TDR-based Water Content Estimation on Globigerina Limestone Through Permittivity Measurements

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Abstract – **Most monuments and historical buildings in the Maltese Islands are made of the local Globigerina Limestone (GL). This type of stone, however, is very delicate and prone to degradation caused by the environmental conditions of the islands. Hence, for the preservation of the Cultural Heritage monuments, it is necessary to promptly assess the health status of these structures and, in particular, their water content (which represents one of the major causes of degradation).**

Starting from these considerations, in this work, a time domain reflectometry (TDR)-based method for estimating water content of GL is presented. More specifically, the proposed method relies on estimating the water content value of the GL structure from TDR-based dielectric permittivity measurements. To verify the suitability of this system, experimental tests were carried out on a GL sample. The results anticipate the strong potential of the proposed method for practical applications in the Cultural Heritage diagnostics.

I. INTRODUCTION

The Maltese Islands are mostly composed of sedimentary rocks, such as the Coralline Limestone and Globigerina Limestone (GL), which have been used since prehistoric times for the construction of buildings and monuments [2]. Unfortunately, stones used in sculpture and architecture, as well as rocks in their original location, are exposed to environmental weathering. This results in modifications of the microstructure of the stones (such as open porosity, chemical-mineralogical composition of the phases, etc.), which, in turn, lead to changes of the me-

chanical properties [3].

This is particularly true for GL, which is highly porous and is typically used both in modern and historical architecture. Due to Malta's marine environment, salt crystallization in the stone's pore spaces (especially through alveolar weathering) is one of the major causes of damage in many buildings made of GL [4]. In addition to this, also rain and the presence of moisture is another major deterioration cause. As a result, the study of water absorption characteristics of GL (which are fundamental when durability is being considered) has attracted much research interest [5].

It is well known that the dielectric characterization of a material represents a useful solution for assessing water content. Nevertheless, in the literature, little information is available on the dielectric characteristics of GL.

Starting from these considerations, in this work, a time domain reflectometry (TDR)-based method for estimating water content of GL is presented. The proposed method relies on estimating the GL's water content value from TDR-based dielectric permittivity measurements. To verify the suitability of this system, experimental tests were carried out on a GL sample both to investigate the dielectric characteristics of GL and to establish an empirical relationship between water content of the stone and dielectric permittivity. In practical applications, by measuring the dielectric permittivity of the structure, it would be possible to retrieve the corresponding water content. As reported in the following, the obtained results anticipate the strong potential of the proposed method for practical applications in the Cultural Heritage diagnostics.

II. BACKGROUND

TDR is a very flexible measurement technique, and thanks to its adaptability, it is employed for a number of applications, such as soil moisture content measurements [6]; for the characterization of electrical components [7]; for the leak localization in underground water pipes [8]; for dielectric permittivity measurements; for wire faults localization [9]; for moisture content measurements in the agrifood industry [10]; for rising damp monitoring [11]; and, possibly, also for investigating magnetic properties [12].

The typical instrumental setup for TDR-based in situ water content measurements includes *i*) a portable reflectometer; and *ii*) a probe (in addition to a laptop for data processing).
In TDR measurements, an appropriate electromagnetic test signal (typically, a voltage step signal with very fast risetime or a pulse-signal) is propagated along a probe, which is inserted into or placed in contact with the material under test. Any impedance variation causes the partial reflection of the propagating test signal. The reflected signal carries information on the dielectric characteristics of the material in which the probe is inserted. Therefore, through a suitable data-processing of the reflected signal, it is possible to retrieve other intrinsic (qualitative and quantitative) characteristics of the material under test.
The direct output of a TDR measurement is a reflectogram, which displays the reflection coefficient (ρ) as a function of the apparent distance, d^{app}. The behavior of ρ is strictly associated with the impedance variations encountered by test signal as it propagates along the probe. The quantity d^{app} can be considered as the *equivalent physical distance* that would be travelled by the electromagnetic test signal, in the same time interval, if the signal were propagating at the speed of light in vacuum, $c = 3 \times 10^8$ m/s. The quantity d^{app} is related to the 'actual' physical length traveled by the test signal, d, through the following equation:

$$d^{\mathrm{app}} = \sqrt{\varepsilon^{\mathrm{app}}} \cdot d, \qquad (1)$$

where $\varepsilon^{\mathrm{app}}$ is the apparent relative dielectric permittivity of the material in which the probe is inserted.
In practice, to evaluate the dielectric characteristics of materials, the apparent length of the probe is measured from the reflectogram, and the corresponding $\varepsilon^{\mathrm{app}}$ is retrieved.

As for TDR-based water content measurements, they rely on the fact that the relative dielectric permittivity of water (approximately 78 at 1.8 GHz ($25^{o}C$)[13]) is considerably higher than the typical relative permittivity of many dry stones. Therefore, the presence of water leads to a considerable increase of the overall dielectric permittivity of the moist stone.
On such bases, different methods can be adopted for retrieving the water content value, θ, from TDR measurements. A simple and adequately accurate approach for deriving the functional relationship between θ and $\varepsilon^{\mathrm{app}}$ relies on the use of empirical, material-specific calibration curves. These are derived by adjusting the stone to a reference (known) water content value, and then measuring the corresponding $\varepsilon^{\mathrm{app}}$ values. The measured $\theta - \varepsilon^{\mathrm{app}}$ are then fitted and the obtained curve is used as calibration curve. For successive water content measurements on the same material, it suffices to measure $\varepsilon^{\mathrm{app}}$ and the corresponding (unknown) θ value is retrieved from the calibration curve.

III. MATERIAL AND METHODS

A. *Material and instrumental setup*

GL is a very highly porous limestone, mainly composed of calcite crystals and fossils, including globigerinae, shells and sea urchins (it may also contain small quantities of quartz, feldspars and clays). GL has fine grains, bonded by carbonatic cement, which however does not completely fill the pores [3]. As reported in [1], understanding the deterioration mechanisms of Globigerina Limestone permits criteria for proper conservation treatment to be established. In the experimental tests reported in this work, a GL sample with dimensions 11.5 cm × 5.5 cm × 4.5 cm was used.

TDR measurements were performed through the Campbell Scientific TDR100 instrument, which is a low-cost portable TDR unit. The test signal is a step-like voltage signal with rise time of approximately 200 ps and amplitude of 250 mV. One of the advantages of this TDR instrument is that it supports multiplexers, thus allowing the simultaneous connection of several probes to a single TDR unit. This feature is particularly useful for practical purposes, as it can lower the implementation costs.

For the TDR measurements, a two-rod probe fabricated in-house was used. The length of the probe was approximately L^{P} = 9.5 cm. Each rod was made of brass and its diameter was 5 mm, while the mutual distance between the rods was 15 mm (Fig. 1). The probe head, which provided mechanical stability, was made in PVC. Inside the probe head there is the electric transition from a bifilar transmission line (represented by the two rods) into a coaxial transmission line (for the connection to the TDR instrument).

TDR measurements were carried out by drilling two holes in the GL sample block. The dimensions and mutual distance between the holes were such the probe could be inserted and could be adherent to the material.

Moreover, experimental characterization of the dielectric properties of GL was also conducted under controlled conditions in the laboratory. Samples of GL were obtained from various boreholes around Malta and measured using a transmission waveguide technique which enabled measurements from 1.7 to 2.6 GHz. GL samples were cut to fit the rectangular waveguide aperture using a Vector Network Analyzer (VNA), as shown in Fig. 2. The measured reflection and transmission coefficients were then converted to the corresponding permittivity values using the Nicholson-
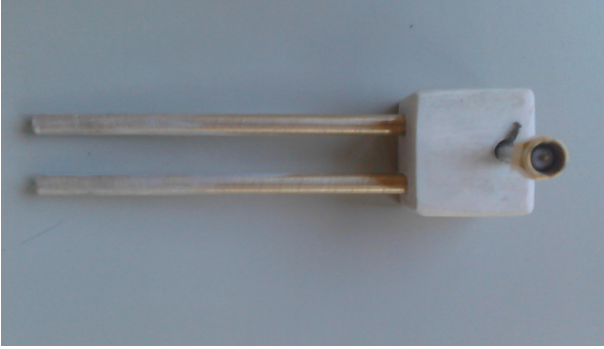
Fig. 1. Picture of the used two-rod probe.

Sample holder

Fig. 2. The samples and measurement setup used to measure the permittivity of GL in the laboratory.

Ross Weir method [14].

B. Methods

First, the stone sample was dried at 105 °C in an oven, for 24 hours. The weight of the dry sample, W^{dry}, was measured through an electronic balance (with an uncertainty of 0.1 g).

Successively, the sample was immersed into tap water for three hours, so as to make sure that the sample was fully saturated with water. Because of the high porosity of the stone and because of the small dimensions of the sample, this time was sufficient to achieve saturation.

Starting from the water-saturated condition, the GL sample was let to dry in air. Approximately every three hours, the weight of the sample, W_{i}, was measured. The

corresponding volume of water, $V_{\mathrm{i}}^{\mathrm{wat}}$, was evaluated as

$$V_{\mathrm{i}}^{\mathrm{wat}} = \frac{W_{\mathrm{i}} - W^{\mathrm{dry}}}{\delta} \qquad (2)$$

where $\delta \cong 1 \ \mathrm{kg/dm^3}$ is the density of water.

Hence, for each i-th moistening condition, the reference water content value was evaluated as

$$\theta_{\mathrm{i}}^{\mathrm{ref}} = \frac{V_{\mathrm{i}}^{\mathrm{wat}}}{V^{\mathrm{stone}}} \qquad (3)$$

where V^{stone} is the total volume of the stone sample.

In the drying procedure, a total of 22 water content values were obtained, starting from $\theta_1^{\mathrm{ref}} = 23.3\%$ to $\theta_{22}^{\mathrm{ref}} = 0.0\%$. For each value of $\theta_{\mathrm{i}}^{\mathrm{ref}}$, the corresponding apparent dielectric permittivity of the stone sample was evaluated through TDR measurements. A similar procedure of drying and saturation of GL samples obtained from boreholes was done. This enabled dielectric measurements of dry and water-saturated GL which resulted in a range of permittivity values obtained under controlled conditions in the laboratory. The procedure followed for drying and saturating the samples is described in Fig. 3.

IV. EXPERIMENTAL RESULTS

Fig. 4 shows one of the acquired reflectograms in the different water content conditions (in particular, the figure shows 22 reflectograms, each referring to a different value of $\theta_{\mathrm{i}}^{\mathrm{ref}}$ value). As expected from (1), as the sample dried up, the apparent length of the probe decreased.

For each $\theta_{\mathrm{i}}^{\mathrm{ref}}$ value (for $i = 1, ..., 22$), the apparent dielectric permittivity was evaluated as follows. Ten repeated TDR measurements ($j = 1, ..., 10$) were carried out.

For each of the ten reflectograms, the corresponding first derivative was evaluated, and the $L_{\mathrm{i,j}}^{\mathrm{P,app}}$ value was determined from the peaks of the first derivative [10], as shown in Fig. 5. This figure shows one of the ten repetitions carried out for $\theta_{\mathrm{i}}^{\mathrm{ref}} = 13.9\%$.

Finally, for each $\theta_{\mathrm{i}}^{\mathrm{ref}}$ value, the ten evaluated values of $L_{\mathrm{i,j}}^{\mathrm{P,app}}$ were averaged, and the corresponding apparent dielectric permittivity was evaluated by rearranging (1):

$$\varepsilon_{\mathrm{i}}^{\mathrm{app}} = \left(\frac{\frac{1}{10} \sum_{j}^{10} L_{\mathrm{i,j}}^{P,app}}{L^P} \right)^2 \qquad (4)$$

The obtained $\theta_{\mathrm{i}}^{\mathrm{ref}} - \varepsilon^{\mathrm{app_i}}$ points are plotted in Fig. 6, to retrieve the calibration curve. Results show that the value of the dielectric constant varies from approximately 4.3 (in dry condition) to 13.0 (in water-saturated condition). Hence, in practical diagnostics on Cultural Heritage structures, dielectric permittivity measurements can be used to infer the water content of the monitored structure. It should be mentioned that, in this work, a two-rod probe was used to better characterize the dielectric permittivity of GL. However, in practical diagnostics on Cultural
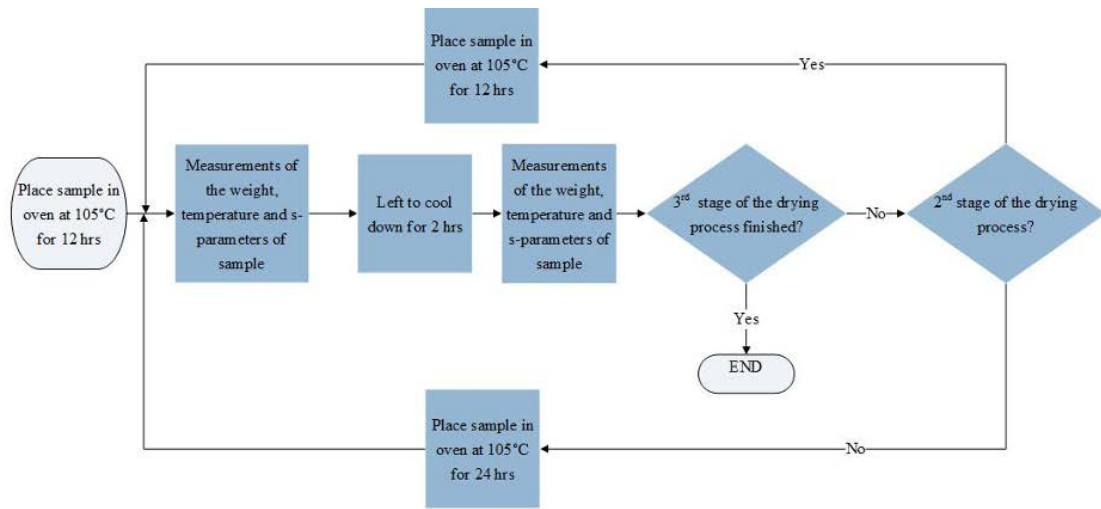
(a) Drying procedure
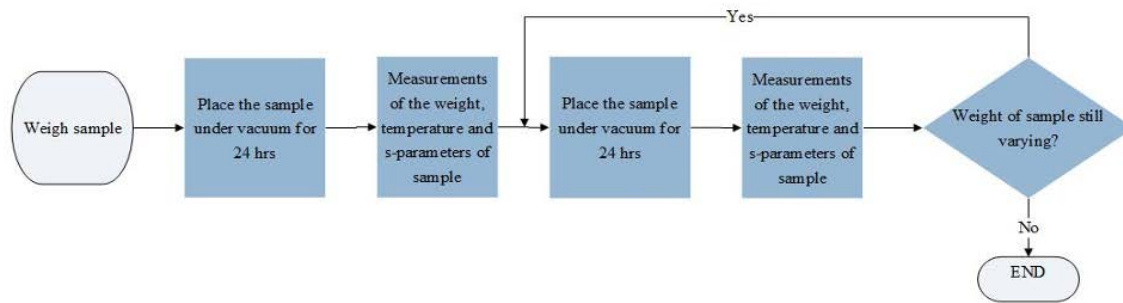
(b) Saturation procudure

Fig. 3. The procedure followed for (a) drying and (b) water-saturing the samples for the dielectric measurements in the laboratory.
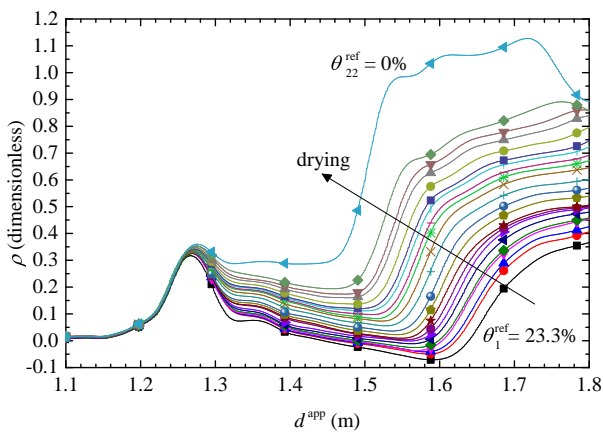

Fig. 4. Reflectograms acquired for different values of θ_i^{ref} (i = 1, ..., 22), as the sample dried up.
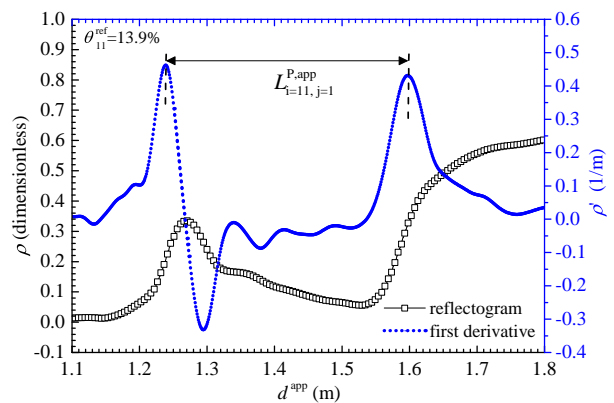

Fig. 5. Example of one of the ten reflectogram acquired for $\theta_{11}^{\mathrm{ref}} = 13.9\%$. The corresponding first derivative and the apparent length of the probe are also indicated.
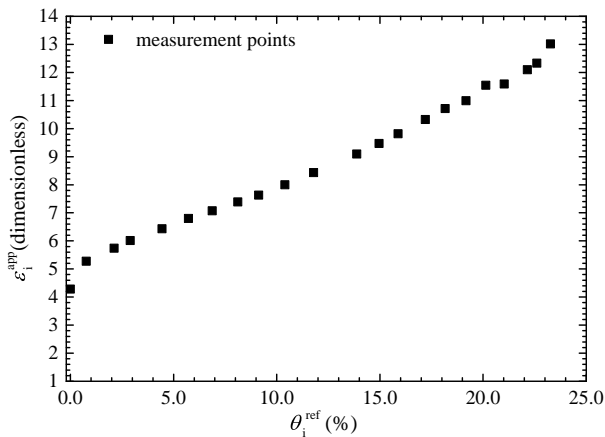
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Fig. 6. Empirical relationship between $\theta_i^{\text{ref}} - \varepsilon_i^{\text{app}}$.
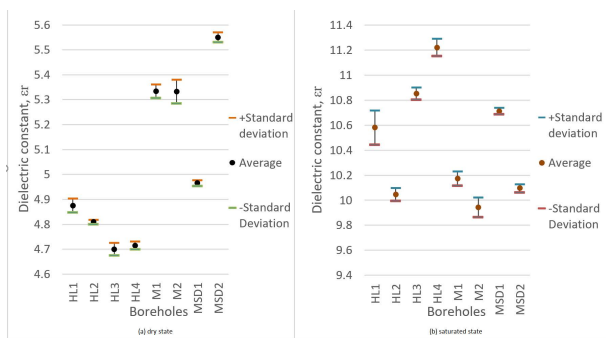

Fig. 7. The average permittivity of GL samples as obtained from each borehole for the (a) dry state and (b) saturated state, together with the corresponding 95% confidence interval.

Heritage structures, a noninvasive probe (such as a patch antenna) may be used to guarantee noninvasiveness of the system. The averaged value of the measured permittivity for both saturated and dry state of all the samples within each borehole as obtained from measurements in the laboratory are presented in Fig. 7. In particular, at 2 GHz, the average permittivity of GL in the dry state resulted to be 5.025 ± 0.065. In the saturated state at 2 GHz, the average permittivity of GL resulted to be 8.396 ± 0.072.

V. CONCLUSION

In this work, a time domain reflectometry (TDR)-based method for estimating water content of GL was presented. More specifically, the proposed method relies on estimating the water content value of the GL structure from TDR-based dielectric permittivity measurements. To verify the suitability of this system, experimental tests were carried out on a GL sample. The obtained results anticipate the strong potential of the proposed method for practical applications in the Cultural Heritage diagnostics.

The advantage of employing TDR is that the necessary instrumentation is low cost, easy to use and portable. It should be mentioned that, in this work, a two-rod probe was used to better characterize the dielectric permittivity of GL. However, in practical diagnostics on Cultural Heritage structures, a noninvasive probe (such as a patch antenna) may be used to guarantee noninvasiveness of the system.

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