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# Determination of liquid water content and dielectric constant in porous media by the capacitive method

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## Abstract

A capacitive sensor-based apparatus has been settled to determine the liquid water amount and dielectric constant in consolidated porous media. This technique relies on the dielectric properties of water, air, and mineral substrate. The experimental procedure is described for successively oven-dried samples at 323 K. It allows us to determine the sample dielectric constant as a function of the sample water amount. For limestones from Caen region, an affine relationship is found at 293 K. This is then compared with other empirical soils data and with existing homogenisation techniques applied to undeformable heterogeneous dielectrics. *To cite this article: T. Fen-Chong et al., C. R. Mecanique 332 (2004).*

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## Résumé

**Détermination, par la méthode capacitive, de la teneur en eau liquide et de la constante diélectrique dans un milieu poreux.** Un dispositif de sonde capacitive a été mis au point pour déterminer la teneur en eau liquide et la constante diélectrique dans un milieu poreux consolidé. Cette technique utilise les propriétés diélectriques de l'eau, de l'air, et du substrat minéral. Le procédé expérimental est décrit pour des échantillons séchés au four à 323 K. On peut alors évaluer la constante diélectrique de l'échantillon en fonction de sa teneur en eau. On trouve une relation affine dans le cas des pierres calcaires de Caen à 293 K. Ceci est ensuite mis en perspective avec d'autres résultats empiriques obtenus sur des sols ainsi qu'avec des modèles classiques d'homogénéisation de diélectriques hétérogènes et indéformables. *Pour citer cet article : T. Fen-Chong et al., C. R. Mecanique 332 (2004).*

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## 1. Introduction

Most civil engineering or geotechnical durability problems involve water inside porous media through mechanical softening of soils and structure foundations, harmful chemical agents ingress [1], or freezing [2]. Water content also plays a key role in soil physics, agronomy, or biology [3,4], and in remote sensing-based geophysical exploration through the dielectric properties of Earth's materials [5–7].

One way to evaluate water content in porous media consists in using the contrast between the dielectric constant of liquid water and those of water vapour, air, and solid minerals. This can be conveniently done by either time domain reflectometry or capacitance techniques [8].

Time domain reflectometry is based on the delay produced on an electromagnetic wave that travels along a probe inserted in the medium with pulse rise frequency typically in the 10 Hz to 1 GHz range. It has become the most popular technique to determine soil water content [9,10,3]. The capacitive method consists in measuring the electric capacity of a sample [11,12,10,13]. It operates typically in the radio-frequency range from 10 MHz up to several 100 MHz. It has been particularly studied in the scientific and engineering network of the Laboratoire Central des Ponts et Chaussées for engineering in situ practice and low-cost advantages (see reviews in [14,11] and recent development for centrifuged porous media by Dupas et al. [15]). A capacitive probe has also been carefully developed for soil moisture measurement by the Institut National de la Recherche Agronomique jointly with the Institut Universitaire de Technologie de Marseille [12,16].

This Note deals with the settlement and use of a 33 MHz oscillator circuit-based capacitive method on consolidated porous media such as Caen stones. An affine curve between the sample dielectric constant and its water content is obtained from drying tests. This is compared with soil data since none are available in the literature for consolidated porous media, and analysed through homogenisation techniques.

## 2. Dielectric capacitive method

### 2.1. Basic measurement principle

The local spatial redistribution of polarised electrical charges in a material sample under an applied electric field is characterised by its dielectric constant (dielectric permittivity relative to that of free space)  $\epsilon^* = \epsilon - j\epsilon_r$ , where  $j^2 = -1$ . The real part,  $\epsilon$ , relates to the behaviour of an ideal insulator and characterises the degree of electric polarisability of the material while the imaginary component,  $\epsilon_r$ , is associated with the electric energy dissipation into heat, due to electrical conduction and polarised charges fluctuations, and is responsible for the phase difference between the applied alternating electric field and the polarisation. Both real and imaginary parts are accessible by measuring the medium electric impedance [5,3], while a simpler capacitive sensor-based apparatus can provide the real part  $\epsilon$  via the medium electric capacity (note that  $\epsilon_r$  can be simply obtained by a resistive method).

Partially water saturated porous media such as soils, stones, and concrete, are composed of liquid water, water vapour, air, and solid minerals; their dielectric constant depends on the dielectric constant and volume fraction of each phase. Accordingly, any variation of the liquid water content (for instance by liquid–vapour phase change during drying) gives rise to capacity change. This is all the more sensible since the dielectric constants of water vapour, air, and solid minerals are usually considered constant and equal to 1, 1, and between 2 to 10, in contrast to the high value of the dielectric constant of bulk pure water  $\epsilon_w = 80.2$  at 20 °C [17]. Although the physico-chemical interactions slightly affect the picture, several authors consider that between 1 MHz and 1 GHz the soil dielectric constant is mainly sensitive to the soil water content and weakly sensitive to the excitation frequency, soil-specific parameters such as grain-size distribution, chemical composition, apparent density, soluble salts (under a critical value), and temperature [9,11]. Recently this assumption has been reduced to the frequency range 25–200 MHz [3]. In addition, comparative studies between different methods show satisfactory agreement, such that [3,13] drew the conclusion that soil water content can be easily derived by a simple capacitive measurement.

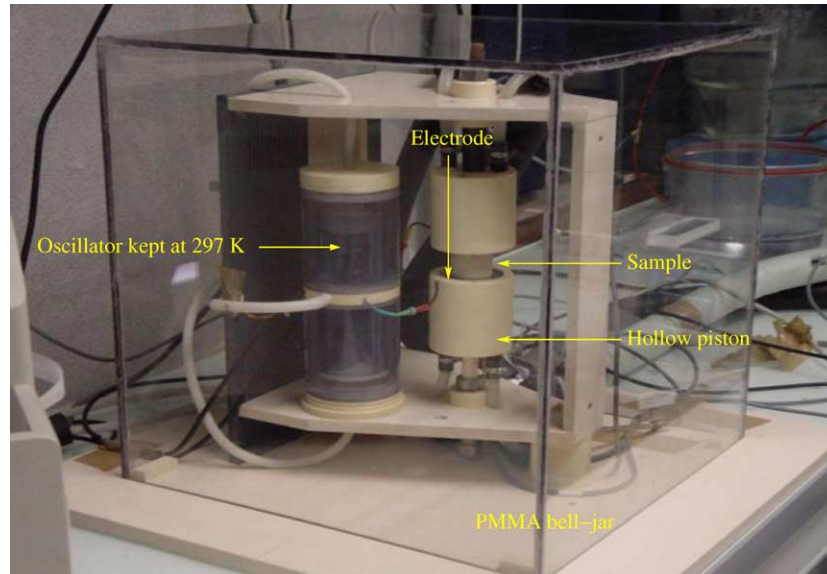


Fig. 1. Experimental set-up. The electrodes are 6 cm long with variable separation distance. The cylindrical samples are around 1 cm thick and have a mean diameter of 4 cm. The oscillator circuit has been placed into a plastic box embedded in a hollow shell containing thermostated water to maintain the oscillator circuit temperature at 24 °C.

## 2.2. Experimental equipment and procedure

The sample is inserted between two plane and circular stainless steel electrodes as shown in Fig. 1. All of them are connected to a 30–50 MHz oscillator circuit. This device, designed and manufactured by the Laboratoire Régional des Ponts et Chaussées at Rouen (France), was implementing a frequency divider in order to reach a low frequency range (in the order of several kHz) before transmitting the signal to a digital storage oscilloscope TDS1002 TEKTRONIX (for signal-shape check) and to a multifunction counter-timer 34907A AGILENT. The excitation frequency chosen in this study is about 33 MHz.

The experimental apparatus measures the resonant frequency  $\tilde{\nu}$ . A linear calibration curve between  $\tilde{\nu}$  and capacity  $C$  is obtained by the use of different commercial ceramic capacitors: each of these electronic components was associated in parallel with different commercial resistors in order to check that the ionic conduction-induced dielectric loss (part of the resistive term  $\epsilon_r$ ) of the non-ideal insulator porous materials does not influence the capacitive measures. Then, for each sample of known geometry and water content,  $\tilde{\nu}$  is recorded and turned into  $C$ , the latter being related to the sample dielectric constant by the well-known relation  $C = F\epsilon$ , where  $F$  is an edge effect-corrected geometrical factor [18]. Water content is here expressed as water saturation degree (ratio of water volume fraction over the sample porosity),  $S_\ell$ , and measured by weighing, thus providing the  $\epsilon(S_\ell)$  curve.

## 3. Experimental tests on Caen stones

### 3.1. Materials characterisation and preparation

Caen stones are homogeneous materials with fine grains. For our materials, mean porosity  $\phi$  is about 39% with most pore radii between 10  $\mu\text{m}$  and 30  $\mu\text{m}$ , the apparent density  $\rho_a \simeq 1.6 \text{ g/cm}^3$  and the mineral density about 2.6  $\text{g/cm}^3$  (all data from mercury porosimetry). The mineral substrate is composed of mainly calcite ( $\text{CaCO}_3$ ) and some quartz (rhomboedric silica) according to X-ray analysis. Chemical analysis of the water solution in

contact with a saturated Caen stone sample for 1 month (for thermodynamic equilibrium) revealed a 70 mg/l  $\text{Ca}^{2+}$  concentration, some traces of  $\text{K}^+$  and of other non-identified elements, and a  $\text{pH} \simeq 8.3$ . The pore water dielectric constant,  $\epsilon_w$ , was evaluated by comparing the resonant frequency of the previous solution with those of different fluids of known dielectric constants. It is found twice that  $\epsilon_w \simeq 80$ , which coincides with the real dielectric constant of pure water. The mineral substrate dielectric constant,  $\epsilon_m$ , was obtained by saturating the porous material with successively different fluids. This was done with air (dried samples), hexane ( $\epsilon_f = 1.9$ ), ethanol ( $\epsilon_f = 25$ ), and water (saturated samples). The real dielectric constant of each sample was then measured, thus giving an experimental curve  $\epsilon(\epsilon_f)$ . Estimation of  $\epsilon_m$  was done by looking for the dielectric constant of a fictive fluid for which the whole sample is dielectrically homogeneous. In this way it is found that  $\epsilon_m \simeq 3.3$ . The same procedure yields a value of 3.8 for silica constituting home-made fused glass beads, which is in good agreement with data handbook of [19].

Cylindrical samples were cored and sawed from Caen stone benches. They were then cleaned up from scraps, dried at 323 K in an oven for 3 days before weighing. Each sample was then saturated with degassed home-made distilled water at 3 kPa air pressure. Different water degree saturations were achieved by using an oven at 323 K. Then the sample was rapidly transferred into the capacitive electrodes, wrapped in plastic adhesive and tests were run for 10 min. Weighing was realised just before and after each capacitive test to evaluate the water mass content and to check that no significant water evaporation occurred.

### 3.2. Results and data analysis

Fig. 2 shows an affine relationship between the sample dielectric constant and the sample water degree saturation:  $\epsilon = 2.1 + 9.4S_\ell$  (correlation coefficient of 0.997). As our material mean porosity  $\phi \simeq 39\%$ , the previous linear fit can be expressed in terms of sample volumetric water content  $\theta_\ell$  (ratio of liquid water volume over the sample volume) in the range  $0 \leq \theta_\ell \leq 39\%$ , see Table 1. Affine  $\epsilon(S_\ell)$  curves have also been obtained by [12] at 38 MHz (other ones are asserted in [14]). Table 1 reports one of their results which is closest to ours. All the other experimental results are not affine for the whole water volumetric content range (0–50%) and only Topp et al.'s

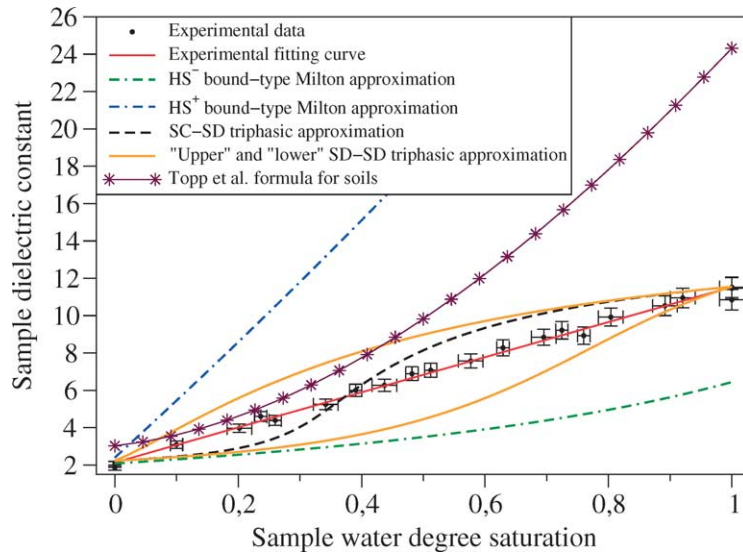


Fig. 2. Sample dielectric constant as a function of sample water degree saturation determined at a frequency of 33 MHz with some theoretical homogenisation predictions.  $\text{HS}^-$  and  $\text{HS}^+$  refer to the lower and upper Hashin–Shtrikmann bounds, SC stands for self-consistent scheme and SD refers to the self-differential scheme.

Table 1

Review of some water volumetric content in porous media. Other empirical models are reported in [8]. TDR: time domain reflectometry, IM: impedance measurements, CM: oscillator circuit-based capacitive method

Authors' work	Type of porous media	Experimental fit
Topp et al. [9] (TDR, 1 MHz $\leq \nu \leq$ 50 MHz)	Mineral soils with some organic material traces: sandy loams, clay loams + water with 0.01 M CaSO <sub>4</sub>	$\epsilon = 3.03 + 9.3\theta_\ell + 146.0\theta_\ell^2 - 76.7\theta_\ell^3 \pm 1.1$
Topp et al. [9] (TDR, 1 MHz $\leq \nu \leq$ 50 MHz)	Glass beads (30 $\mu$ m) + water or water with 2.112 mg/l NaCl	$^\dagger \epsilon = 3.6 + 41.4\theta_\ell + 63.2\theta_\ell^2 - 26.7\theta_\ell^3 \pm 0.4$
Campbell [5] (IM, $\nu =$ 50 MHz)	Clay and silty soils with apparent density, mineral density, and porosity close to those of Caen stones + water	$\epsilon = 3.27 + 0.48\theta_\ell - 0.012\theta_\ell^2 + 0.00037$
Gaudu et al. [12] (CM, $\nu =$ 38 MHz)	4 mm silty granulates + water	$\epsilon = 2.8 + 27.4\theta_\ell$
Eller and Denoth [3] (IM, $\nu =$ 32 MHz)	Various soils + water	$\theta_\ell = 1.484\epsilon - 0.006\epsilon^2 - 0.332$
Gardner et al. [13] (CM, 80 MHz $\leq \nu \leq$ 150 MHz)	Various soils containing sand, silt, clay, and organic carbon + water	$\sqrt{\epsilon} = -1.208 + 9.93\theta_\ell + 2.454\rho_a$
Gardner et al. [13] (CM, 80 MHz $\leq \nu \leq$ 150 MHz)	Silica sand + water	$\sqrt{\epsilon} = 1.075 + 8.528\theta_\ell + 0.271\rho_a$
This work (CM, $\nu =$ 33 MHz)	Calcite and quartzite Caen stones + home-distilled water + 70 mg/l Ca <sup>2+</sup>	$\epsilon = 2.1 + 24.1\theta_\ell$

<sup>†</sup> Mean values from four run tests.

results show a fit affine component in the same order as our affine curve fit. In these works, there usually exists a linear domain over  $0 \leq \theta_\ell \leq 25\%$  (or  $0 \leq S_\ell \leq 0.6$ ), which is less than ours: in fact one can see that the non-affine part of Topp et al. fit leads to higher values than ours for  $S_\ell \geq 0.6$ . This may be due to distinct microstructural morphologies of the materials and to the unconsolidated state of soils, as confirmed by the following up-scaling models.

Our result can be approximated by classical homogenisation techniques applied to undeformable triphasic partially water saturated porous media [20–22] (see formulae in Appendix), provided that a slightly higher mean porosity is introduced ( $\phi = 41.2\%$  instead of 39%, which should lie within the experimental error). The rigorous second order Hashin–Shtrikmann bounds are calculated through the Milton multicoated spheres approximation (identical to the Maxwell–Garnett approximation for biphasic composites). Search for better bounds leads to combination of self-consistent (SC) and self-differential (SD) schemes as follows. Since the spatial distribution of gas and water inside a pore is not known, the self-consistent scheme was first applied for the pore network and then combined with the self-differential scheme for modelling the pore/mineral skeleton morphology. On the other hand, in the SD-SD approximation, both in-pore mixture and pore/skeleton are represented by the self-differential scheme. By this way, all SC-SD and SD-SD combinations yield the same constant value  $\epsilon(S_\ell = 0) = 2.2$  near the experimental one, 2.1. The fitted experimental value of about 11.5 is also recovered at full saturation by all these approximations, which is not the case if a 39% mean porosity was taken (this would lead to  $\epsilon(S_\ell = 1) = 10.7$ ). Accordingly, the SC-SD approximation is relatively close to the experimental curve and ‘upper’-like and ‘lower’-like triphasic composites bounds are obtained respectively by incrementing air inclusions into a water continuous phase and by the inverse procedure in the SD-SD approximations, but no theoretical proof of this guess exists to our knowledge.

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## Appendix. Homogenisation formulae

For  $n$  isotropic phases  $i$  of  $f_i$  volume fraction and  $\epsilon_i$  dielectric constant, the Hashin-Shtrikmann bounds are given by the Milton multicoated spheres approximations

$$\left( \sum_{i=1}^n f_i (2\epsilon_{\min} + \epsilon_i)^{-1} \right)^{-1} - 2\epsilon_{\min} \leq \epsilon \leq \left( \sum_{i=1}^n f_i (2\epsilon_{\max} + \epsilon_i)^{-1} \right)^{-1} - 2\epsilon_{\max}$$

where  $\epsilon_{\min} = \text{Min}_{i \in [1;n]} \{\epsilon_i\}$  and  $\epsilon_{\max} = \text{Max}_{i \in [1;n]} \{\epsilon_i\}$ , while the self-consistent (or Bruggeman) approximation comes down to solve the following implicit equation for the unknown homogeneous equivalent medium dielectric constant  $\epsilon^{\text{SC}}$

$$\sum_{i=1}^n f_i \frac{\epsilon_i - \epsilon^{\text{SC}}}{\epsilon_i + 2\epsilon^{\text{SC}}} = 0$$

For biphasic material made of an inclusion phase (index 2) embedded in a continuous matrix phase (index 1), the incremental procedure of the self-differential scheme leads to the following implicit equation

$$\left( \frac{\epsilon_2 - \epsilon^{\text{SD}}}{\epsilon_2 - \epsilon_1} \right) \left( \frac{\epsilon_1}{\epsilon^{\text{SD}}} \right)^{1/3} = f_1$$

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