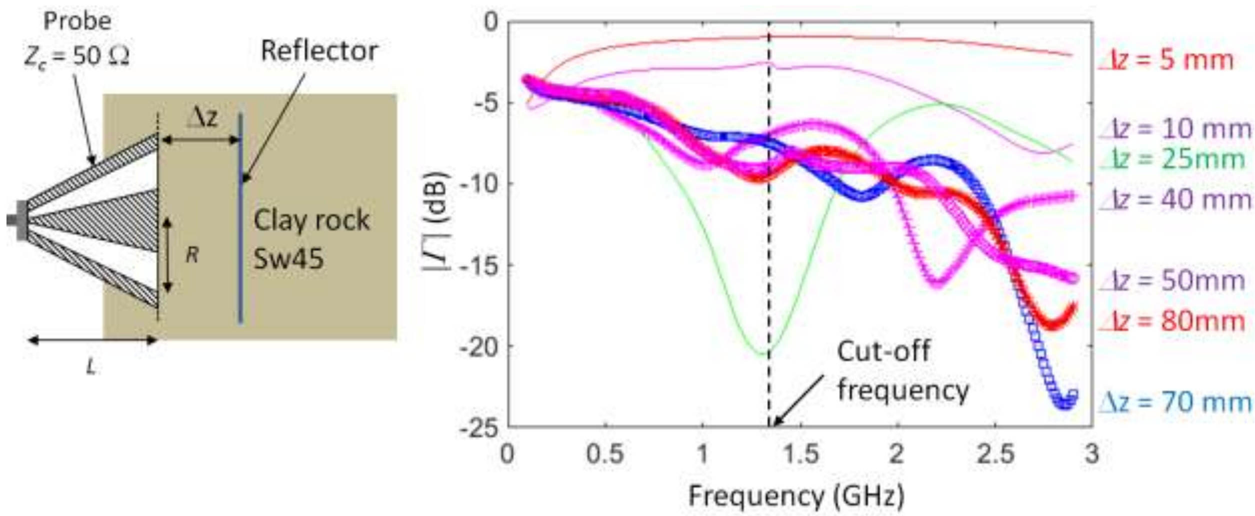

Fig. 5: *FE simulation of the reflection coefficient* $|\Gamma|$ *in the input plane of an open-ended probe featuring L = 5 cm, R = 4 cm and* Z_c *= 50 Ω when a reflector is placed inside the material under test (clay rock with Sw = 45 %) at a distance Δz. The cut-off frequency stands for the frequency from which the probe is no longer single-mode.*

Simulations of the reflection coefficient Γ at the input of the same probe placed inside in the same clay-rock and where a metallic reflector was placed at a distance Δz of the end of the probe were performed for different values of Δz (Fig. 5). These simulations aimed at quantitatively approximate the sounded volume. As the influence of the presence of the reflector on Γ can be observed up to $\Delta z = 80$ mm, the probe/material interaction depth can be considered as being of several centimeters on this example. This result is promising in the perspective characterizing heterogeneous materials featuring REV in the order of several cm (cubed). It is to be noted that the position of the reflector influences the frequencies at which Γ features minima and maxima. This phenomenon is attributable to destructive and constructive interference.

4.2 Sensitivity of the probe to dispersive clay rocks featuring different water saturations

Another point requiring examination is the ability of the probes to discriminate dispersive material featuring different water saturations. The simulations reported in Fig. 6 where carried out to that aim. The aqueous NaCl solution, the methanol and the short circuit represent possible calibration loads [19], while clay rocks featuring a *Sw* ranging from 16 % to 99 % represent possible dispersive materials to characterize. According to the simulation results, the probe input reflection coefficient actually depends on water saturation.

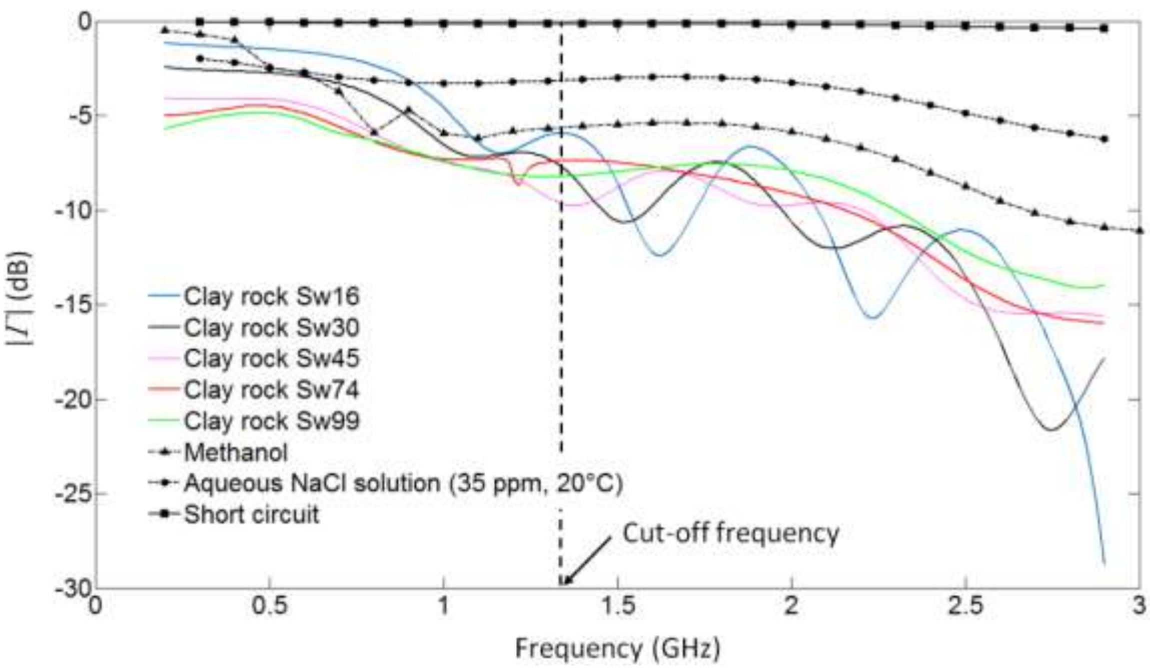

Fig. 6: *FE Simulation of the reflection coefficient* $|\Gamma|$ *in the input plane of an open-ended probe featuring L = 5 cm, R = 4 cm and* $Z_c = 50\ \Omega$ *for different dispersive materials under test (or loads).*

5 CONLUSION

The use of broadband dielectric spectroscopy implementing a large open ended probe has been proposed with a view to characterize the hydro-mechanic properties (water saturation, density…) of heterogeneous and dispersive clay materials and SHM applications. The probe, which is coaxial an cone shaped, is destined to be put in contact with the material to characterize so as to carry out a reflectometry measurement in the microwaves domain. The measured signal is in principle a function of the dielectric permittivity of the material under test, which depends on its hydro-mechanic properties. Simulations

(based on FE modeling) of probes placed in the presence of different materials, among which clay-rocks, have given promising results regarding the discrimination of such materials featuring different water saturations. Simulations performed in order to study the sounded volume of clay materials also have given promising results as regards of the characterization of heterogeneous materials featuring a representative elementary volume in the order of several cm (cubed) like bentonite materials used in nuclear waste repository seals.
Therefore, the design of a probe prototype with a view to moisture measurement of heterogeneous clay materials and SHM applications can be envisaged.

REFERENCES

[1] Kremer, F., "Broadband dielectric spectroscopy", Berlin: Springer, 2003.

[2] Königer, F., Emmerich, K., Kemper, G., Gruner, M., Gaβner, W., Nüesch, R., & Schuhmann, R., "Moisture spreading in a multi-layer hydraulic sealing system (HTV-1)". Engineering Geology, 98(1), 41-49, 2008.

[3] Asami, K., "Characterization of heterogeneous systems by dielectric spectroscopy". Progress in Polymer Science, 27(8), 1617-1659, 2002.

[4] Lesmes, D. P., & Morgan, F. D., "Dielectric spectroscopy of sedimentary rocks". Journal of Geophysical Research: Solid Earth (1978–2012), 106(B7), 13329-13346, 2001.

[5] Jonscher, A. K., "Dielectric relaxation in solids". Journal of Physics D: Applied Physics, 32(14), 1999.

[6] Wagner, N., T. Bore, J.-C. Robinet, D. Coelho, F. Taillade, and S. Delepine-Lesoille "Dielectric relaxation behavior of Callovo-Oxfordian clay rock: A hydraulic-mechanical-electromagnetic coupling approach". J. Geophys. Res. Solid Earth, Volume 118; Issue 9, Pages 4729 – 4774, 2013.

[7] Bore, T., Wagner, N., Delepine-Lesoille, S., Taillade, F., Six, G., Daout, F., & Placko, D., "3D-FEM modeling of F/TDR sensors for clay-rock water content measurement in combination with broadband dielectric spectroscopy". In Sensors Applications Symposium (SAS), 2015 IEEE, 1-6, April 2015.

[8] European project MODERN (Monitoring Developments for Safe repository Operation and staged closure" n°262598 (FP7 Euratom) WP4-Case Study pf monitoring at all stages of the disposal process".

[9] http://www.Cigéo.com/

[10] Buschaert, S., Lesoille-Delepine, S., Bertrand, J., and Landais, P., "Andra's geologic repository monitoring strategy". Proc. of the 5th Clays in Natural and Engineered Barriers for Radiocative Waste Confinement, Montpellier, 2012.

[11] Topp, G. C., Davis J. L. and Annan A.P., "Electromagnetic determination of soil water content: Measurement in coaxial transmission lines". Water Resources Research, 16(3), 574-582, 1980.

[12] Kupfer K., Trinks E., Wagner N. and Hubner C., "TDR measurements and simulations in high lossy bentonite materials". Meas. Sci. Technol. 18 1118, 2007.

[13] Marsland T. and Evans S., "Dielectric measurements with an open-ended coaxial probe". IEEE Proc. Microw., Antennas Propag., vol. 134, no. 4, pp. 341–349, Aug. 1987.

[14] Grant, J. P., Clarke, R. N., Symm, G. T., & Spyrou, N. M., "A critical study of the open-ended coaxial line sensor technique for RF and microwave complex permittivity measurements". Journal of Physics E: Scientific Instruments, 22(9), 757, 1989.

[15] Meaney, P. M., Gregory, A. P., Epstein, N. R., & Paulsen, K. D., "Microwave open-ended coaxial dielectric probe: interpretation of the sensing volume re-visited". BMC medical physics, 14(1), 3, 2014.

[16] Wagner, N., Schwing, M., & Scheuermann, A., "Numerical 3-D FEM and experimental analysis of the open-ended coaxial line technique for microwave dielectric spectroscopy on soil". Geoscience and Remote Sensing, IEEE Transactions on, 52(2), 880-893, 2014.

[17] Otto, G. P., & Chew, W. C., "Improved calibration of a large open-ended coaxial probe for dielectric measurements. Instrumentation and Measurement". IEEE Transactions on, 40(4), 742-746, 1991.

[18] Wagner N., Bore T., Robinet J.-C., Coehlo D., Taillade F., Delepine-Lesoille S. "Dielectric relaxation behaviour of Callovo-Oxfordian clay-rock" Journal of Geophysical Research: Solid Earth, vol. 118, 1–16, 2013.

[19] Kaatze, U., "Reference liquids for the calibration of dielectric sensors and measurement instruments". Meas. Sci. Technol., 18(4), 967–976, 2007.