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Microwave reflectometric methodologies for water content estimation in stone-made Cultural Heritage materials

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ABSTRACT

In this work, the assessment of water content of building materials, to be used for moisture content monitoring of Cultural Heritage structures is addressed. To this purpose, different methods and probes were comparatively used to infer, noninvasively, the relationship between the water content of stone materials and the reflection properties at microwave frequency of the material. In particular, three types of probes were used: an open-ended coaxial probe; a patch resonator; and an open-ended waveguide. In addition, two different measurement instruments were compared; namely, a vector network analyzer and a time-domain reflectometer.

Experimental tests were carried out on two types of stone materials (*gentile* and *leccese* stone), which are typically found in Cultural Heritage structures of Southern Italy. For each of the considered measurement systems, the experimental results and the related uncertainty evaluation are reported.

1. Introduction

In the last few decades, the interest of the Scientific Community in monitoring and preserving Cultural Heritage objects has increased considerably [1–3], also due to the widespread awareness of the importance of preserving the invaluable cultural treasures. Indeed, the deterioration process of ancient structures is intrinsically related to the occurrence of particular microclimatic conditions inside monumental buildings/structures [4]; in particular, moisture is one of the major causes of decay of ancient building materials [5–7]. As a result, a wide range of measurement techniques is applied for detecting moisture presence [8]. Among these techniques, ground penetrating radar (GPR) [9], Electrical Resistivity Tomography (ERT) [10,11] and infrared thermography [12,13] have been extensively used for non-invasive moisture characterization of subsurfaces; however, the use of these techniques requires the operator to possess in-depth technical/scientific knowledge to obtain accurate results. Another possible strategy, particularly useful for investigating small material volumes and/or for a preliminary test on sample materials, is to infer the moisture content of the structure from microwave-based dielectric-permittivity measurements on the structure [14]. For example, in [15], time domain reflectometry (TDR) was employed in conjunction with a microstrip antenna (used as a sensing element) for noninvasive estimation of water content of granular materials.

Starting from these considerations, in this work, different methods and probes were comparatively used to infer, noninvasively, the relationship between the water content θ_v of stone materials and the reflection properties at microwave frequency of the material. In fact, microwave measurements allow noninvasive approach; they are easy for in-the-field use and they can be implemented in low cost versions.

For comparative purpose, in the present paper, three types of probes (an open-ended coaxial probe; a patch resonator; and an open-ended waveguide) and two different measurement instruments (a vector network analyzer and a time-domain reflectometer) were employed for the characterization of water content of stone materials.

The basic principle is to exploit the relation between water content and different electrical quantities measured through the proposed systems. In fact, all the considered methods rely on the fact that the presence of water, whose relative dielectric permittivity is in the order of 78 [16], increases the dielectric permittivity of the considered stone materials (which, in dry conditions, exhibit a relative dielectric permittivity of the order of 5–6).

Experimental tests were carried out on two types of materials, namely *gentile* and *leccese* stones: these materials are typically found in Cultural Heritage structures in Southern Italy and they are particularly affected by deterioration and decay phenomena

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[17]. Measurements were performed for increasing level of water content of the stone samples, and the empirical relationship between each considered level of water content and the corresponding measured quantity were derived. For each proposed measurement system, also the corresponding uncertainty was evaluated.

2. Material and methods

2.1. Types of stones

As aforementioned, two types of stones were considered: *gentile* stone and *leccese* stone.

The former is a calcarenitic ground stone, and its mineralogical composition is that of calcite [18]. Thanks to its good workability properties, this stone has been widely used in the building sector with several functions, for ashlars and load-bearing elements but also for coatings, decorations and statuary [19].

Leccese stone has been used, since ancient times, to construct private and public buildings, and as ornamental stone [20]. It has strong aptitude towards absorbing and retaining water; unfortunately, its porosity makes it prone to degradation [17].

For each type of stone, one sample was cut with the following dimensions:

- 3.0 cm × 7.7 cm × 10.1 cm for the *gentile* stone;
- 1.5 cm × 8.2 cm × 10.1 cm for the *leccese* stone.

2.2. Moistening procedure

For each stone sample, to obtain different water content conditions, the following moistening procedure was carried out:

(1) drying of the sample in a microwave oven;
(2) weighing of the dry sample, W_{dry};
(3) bath in deionized water until saturation (which, for the considered samples, took less than two hours);
(4) weighing of the sample, W_i;
(5) assessment of the volumetric water content, θ_v:

$$\theta_v = \frac{(W_i - W_{dry}) \cdot \rho_w}{V_{stone}} \times 100 \quad (1)$$

where V_{stone} is the volume of the stone sample, and $\rho_w \cong 0.996\ \mathrm{g/cm^3}$ is the density of water.
(6) measurement on the sample through each of the considered measurement methods;
(7) oven drying for a limited amount of time, in order to remove part of the moisture.

Steps 4–7 were repeated for each drying step, until complete dry up of the sample. In this way, it was possible to bring the samples to reference (known) water content values, θ_v, to be used for testing the measurement systems. Table 1 summarizes the reference values of θ_v for the two stone samples.

2.3. Measurement systems and probes

As mentioned in Section 1, three different probes were used:

- an open-ended coaxial (OEC) probe;
- a patch resonator (PR); and
- an open-ended waveguide (OEW).

The OEC probe is the High Temperature Probe available in the Keysight 85070E kit. This probe, which has a useful bandwidth of 20 GHz, is suitable for dielectric-permittivity measurements on liquids and on flat-surface solid materials [14]. The OEC probe requires a sample having dimensions, at least, 2 cm of diameter and $2/\sqrt{\varepsilon_r}$ cm thickness, where ε_r is the real part of the relative dielectric permittivity of the sample (both the stone samples satisfied this requirement). Fig. 1(a) shows a picture of the experimental setup. For each θ_v value, measurements on the stone samples were carried out through a vector network analyzer (VNA, model Agilent E8363C) to determine the $\varepsilon_r - \theta_v$ relationships.

In the measurement carried out through the PR probe, the goal was to assess the relationship between the resonant frequency of the patch resonator (f_r) and the water content of the sample. To this purpose, a patch resonator was designed and fabricated. As reported in [14], the used resonator has two mutually-isolated ports; however, only one port was used for the experiments (measurement of the changes in isolation between the two ports might be useful to characterize anisotropic materials, which was not the present case). As shown in Fig. 1(b), the reflection scattering parameter, $S_{11}(f)$, was measured by placing the resonator in contact with the stone samples, moistened at different reference values of θ_v. From the magnitude of the $S_{11}(f)$, the corresponding f_r value was inferred and associated to the θ_v reference value.

The $S_{11}(f)$ values were measured through the VNA and, for comparison, also through a time-domain reflectometer. The TDR measurements were carried out through the Campbell Scientific TDR100 reflectometer [21] to assess the possibility of employing TDR instrumentation, which is usually less expensive than VNAs and more suitable for in-the-field applications. A fast Fourier transform (FFT)-based algorithm was used to evaluate $S_{11}(f)$ from the TDR measurements [22].

Finally, dielectric-permittivity measurements were also carried out through an OEW probe. In this last case, the variation of the magnitude of the $S_{11}(f)$ (measured through a standard WR90 waveguide, connected to the VNA and averaged over the X-band frequency range) was related to the water content of the stone samples. It should be pointed out that, to preserve the noninvasiveness of the system, rather than embedding a stone sample in a portion of rectangular waveguide, a section of WR90 waveguide was used with one flange placed in contact with the materials samples, and the corresponding $S_{11}(f)$ was measured. Fig. 1(c) shows a picture of the setup.

Fig. 2 shows a sketch of one flange of the OEW probe and its dimensions $A = 22.86$ mm and $B = 10.16$ mm. The operating frequency range of the probe was 8.2–12.4 GHz.

3. Experimental results and uncertainty evaluation

3.1. Open-ended coaxial (OEC) probe

First, the measurements system was calibrated using the shorting block available with the kit, air and deionized water as standards.

Table 1
Reference water content levels for *gentile* and *leccese* stones.

Type	Reference θ_v(%) values						
leccese	21.6	18.4	15.2	12.0	8.0	4.0	0.0
gentile	10.7	8.1	6.4	4.3	2.6	0.0	–

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