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# Calibration process of a capacitive probe for monitoring of reinforced concrete nuclear structures

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## ABSTRACT

Nuclear and civil engineering structures are subjected to environmental conditions (rain, wind, temperature variations, etc.), as well as to various physical, mechanical and chemical aggressions that affect their durability.

There are two types of methods for assessing structures durability: destructive methods (D) and non-destructive methods (ND). ND techniques are preferred in areas where there are limits on direct destructive methods such as the difficulty of extracting samples (accessibility, sensitivity of the structure). Monitoring approaches are complementary to ND investigation because they allow continuous recording even in inaccessible areas with limitation of human intervention.

In our work, we are interested in the development of an embedded capacitive sensor. Thus, in this paper, we propose the use of standard liquids (with well-known permittivity values) in order to carry out a prior calibration of the sensor which will be embedded later in reinforced concrete structures. Liquids were selected for their relative permittivities that cover the concrete range (2 to 30) at the frequency range of capacitive method. Liquid permittivities are extracted on one hand from literature review. On the other hand, measurements were performed with two coaxial probes. These probes were developed in research works and their principle consists on an open-ended coaxial line connected to a network analyzer. The obtained dispersion curves of the permittivities were adjusted then with the Debye model. Results show that Debye's model can adjust our measurements with a good correlation. Permittivity values in the working frequency range of the capacitive probe are thus extracted and compared to values published in literature. Once calibration is done, capacitive sensor is ready to be embedded in concrete to monitor reinforced concrete nuclear structures.

**Keywords:** Dielectric permittivity measurement, Capacitive probe, Calibration, Durability

## 1. INTRODUCTION

Durability of a structure conveys to a set of technical specifications based on direct (D) or indirect (ND) test methods. In this work, we are in an intermediate situation since we aim to develop a sensor to be drown in reinforced concrete nuclear structures in order to determine the water content gradient, an essential information for the development of most water content pathologies, in particular corrosion of reinforcements. Thus, such sensor is somewhat intrusive, but still provides ND measurements.

As part of the of the French ANR-SCaNING project, the aim is to monitor concrete using three sensors: capacitive, resistive and ultrasonic. Concerning our study, the final objective is to design an embedded capacitive sensor to follow the evolution of the water content gradient by measuring dielectric permittivity over time.

The capacitive method is considered as an electromagnetic method sensitive to water content [1].

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Many studies have been published on capacitive surface probes domain [2]. Its principle in civil engineering consists in applying an alternating current between two electrodes placed on the surface of a sample and measuring the resonance frequency of the electrode-concrete system. The circuit oscillates at a frequency close to 33 MHz in order to minimize the influence of temperature and ion conduction [2].

Therefore, and before embedding the sensor and curing concrete, a calibration procedure should be performed in standard materials with well-known permittivities in order to relate the measured resonance frequency to the permittivity values. In this article, a study was established to find standard liquids with known permittivity first based on the literature. In a second step, an experimental campaign was carried out in order to verify the permittivity values of those calibration materials with two open coaxial probes [3,4].

Hence, the definition of the permittivity, the coaxial probes and the dielectric models will be presented in section 2. Next, the different bibliographic permittivity results obtained for the standard materials will be shown. We then move on to the experimental campaign. We will define the two probes used to measure the permittivity values. The dispersion curves obtained are presented in section 4. A comparison between the results of the bibliographic and experimental study is performed at the end.

## 2. DIELECTRIC PERMITTIVITY

### 2.1. Definition

The dielectric permittivity, noted  $\epsilon$ , is related to the phenomena of electrical polarization and results from a relative displacement of the bound charges of the matter exposed to an external electric field [5]. It is expressed in Farad per meter (F/m) and appears in the constitutive relation  $\vec{D} = \epsilon \vec{E}$  where  $\vec{D}$  is the electric displacement field (C/m<sup>2</sup>) and  $\vec{E}$  the electric field (V/m).

In lossy materials like concrete, permittivity is represented by a complex quantity such as  $\epsilon = \epsilon' - j \epsilon''$ . Real and imaginary parts of the complex permittivity can be divided by the vacuum permittivity  $\epsilon_0 = 8.854187 \times 10^{-12}$  (F/m) in order to obtain the relative permittivity. The real relative part  $\epsilon'_r$  called dielectric constant takes into account the polarization potential of concrete and the imaginary relative part  $\epsilon''_r$  represents losses (both dielectric and by conduction) in concrete during electromagnetic wave propagation [2].

As mentioned in [6], the capacitance is the ability of the material to store electric energy and it depends on the real part of the complex permittivity. Therefore, we are interested in working on the real part more than the imaginary one.

Throughout the paper, we will work only on the relative permittivity. Thus for simplification purposes, the real relative part of the permittivity will be noted as  $\epsilon'$  and the imaginary relative part as  $\epsilon''$ .

### 2.2. Dielectric permittivity measurement probes

Dielectric permittivity can be measured indirectly using capacitive probes. The principle consists by measuring a resonance frequency which is converted to  $\epsilon'$  by a calibration process [7]. Otherwise, permittivity can be derived directly from the analysis of the transmission and reflection coefficients methods [8]. Coaxial transmission methods provide the advantage of measuring both dielectric constant and magnetic susceptibility. However, those methods are not applicable in many fields such as ND domains because they require the extraction of samples [9]. Reflection methods are used as a solution to this problem. A lot of work has been achieved with reflection methods [8], they proved the capacity of these methods specially in non-magnetic materials. Open coaxial probes are one of these methods, they offer the ability to perform permittivity measurement over a wide frequency band.

Two open coaxial probes are used in this work for measuring selected standard liquids permittivities. The first probe [3] consists on a device characterized by a core radius of 1.2 mm and a metallic shield radius of 4.5 mm built by abrading the surface of a connector (Figure 1.a). This device is connected to a vector network analyser (VNA) that delivers an electromagnetic signal that propagates in the material under test. The reflected signal is then collected and transformed to a

value of  $\epsilon'$  and  $\epsilon''$  by means of a capacitive model [10]. This probe will be called later as Probe 1. The second one [4] consists on a HP 85070B Hewlett Packard dielectric probe kit connected to an HP network analyser, under control of a personal computer (Figure 1.b). The measurements were carried out by ensuring the contact between the surface of the sample and the probe. The apparatus measures a coefficient of reflection which is converted to  $\epsilon'$  and  $\epsilon''$  by a theoretical approach suggested by [11]. This probe will be called Probe 2.



Figure 1: a. Measurement with Probe 1 b. Calibration with Probe 2

### 2.3. Dielectric models

The first dielectric expression was derived by Debye [12] based on permanent dipole moments in dielectric materials (equation 1). Others models was then developed based on Debye's theory such as Cole-Cole (CC), Davidson-Cole (DC).

$$\epsilon_r = \epsilon_\infty + \frac{\epsilon_s - \epsilon_\infty}{1 + j\omega\tau} \quad (1)$$

Where  $\epsilon_s$  and  $\epsilon_\infty$  are respectively the static permittivity and the optical permittivity.  $\omega$  (rad/s) is the angular frequency and  $\tau$  (s) is the relaxation time.

Debye's model is adopted in this work. In order to simulate the frequency behaviour of the real and imaginary parts of the permittivity at low frequencies (<1 GHz) a conductivity term  $\sigma$  is added to equation 1, so equation (1) becomes:

$$\epsilon_r = \epsilon_\infty + \frac{\epsilon_s - \epsilon_\infty}{1 + j\omega\tau} + \frac{\sigma}{j\omega\epsilon_0} \quad (2)$$

### 2.4. Standard liquids selection

Several solid standard materials were already used (Teflon, PVC...) to calibrate the surface capacitive probes [6]. For the present application, we are working with sensors to be embedded in concrete, thus this calibration procedure needs to be adapted. The idea proposed is to first drown the capacitive sensor in liquids or gels with known relative permittivity values in the range of concrete ones ( $2 < \epsilon' < 35$ ) at the frequencies of the capacitive method (~33 MHz). The search for gels or liquids is summarized in the following table.

Table 1: Standard liquids, references and characteristics

Liquids	References	Frequency range	$\epsilon'$	T (°C)
Methanol	[13]	0 to 100 MHz	<b>32</b>	22

	[14]	10 MHz	<b>34.8</b>	20
	[15]	1 KHz-1 MHz	<b>33.64</b>	20
Ethanol	[13]	0 to 100 MHz	<b>24</b>	22
	[16]	975 MHz	<b>23.6</b>	25
	[17]	50 KHz	<b>25.3</b>	20
Liquid Paraffin	[18]	38 MHz	<b>2.7</b>	-
	[19]	3 GHz	<b>2.25</b>	-
Acetone	[20]	Very Low frequencies	<b>21.13</b>	20
	[21]	1 GHz	<b>22</b>	-
	[22]	0 to 2 GHz	<b>20.5</b>	20
Amyl Alcohol	[23]	DC to 5 GHz	<b>15.152</b>	20
	[24]	10 MHz to 32 MHz	<b>15</b>	25
	[25]	10 MHz to 10 GHz	<b>15.6</b>	20

### 3. STANDARD LIQUIDS PERMITTIVITY MEASUREMENT

#### 3.1. Measurement process principle

Before starting measurements, a calibration process is required for the two probes. Measurements were performed in Methanol, Ethanol, Acetone, Amyl Alcohol, Liquid paraffin and a Methanol-Ethanol mixture (50%-50% proportion to obtain  $\epsilon' \approx 28$ ) with the two probes defined in section 2.2

Once measurements are done, results appear in the form of  $\epsilon'$  and  $\epsilon''$  as function of the frequency. The frequency range for the Probe 1 is [50 MHz, 2 GHz] with 3900 measurement points and for Probe 2 [200 MHz, 2 GHz] with 200 points. After that, dispersion curves obtained are adjusted by Debye's model presented in section 2.3. Since we are interested in the value of permittivity at 33 MHz, an extrapolation process has been done in order to extract the value of  $\epsilon'$  at 33 MHz. Dispersion curves, extrapolation results and a comparison with literature results are shown in the next section.

#### 3.2. Results and discussion

Optimisation was computed over the whole frequency range for both probes and for each standard liquid. Figure 2 shows the results for Methanol and Acetone. The same analysis was conducted for the four other liquids defined above (not shown here). Results for Probe 1 and Probe 2 are shown in Figure 2 with the respective indexes (1 and 2), where Re and Im stand for the real and imaginary part respectively, and "mes" or "extra" stand for measured or extrapolated (adjusted) respectively.

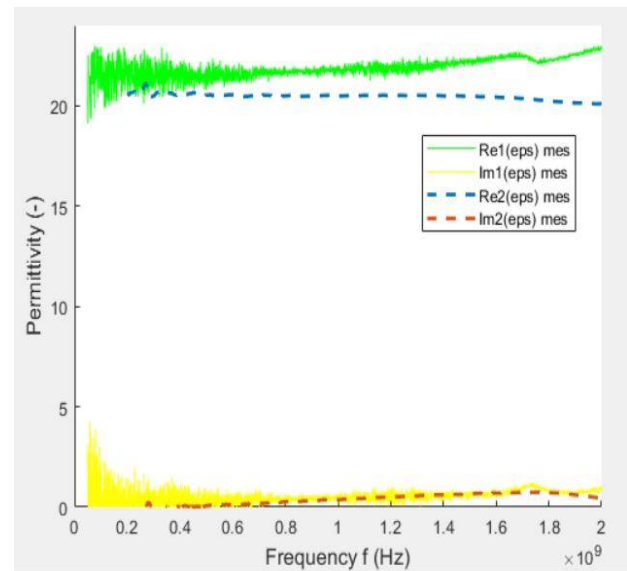
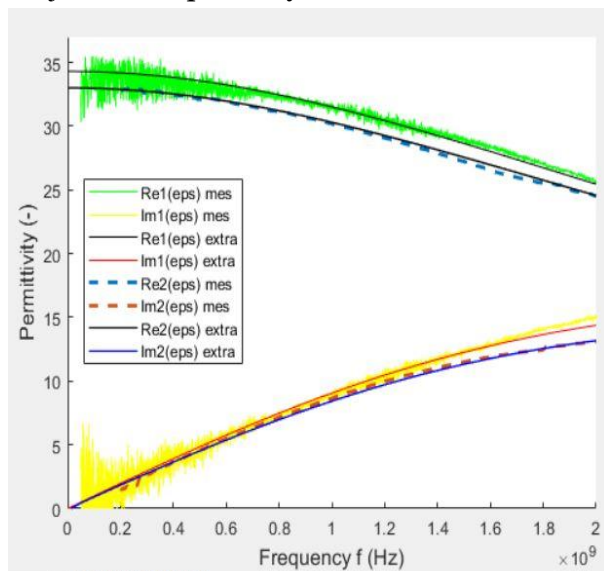


Figure 2: Dispersion curves for measured and adjusted permittivities for a) Methanol and b) Acetone. Results show that Debye's Model can fit the dispersion curves for Methanol, Ethanol, Methanol+Ethanol mixture and Amyl Alcohol. The curves of Acetone and Paraffin show a trend that is not adjustable by Debye's model due to the limited frequency range insufficient for fitting the optimised curves for these liquids. Even though, those curves tend to the literature values (Table 1) at low frequencies. For the other liquids, Table 2 shows the normalized residual " $R_n$ " defined in equation 3 for each curve as well as the extrapolated  $\epsilon'$  at 33 MHz compared to the minimum and maximum  $\epsilon'$  values of the literature (detailed in Table 1).

$$R_n = \frac{\sum(\epsilon'_{mes} - \epsilon'_{extra})^2}{N} \quad (3)$$

Where N is the number of measurement points defined in section 3.1

Table 2: Comparison of extrapolated and literature values of  $\epsilon'$

Product	$\epsilon'$ extrapolated at 33 MHz Probe 1	$\epsilon'$ extrapolated at 33 MHz Probe 2	$\epsilon'$ minimum literature	$\epsilon'$ maximum literature	$R_{n1}$ (Probe 1)	$R_{n2}$ (Probe2)
Methanol	34.3	32.9	32	34.8	0.57	0.06
Methanol +Ethanol	29.9	28.9	27.8	30.1	0.5	0.02
Ethanol	24.9	24.3	23.6	25.3	0.27	0.05
Amyl Alcohol	15.2	15.8	15	15.6	0.09	0.18
Acetone	-	-	20.5	22	-	-
Liquid paraffin	-	-	2.25	2.7	-	-

It can be deduced from Table 2 that the relative permittivity values  $\epsilon'$  of our liquids are in agreement with the literature given. The average of the two extrapolated values obtained with these two probes will be adopted in the calibration process of our embedded capacitive sensor.

#### 4. CONCLUSION

This study aimed to measure the dielectric permittivity of standard liquids that will serve as a calibration input for future measurements with our capacitive sensor in reinforced concrete nuclear structures. To do this, we started by a bibliographic research on standard liquids. Some liquids were selected for their relative permittivity values  $\epsilon'$  that cover the concrete ones at working frequencies of the capacitive method. At a later time, measurements with two coaxial probes were conducted. Adjustment with Debye's model was the third step. At last, an extrapolation process was performed in order to obtain permittivity values at working frequencies of the capacitive method. Values obtained with the two probes were confirmed by the literature. For the future steps, the average of the two extrapolated values obtained with the two probes will be considered for the calibration process of our capacitive sensor that will be embedded in reinforced concrete nuclear structures.

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