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Combination of Two Measurement Techniques to Expand the Measurements Frequency Range of the Dielectric Permittivity

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Abstract—Usually, knowledge of dielectric properties of material, as a function of frequency, represents a key issue in scientific fields and several industrial applications. At LNE-CETIAT, in partnership with Institut Fresnel UMR 7279[2], a set of capacitive and coaxial cells, dedicated to the measurement of complex dielectric permittivity, have been developed. The present paper focuses on the experimental calibration and validation of two cells using low and medium dielectric loss materials. It gives the main measurement results obtained on three different materials: decanol alcohol, polytetrafluoroethylene (PTFE) and polyvinyl chloride (PVC) in the frequency range from 3 MHz up to 2 GHz.

1. INTRODUCTION

Nowadays, measurement of the complex dielectric properties (permittivity or permeability) of materials at radio-frequencies and in the microwave spectrum is a great challenge for materials' study, for example, in thermal and drying processes in industry, material engineering or medical applications like tumor ablation.

Although there are several methods to measure these characteristics, each method has limitations in terms of product type and especially in the frequency spectrum. However, in order to extract some information on relaxation processes, it is paramount to measure the dielectric permittivity in a wide frequency range. For instance, the most commonly used techniques are coaxial line measurement and capacitive techniques.

Theoretically, the coaxial method is available from DC to a maximal frequency determined by the coaxial dimensions and material permittivity. Nevertheless, in practice, the minimum measurement frequency is limited to some tenth of MHz due to the calculation method as studied in [1] and explained in [2]. On the other hand, the resonance frequency of the capacitive system reduces the measurement band to a frequency below 100 MHz.

In this letter, we will focus on low and medium loss solid and liquid materials and the calibration/validation of the two cells: capacitive and coaxial. In the literature, PTFE is generally used as a low loss reference material to validate permittivity measurement [3, 4].

The results obtained with two cells, for liquid material, decanol, and for two solid materials, polytetrafluoroethylene (PTFE) and polyvinyl chloride (PVC) are presented.

The measurements are done between 3 MHz and 2 GHz, using a capacitive technique in [3 MHz–100 MHz] and using coaxial line in [100 MHz–2 GHz]. The measurement results are then compared with *EpsiMu*® tool [5], which stands for a reference instrument fully validated.

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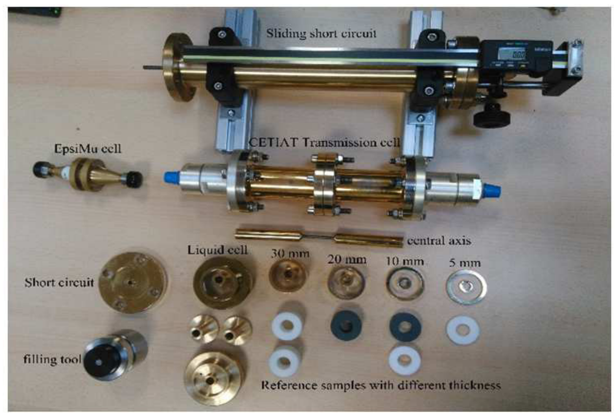
2. MATERIALS AND METHODS

2.1. Capacitive Technique

Typically, at low frequencies, below 100 MHz, the parallel plate capacitor methods are used to measure dielectric permittivity. The main reason for this choice is the simplicity of this technique and the acceptable measurement accuracy with many types of materials in the low radio-frequencies band. (Figure 1(a)) shows the capacitive cells developed (for liquids and solids) that can be used from 3 MHz up to 100 MHz. The sample of the material under test is placed between two parallel electrodes, and the complex dielectric permittivity $\varepsilon_r^* = \varepsilon' - j\varepsilon''$ can be derived from the comparison between measured impedance with and without material [6].

(a)



(b)

Figure 1. (a) Capacitive cell, (b) coaxial cells with sample holders for solids and liquids.

The measurement requires a sample with a diameter smaller than the electrodes disc in order to limit the edge effect. The impedance values are obtained with a Vector Network Analyzer (VNA) using a reflection configuration (S_{11}/1 Port measurement). However, as explained in [7], this configuration introduces parasitic parameters: inductances and capacitances. These parameters are precisely determined using well-known reference liquids (alcohols) in a liquid cell which has the same dimensions of the solid sample holder. The final expressions used in the calculation are as follows:

$$\varepsilon' = -\frac{d}{\varepsilon_0 A}\left(\frac{D}{(C^2+D^2)\omega} + B\right) \quad (1)$$

$$\varepsilon'' = \frac{Cd}{(C^2+D^2)\omega\varepsilon_0 A} \quad (2)$$

where d and A are the sample thickness and surface. B, C and D are three parameters calculated from the measurement impedance as explained in [7].

2.2. Coaxial Technique

Using coaxial line, two non-resonant techniques can be used: transmission/reflection technique [3] and reflection technique [8]. These different techniques can be grouped into four possible configurations [9, 10]: Short, Open, Load and Transmission. Each of these configurations gives an equation [10] which allows to determine the complex permittivity. To benefit from the advantages that offer each method, an air coaxial line cell (1" 5/8) (Figure 1(b)) and a sliding short circuit are built. The sliding short circuit allows the realization of two configurations among the four: in "Short" configuration by placing the short circuit directly after the sample and in the "Open" by changing the position of short circuit at a $\lambda/4$ distance, and therefore for each position, the value of the permittivity at the correspond frequency can be calculated [8].

In this paper, we choose to use the "Transmission" configuration because it is a broadband method. To calculate complex dielectric permittivity, we use the iterative one-parameter inversion algorithm described in [3]. This algorithm requires initial guess values. To calculate the initial values for all frequencies, we choose to apply the faster measurement process applied by *EpsiMu*® [5]. This tool † based on Nicolson and Ross [11] and Weir [12] algorithm (NRW algorithm). To apply the NRW algorithm, the reference calibration plane of the VNA has to be placed in front of the two faces of the sample also called de-embedding process [13]. Then, using its S parameters, we can calculate the value of the complex dielectric permittivity. Due to the nonmagnetic property of the measurement materials ($\mu_r = 1$), we can use the one-parameter NIST iterative algorithm [3]. As we analyze lossless materials, the uncertainty in the measured phase of S_{11} is very wide, and on the other hand, the transmission parameter S_{21} is strong [3]. For this reason, we have chosen to use the S_{21} parameter to calculate the complex permittivity:

$$\frac{S_{21}}{S_{21}^{\text{empty}}} = \frac{T(1-\Gamma^2)}{1-\Gamma^2 T^2} \tag{3}$$

where the measured parameters are:

- S_{21} is the measured transmission S-parameter of the cell loaded with the material,
- S_{21}^{empty} is the measured transmission S-parameter of the empty cell.

And the calculated parameters are:

- The reflection coefficient: $\Gamma = \frac{\gamma_0 - \gamma_1}{\gamma_0 + \gamma_1}$
- The transmission coefficient: $T = e^{-\gamma_1 L}$

where $\gamma_0 = j\frac{2\pi f}{c}$ and $\gamma_1 = j\frac{2\pi f}{c}\sqrt{\varepsilon_r^*}$ are the propagation constants in the transmission lines filled with free space and the sample respectively; $c = 2.9979 \cdot 10^8$ is the speed of light in free space (m/s); L is the length of sample of the material under test (m); f is the measurement frequency (Hz).

3. RESULTS AND DISCUSSION

In order to validate the measurements provided by capacitive technique and coaxial line, a liquid reference material has been used: decanol. In addition, two solid materials have been selected:

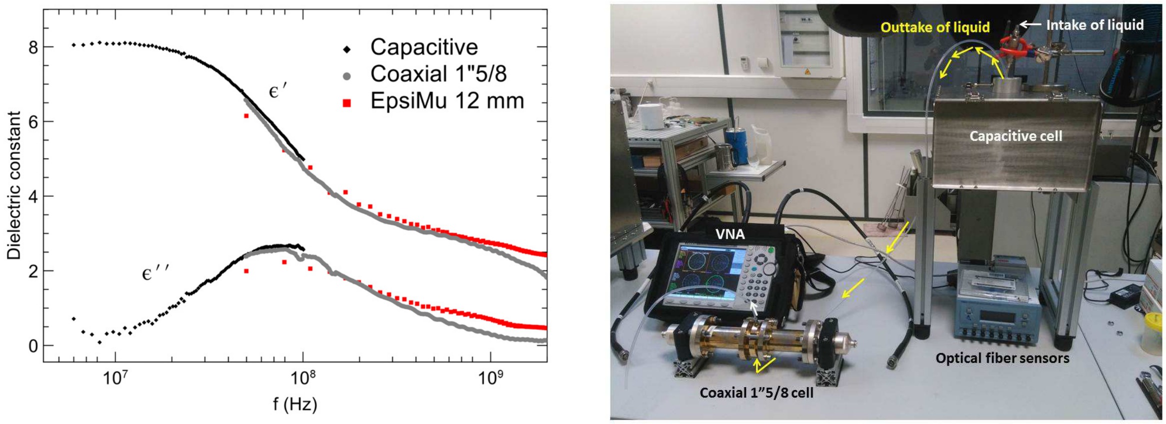
- Low loss material: Poly (tetrafluoroethylene): PTFE.
- Medium loss material: Poly (vinyl chloride): PVC (Used as an application material).

In radio-frequency range [3 MHz–100 MHz], measurements are made with three samples of different diameters. In microwave range [50 MHz–2 GHz], using the same materials (same density) we have made coaxial samples. We compare the measurement results with *EpsiMu*® tool where the measurement is done by using a sample with the same density. All measurements are made at room temperature (22°C ± 2) and at relative humidity (37%RH ± 2).

3.1. Decanol Measurement

In this part, decanol alcohol is measured by using the three cells described before: capacitive cell, coaxial 1" 5/8 line and *EpsiMu*® tool. The capacitive and coaxial 1" 5/8 are used with the same liquid as illustrated in the Figure 2(b). Indeed, both of the samples holders dedicated to liquids have two holes which allow the intake and outtake of liquid and facilitate the preservation of the same conditions for both measurements. Figure 2(a) shows the measurement results with decanol 25°C compared to

† Description of the EpsiMu tool kit: http://www.epsimu.fr/, CCRM, Marseille.



(a) (b)

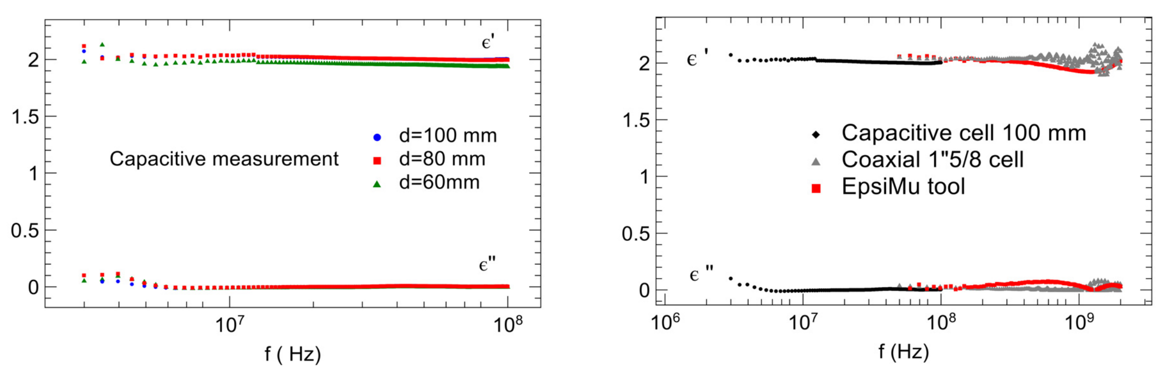
Figure 2. Decanol at 25°C: (a) Measurement results, (b) experimental liquid manipulation.

EpsiMu® measurement up to 6 GHz in the same temperature. Comparing different results, we can conclude the agreement between the measurements realized by using the capacitive cell and the coaxial 1" 5/8. This agreement can be noticed in the overlapping area of two cells [50 MHz–100 MHz]. On the other hand, measurement with *EpsiMu*® tool shows a slight difference which can be mainly explained by the difference in temperature of measurement. All measurements remain within 5% [2].

3.2. Poly (tetrafluoroethylene): PTFE

As explained previously, the sample diameter must be below 120 mm in order to avoid any edge effects in the capacitive cell. To study the diameter effect on the permittivity measurement, three samples with different diameters: 100 mm, 80 mm and 60 mm and with the same thickness 10 mm, are tested. Figure 1 shows pictures of the PTFE capacitive samples. For these three measurements, the real and imaginary parts are presented in Figure 3(a).

Analyzing the measurement results, we can observe the sensitivity to the diameter affected mostly in the real part at low frequencies. As presented for the capacitive cell, PTFE samples are used to validate the accuracy of the coaxial cell with the same material thickness of 10 mm. In Figure 3(b), we present the results of measurement with the three cells in the band [3 MHz–2 GHz]: Figure 3(b) shows that PTFE experimental permittivity spectrum presents a good agreement between the two measurement techniques in term of real and imaginary parts. At frequencies higher than 1 GHz, results show significant fluctuations that can be explained by the appearance of higher order modes and a small mismatch of the N to 1" 5/8 transitions. Results with *EpsiMu*® tool confirm the correctness of the measurement released by the new cells.



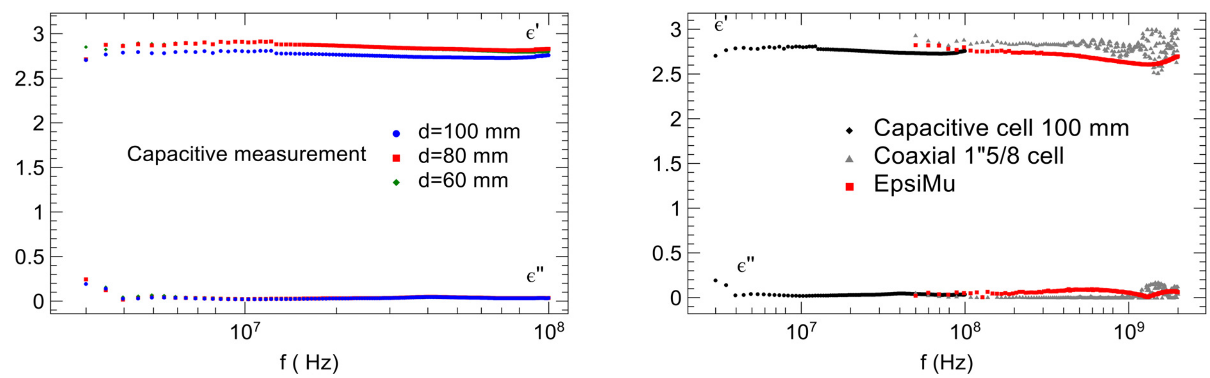
(a) (b)

Figure 3. PTFE: (a) Sample diameter effect, (b) permittivity result with different instruments.

3.3. Application to a PVC Characterization

The same procedure is applied to measure the dielectric permittivity of PVC samples with different diameters. Figure 4(a) shows the measurement results with three different diameters samples.

In order to validate the measurement results, an *EpsiMu*® sample is fabricated by using the same PVC (density) material. The measurements are carried out in the range of frequencies between [50 MHz–3 GHz], and as in the case of the preceding materials, we show the measurement results with capacitive and coaxial cell in Figure 4. Table 1 below summarizes some measurement results carried out on various solids. These results prove the good calibration of the two cells which will be mainly dedicated to the study of moisture in solids. Compared to *EpsiMu*® results, the maximum absolute error due to the sample diameter in the case of capacitive cell is about 0.08 for the real part and 0.03 for the imaginary part which are the same magnitudes in the case of PTFE compared to the reference value [3]. The optimum diameter value for solids is 100 mm.



(a) (b)

Figure 4. PVC: (a) Sample diameter effect, (b) permittivity result with different instruments.

Table 1. Selection of measurement results in some frequencies and comparison with reference values.

Material	Frequency	ϵ'	ϵ''	Method
	20 MHz	2.01	0.056	Capacitive
	60 MHz	2.03	0.0048	
	50 MHz	2.05	0.041	
PTFE	508.25 MHz	2.05	0.02	Coaxial 1" 5/8
	1 GHz	2.03	0.025	
	1.06 GHz	2.04	0.003	[3]
	100 MHz	2.02	0.0285	*EpsiMu*®
	20 MHz	2.76	0.027	Capacitive
	60 MHz	2.72	0.034	
	50 MHz	2.93	0.052	
	508.25 MHz	2.84	0.013	Coaxial 1" 5/8
PVC	1 GHz	2.82	0.026	
	315 MHz	2.72	0.066	
	507.25 MHz	2.69	0.085	*EpsiMu*®
	1 GHz	2.64	0.052	

4. CONCLUSION

This study, in the field of dielectric permittivity, presents the results from a comparison among three different measurement cells. First of all, the combination of two dielectric measurement techniques has enabled us to evaluate them against each other at the same frequencies. Furthermore, it allows for measurements of a wider frequency band due to improved accuracy. The latter is owing to the good agreement between the results of different systems, obtained under identical experimental conditions, and the inclusion of three different materials. As these two cells have been validated, they will be used with confidence in further studies of moist materials.

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