Volume Optimization of Liquids for Dielectric Permittivity Measurements at Microwave Frequencies

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Abstract—An estimation of an optimum cylindrical volume of some liquids for their accurate dielectric permittivity characterization using an open-ended coaxial probe is presented in this paper. The analysis is based on the simulated electric E-field distribution within the liquids. A criteria is proposed, where the dispersed E-field inside the sample drops to 5% of the maximum power supplied by the coaxial probe. The analysis is carried out at frequency points that lie within the most commonly used range following this technique. Comparisons with suggested values in literature are carried out, showing the differences and accuracy of the results.

Keywords—E-field, complex dielectric permittivity, penetration depth, microwave measurements, coaxial probe, permittivity measurements.

I. INTRODUCTION

Characterization of the complex dielectric permittivity of materials \( \varepsilon^* = \varepsilon' - j\varepsilon'' \) provide relevant information for food, medical, industrial and scientific applications [1]. For liquid dielectric measurement, the coaxial probe method is one of the most used due to its simplicity and wide band capability, consisting basically on the use of an open-ended coaxial transmission line with its termination submerged in the liquid-under-test (LUT); properties of the LUT are then estimated based on the reflection coefficients of the probe [1].

Recent studies report the use of the coaxial probe method for estimation of quality in food; in [2] the dielectric characterization of milk samples with different concentration and freshness levels is reported; dielectric measurements of ethanol/water solutions and some alcoholic beverages are carried out in [3] and [4]. For industrial/scientific purposes, a study on the experimental and analytical model of dielectric relaxation of alcohol and water mixtures for several concentration and temperature levels is presented in [5] and [6]. In the medical area, dielectric relaxation models and measurements are studied and presented in [7] and [8], where samples of blood plasma for various inclusions of glucose, and different cells suspension samples are analyzed, respectively. Concerning about the used frequencies, refs. [3], [4], and [6] present results at ranges going from hundreds of MHz up to 20 GHz, while [2] and [5] report ranges up to 2 GHz only; on the other hand, results presented in [7] and [8] are extended up to 40 GHz and 50 GHz, respectively.

It has been shown, from the dielectric relaxation analysis of aqueous, alcohol, and glucose solutions [3], [6], [8], that the highest relaxation frequency is occurring precisely for pure water, between 16 GHz and 17 GHz, depending on the temperature; thus, an accurate dielectric relaxation spectra of the mentioned substances, mixtures, or biological samples can be obtained from measurements going from hundreds of MHz up to 20 GHz.

Furthermore, most of the reported studies in the literature presenting experimental dielectric spectra of liquids using the coaxial probe method make use of large amounts of the LUT, usually > 50 mL, in order to ensure all the electric E-field interact within it by assuming an infinite volume from an electromagnetic view. An estimation of the E-field penetration depth in water and saline based on simulations is presented in [9] from which a reasonable amount of volume can be calculated for accurate measurements; however, the study is limited up to 4 GHz. The commercially available coaxial probe kit by Keysight® [10] suggest simply a liquid sample size in terms of insertion and diameter around the probe. The penetration depth \( d_p \) approximation, which is dependent on the material dielectric properties, is mentioned in [11] in an attempt to estimate the required amount of sample for the transmitted microwave power to drop to \( 1/e \) (36.8%) of the incident power; nevertheless, the formula works under some ideal conditions rather than open-ended coaxial lines.

A study on the E-field distribution in some liquid samples using the coaxial probe for estimation of their dielectric permittivity values is presented in this paper. The purpose of the analysis is to optimize the experimental setup by determining a minimum liquid amount while maintaining accurate measurements. Some of the most analyzed substances, such as alcohols and deionized water, are used as the LUT’s. The results are based on simulations carried out at 1 GHz, 10 GHz, and 20 GHz, in order to cover a highly used frequency range, and impedance parameters are used for the analysis.

The paper is organized as follows: Section II presents the dielectric spectra and dielectric parameters of the analyzed samples; Section III deals with the E-field distribution and power density inside the LUT’s; the literature and proposed optimizations of the setup in terms of LUT amount are detailed in Section IV; conclusions are presented in Section V.
II. DIELECTRIC PERMITTIVITY OF THE SAMPLES

The coaxial probe kit N1501A slim-form, from Keysight® [10], is used for the experimental measurements of the samples following the setup shown in Fig. 1. The samples used in this work are ethanol (99% purity), methanol (99% purity), rum (Havana Club añejo 3 años Cuban production), and deionized water, and are referred to as LUT’s A, B, C and D, respectively. Initially, all the LUT’s are prepared in 20 mL samples and are measured 5 times each from 500 MHz to 15 GHz at a temperature of 22.5°C; the obtained values are averaged. In order to estimate $\varepsilon^*$ of the liquids at the proposed frequencies, the relaxation spectra of each LUT is obtained by adjusting the measurements with the Cole-Cole equation [1]

$$\varepsilon^* = \varepsilon_s + \frac{\varepsilon_\infty - \varepsilon_s}{1 + (j\omega\tau)^{1-\alpha}},$$

where $\varepsilon_s$ and $\varepsilon_\infty$ are static- and infinite-frequency permittivities, respectively, $\tau$ represents relaxation time, and $(1-\alpha)$ determines the Cole-Cole relaxation effect. Fig. 2 plots the measured and modeled relaxation spectra of the LUT’s. Table I tabulates the relaxation parameters for each sample, along with its $\varepsilon^*$ values at the proposed frequencies for the $E$-field analysis.

III. E-FIELD DISTRIBUTION AND POWER DENSITY

A. E-field inside the LUT’s

The LUT’s and the used coaxial probe are configured in simulations using Ansys® HFSS, for three frequency points of 1 GHz, 10 GHz, and 20 GHz, each; LUT $\varepsilon^*$ values are taken from Table I. Initially, a large LUT volume of 4.7 mL is used in a cylindrical form having 10 mm base radius and 15 mm height; the coaxial probe, with outer radius of 0.97 mm and characteristic impedance $Z_0 = 50 \, \Omega$, is inserted 5 mm deep and fed with 0 dBm input power.

Figs. 3 and 4 show lateral $x$-$z$, and radial $x$-$y$ planes of the $E$-field distribution around the probe-end in LUT’s A and D, at 1 GHz and 20 GHz, respectively, with the purpose of illustrate the electrical distribution with the lowest and highest $\varepsilon^*$ values and frequencies.

As seen in Figs. 3 and 4, the maximum $E$-field is obtained exactly below the end of the coaxial probe inside the LUT; at low frequencies (1 GHz) the $E$-field trends to propagate more towards the $-z$ direction compared to the radial (horizontal); on the other hand, at higher frequencies (20 GHz), the $E$-field seems to propagate similarly in both, depth and radial.

| TABLE I. RELAXATION PARAMETERS AND PROPOSED PERMITTIVITY VALUES OF THE LUT’S FOR THE ANALYSIS. |
|------------------|-------|-------|-------|-------|
| LUT   | A     | B     | C     | D     |
| $\varepsilon_s$| 24.79 | 33.50 | 61.13 | 80.75 |
| $\varepsilon_\infty$| 4.37  | 6.57  | 7.08  | 5.62  |
| $\tau$ (ps)  | 184.53| 55.72 | 29.12 | 8.75  |
| $1-\alpha$  | 1.000 | 1.000 | 0.955 | 1.000 |
| $\varepsilon^{*}_{1\text{GHz}}$| 13.32–710.12 | 30.56–/8.61 | 58.68–/9.83 | 80.55–/4.27 |
| $\varepsilon^{*}_{10\text{GHz}}$| 4.56–/1.77 | 8.34–/7.13 | 20.75–/21.90 | 62.54–/32.30 |
| $\varepsilon^{*}_{20\text{GHz}}$| 4.63–/0.88 | 6.69–/3.90 | 12.10–/13.94 | 39.63–/37.30 |

*Although $\varepsilon^*$ values are positive in the model, they are presented in negative form ($-j\varepsilon^*$) following the convention stated in [1].
B. Power density inside the LUT’s

A quantification of the power inside the sample from the E-field, \( P = |E|^2 \), being dispersed from the center of the coaxial termination towards the boundaries of the liquid, is obtained for all the LUT’s; two lines are defined for the readings: \( -z \) (depth), and \( +x \) (radial) directions. As a mean of example, Fig. 5 shows the power density along both lines for LUT-D (deionized water) at 1 GHz and 20 GHz in log-scale.

As seen from Fig. 5, the power density drops below 0.1% of its maximum at distances larger than 1 mm towards both directions. Intensity at 1 GHz is higher than that at 20 GHz; this could be explained from the imaginary permittivity, which is related to microwave absorption, having values of \( \varepsilon_{1-GHz} = 4.27 \), lower than \( \varepsilon_{20-GHz} = 37.30 \), for LUT-D.

IV. OPTIMIZATION OF LUT VOLUME

Following a more realistic setup scenario, the LUT is configured to have a fixed radius of 2 mm, and the coaxial probe is immersed 2 mm inside the liquid, emulating the borders of a cylindrical container, and varying only its height; the simulated setup is shown in the inset of Fig. 6. In order to make a first volume optimization, from power calculations along the \(-z\) direction, the penetration depth \( d_p \), being the distance at which the density drops to \( 1/e \) (36.8%) of its maximum inside a material, is obtained for each of the LUT’s and frequencies; such value is proposed in the literature for accurate dielectric measurements [1], [10]-[11]. Showing the concept, Fig. 6 shows the normalized power density along the \(-z\) direction for LUT-A at 1 GHz. Table II tabulates the height values for each of the LUT’s achieving the \( d_p \) distance.

In an effort to further optimize the setup by enhancing accuracy of the measurements with a reasonably low amount of liquid being tested, a value of 5% of the maximum E-field inside the LUT is proposed as the limit for the sample height estimation. In this case, E-field is taken for the calculations (instead of power density or magnetic H-field) because it is directly influenced by the material dielectric parameters [1]. The normalized E-field distribution along \(-z\) is shown also in Fig. 6 for LUT-A at 1 GHz. The proposed optimized height values of each LUT are presented in Table II.

As observed in Table II, the overall maximum height value following the \( d_p \) suggestion is of 2.19 mm, corresponding to LUT-A at 10 GHz; while that at the proposed 5% E-field depth is of 3.22 mm, occurring with LUT-D at 10 GHz. Those maximum height values are configured in simulations for all the LUT’s and frequencies, and results of the normalized complex-impedance \( Z \) are obtained in the Smith chart; a de-embedding procedure of the coaxial probe is made in order to calculate \( Z \) at the probe-end plane. Impedances under the three LUT-volume scenarios are as follows: \( Z \) with the initial large LUT volume, \( Z_{V\rightarrow\infty} \); that at which the power drops to \( d_p \rightarrow 1/e \), \( Z_{5\%} \); and the proposal at which the E-field drops to 5%, \( Z_{5\%} \). The first case is taken as the reference, assuming all the microwave power is consumed inside the LUT, thus, an infinite LUT volume and no error are assumed from the electromagnetic point of view. The second case uses an LUT volume of 21.61 mm\(^3\) or 0.02161 mL; while the last configuration requires an amount of 34.55 mm\(^3\) or 0.03455 mL. In order to illustrate the concept, Fig. 7 shows the Smith chart with impedances under the three scenarios using LUT-A. The reason of analyzing \( Z \) is that it is a direct token of the complex reflection coefficient, which in turn is used for the dielectric permittivity calculations [1], [11].

From Fig. 7, it can be observed that \( Z_{V\rightarrow\infty} \) and the proposal of \( Z_{5\%} \) are very close each other, while \( Z_{5\%} \) trends to appear noticeable distant. All \( Z \) values are close to the open-circuit point, as expected from an open-ended coaxial, and they trend to get far from it while the frequency is increased, as wave dispersion becomes higher in such liquids. Fig. 8 shows deviation in \( Z \) (\( \Delta Z \) in percentage) following both criteria (literature and proposal) for LUT-volume optimization.

As observed in Fig. 8, the largest errors are obtained for \( Z_{P\rightarrow5\%} \), following the \( d_p \) criteria, with average of 28.34% and maximum of 40.13% difference for the real part, and 31.34% average and 49.21% maximum for the imaginary part. Average and maximum error values for \( Z_{E\rightarrow5\%} \) are of 3.80% and 18.15% for the real part, respectively, and 2.06% and 10.05% for the imaginary part, average and peak quantities.
Moreover, the presented analysis in terms of $E$-field distribution, complex impedance, and different frequency points result of high relevance for applications where the dielectric permittivity is to be obtained with the lowest possible amount of sample-under-test, such as in food, alcohol evaluation, blood plasma and cell suspensions in medical and chemical testing, among others.

V. CONCLUSIONS

An optimization in the volume amount of some liquids for its dielectric permittivity characterization using an open-ended coaxial probe at microwave frequencies has been presented. Four liquids having different dielectric permittivity values were first measured having a large volume and its dielectric parameters were configured in simulations at three frequency points, in order to be analyzed and tested. A proposed criteria, based on the $E$-field distribution inside the liquids has been defined and compared with the literature statement, finding the proposal to present higher accuracy while still using a relatively low amount of liquid volume; complex impedance parameters have been used for comparison of the simulations. Deviation in the normalized impedances using both criteria have been detailed and commented. The presented results and procedure can be useful for studies with other materials and frequencies within RF and microwave ranges, and different probe configurations.

REFERENCES