

Complex Permittivity Measurements for Moisture and Salinity Characterization of Building Materials

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Abstract – In this paper, preliminary results of complex permittivity measurements of clay-brick material will be presented. The general aim of the experimental project is to correlate the measured permittivity of construction materials with the content of water and salt, in order to obtain information on the condition of ancient walls by using non-invasive techniques. The complex permittivity values reported in this paper have been obtained by waveguide measurements on samples of clay-brick dried and then immersed in demineralized water or in a saturated solution of demineralized water and NaCl.

I. INTRODUCTION

Moisture and salt concentration are two of the principal factors of degradation of the historical, artistic and cultural heritage objects [1]-[7].

Both factors have a great influence on complex permittivity of materials [8], so the knowledge of permittivity values of construction materials (clay-bricks, tufa-bricks, marble and wood) as function of frequency at fixed environmental conditions is useful in order to find the correlation with the moisture and salinity degree of walls and manufacts. The final aim is to investigate the possibility of using non-invasive microwave-based techniques and method to characterize the moisture and salt contents of stone materials.

In this paper, the values of complex permittivity of one selected stone material (red clay-brick) are presented, as function of moisture and salinity. The data were obtained by measuring the scattering parameters S_{11} , S_{21} , S_{12} and S_{22} from 3.95 to 5.85 GHz, through the transmission-line method in a WR187 waveguide system [9] connected to a Keysight Technologies PNA-L 5230A vector network analyzer (VNA). The S-parameters data were elaborated using the proprietary Keysight Technologies 85071E Materials Measurement Software [10].

II. MATERIALS

A. Type of stone

The samples used for measurements were worked from the same piece of red clay-brick, a material widely used to build walls since ancient times in all the Italian territory.

Typically, a clay-brick is a mixture of pure clay (hydrate silicate of alumina) and sand with a small quantities of lime and oxide of iron (responsible for the red colour) and smaller quantities of other elements (see Table 1).

Table 1. Normal components of clay-bricks.

Component		Weight percentage (%)
Silica (sand)	SiO ₂	50 to 60
Alumina (clay)	Al ₂ O ₃	20 to 30
Lime	CaO	2 to 5
Oxide of iron	Fe ₂ O ₃	≤ 7
Magnesia		< 1

The samples used for measurements were identified with the laboratory internal codes G006 and G007 and have the following dimensions ($l \times w \times h$):

- $(47.16 \times 22.00 \times 17.54)$ mm³ for G006 sample
- $(47.27 \times 22.04 \times 14.36)$ mm³ for G007 sample

B. Moistening procedure

In order to investigate the dependence of the material complex permittivity from water content and salinity, the two samples were first dried in a 1 m³ climatic chamber at 70 °C with no control in humidity for 18 hours, then cooled down to 30 °C and 25% relative humidity (RH)

for 1.5 hours. The dried sample weights (W_{dry}) are the following:

- $W_{dry} = (30.91 \pm 0.01)$ g for G006 sample
- $W_{dry} = (25.37 \pm 0.01)$ g for G007 sample

After the first measurement of complex permittivity, the G006 sample was immersed in a bath of 200 ml of deionized water while the G007 sample was immersed into a saturated solution of NaCl in deionized water (70 g of NaCl in 200 ml of water at laboratory temperature).

After water saturation of samples, new weighing and permittivity measurements were performed after drying the sample in a gravimetric oven set to 100 °C for limited amounts of time, in order to obtain a set of different water and salt water content conditions, until both samples were completely dried (the measured weight resulted near equal to W_{dry}).

The gravimetric moisture content θ_i were calculated as

$$\theta_i = \frac{W_i - W_{dry}}{W_{dry}} \times 100 \quad i = 1, \dots, n \quad (1)$$

where W_i is the weight measured at the i -th step of drying procedure (the index $i = 1$ refers to saturated wet sample).

During the weighing and the permittivity measurements the samples lost water by evaporation. The relative uncertainty was evaluated lower than the global experimental uncertainty of permittivity measurements (about 5%).

III. METHODS

The complex permittivity values were obtained by measuring the four scattering parameters S_{11} , S_{21} , S_{12} and S_{22} from the two ports of a standard WR187 waveguide line (shown in Fig. 1) in which the samples were inserted. The S-parameters data were acquired by a Keysight Technologies PNA-L 5230A VNA and elaborated by means of the proprietary Keysight Technologies 85071E Materials Measurement Software in order to obtain the complex permittivity (and permeability) values.

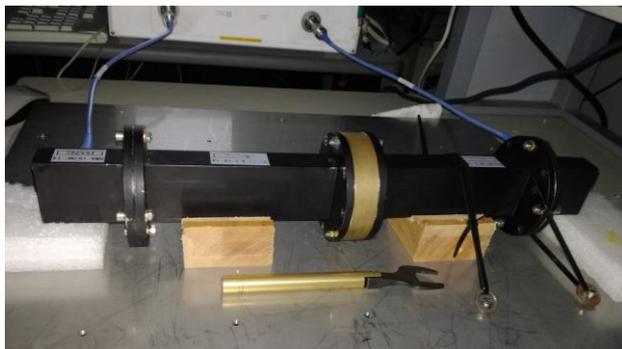


Fig. 1. WR187 waveguide measurement system

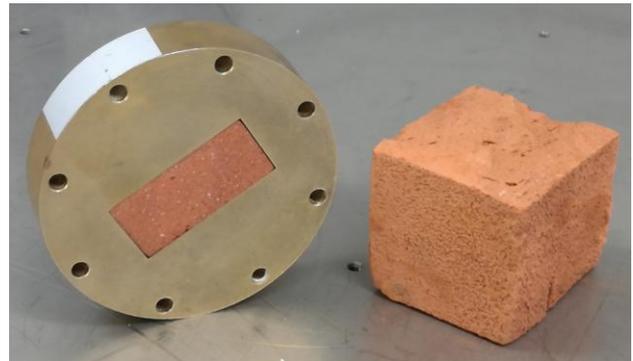


Fig. 2. View of the sample holder with G007 sample and a piece of the clay-brick from which the measured samples were milled.

The WR187 waveguide line is composed by two waveguide to N-type coax adapters, two waveguide straights (about 16 cm in length) and one sample holder made by a WR187 flange of 2.1 mm in length, which is the maximum thickness allowed for the samples.

The standard waveguide WR187 has internal dimensions of 1.872 × 0.872 inches (corresponding to about 47.55 × 2.15 mm) and is operating between 3.95 and 5.85 GHz (G-band). The cut-off frequency for the TE₁₀ mode is 3.153 GHz.

The Keysight Technologies 85071E Materials Measurement Software allows for calculation of complex permittivity values by using three different algorithms:

- (1) Nicolson-Ross-Weir [11]-[12]
- (2) NIST Precision [13]
- (3) Fast Transmission [14]

Some details of the algorithms are reported in [14].

The software requires the sample thickness to be specified and allows for adjusting the distance of the sample from the edge of the sample holder connected to the VNA port 1.

The software also allows user to set up correction for gaps between the sample and the sample holder along the shorter dimension of the waveguide. The dimension of the sample along the other dimension should fit the waveguide as accurately as possible.

At the beginning of measurement sessions, a TRL (Thru, Reflect, Line) calibration was performed, using the sample holder as Line element and a blank flange as Reflect element.

IV. EXPERIMENTAL RESULTS

The complex permittivity of both clay-brick samples were measured at different content of water and salt water. In the following figures, from Fig. 3 to Fig. 6, the real and imaginary part of ϵ are reported as function of frequency. Data are obtained by using the Fast Transmission algorithm.

The experimental data clearly show a pronounced increase of both real and imaginary part of complex permittivity with θ_i for all frequencies. This trend is best highlighted from the plot of the permittivity data as function of θ_i for the selected frequency of 5 GHz (Fig. 7 to Fig. 10).

As expected, the increase of the complex permittivity real part (related to the polarizability of molecules) is of the same order for both samples, the small difference being ascribable to the different thickness of samples. Instead, the imaginary part (related to the conductivity) shows a more pronounced increase for the sample G007 that was immersed in salt water.

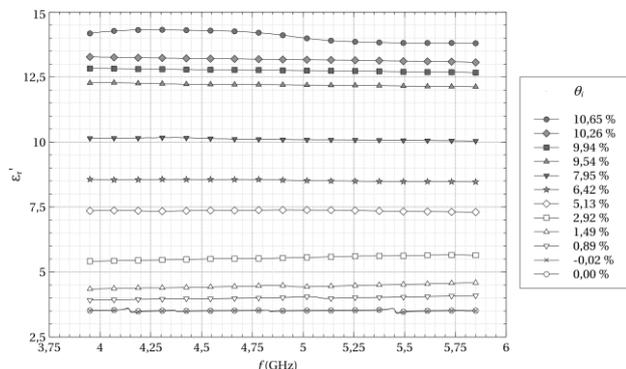


Fig. 3. G006 sample - ϵ_r' as function of frequency for different values of θ_i

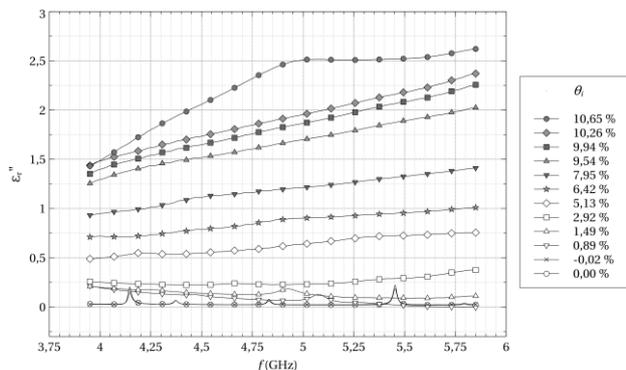


Fig. 4. G006 sample - ϵ_r'' as function of frequency for different values of θ_i

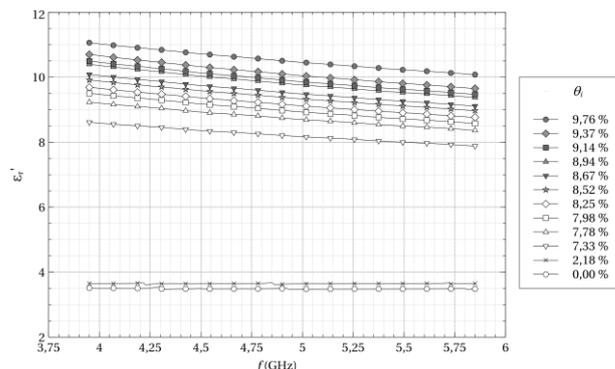


Fig. 5. G007 sample - ϵ_r' as function of frequency for different values of θ_i

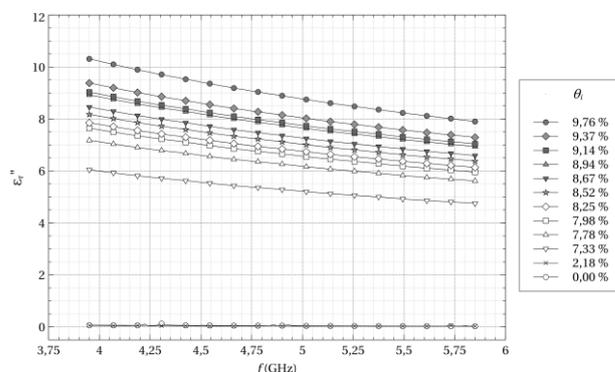


Fig. 6. G007 sample - ϵ_r'' as function of frequency for different values of θ_i

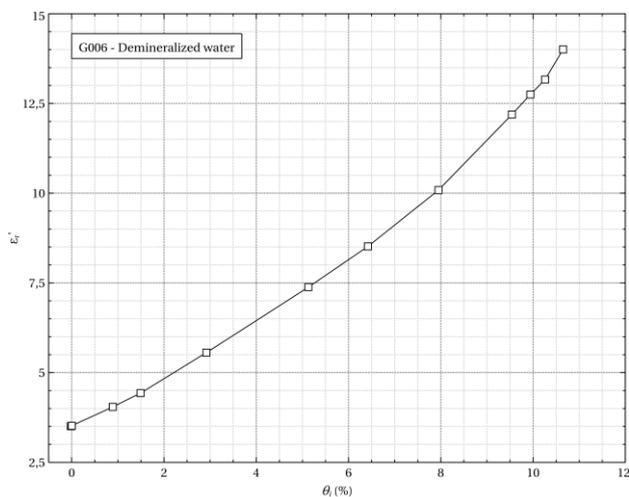


Fig. 7. G006 sample - ϵ_r' as function of θ_i @ 5 GHz

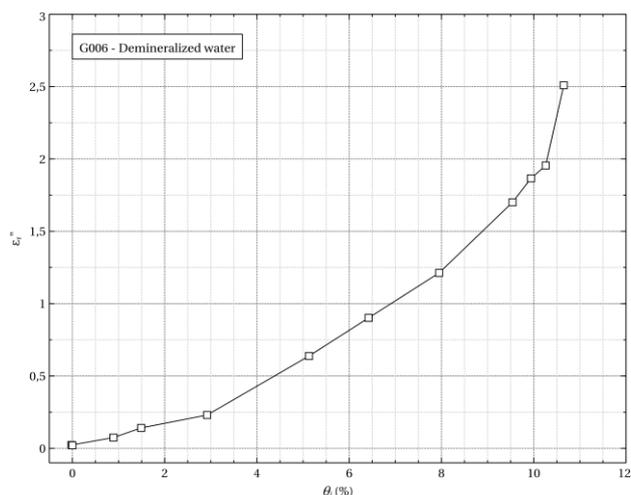


Fig. 8. G006 sample – ϵ_r'' as function of θ_i @ 5 GHz

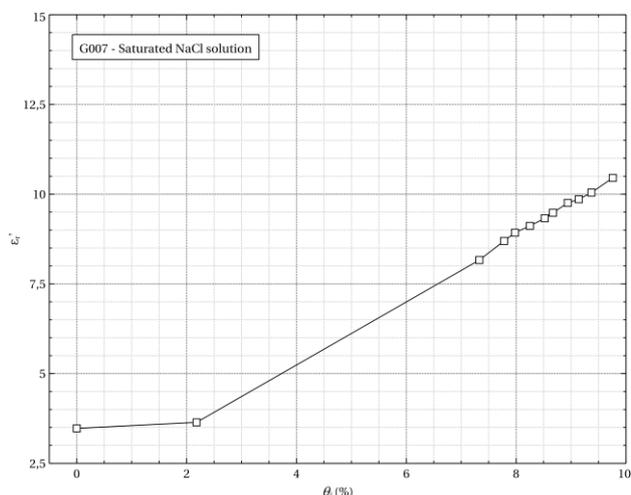


Fig. 9. G007 sample – ϵ_r' as function of θ_i @ 5 GHz

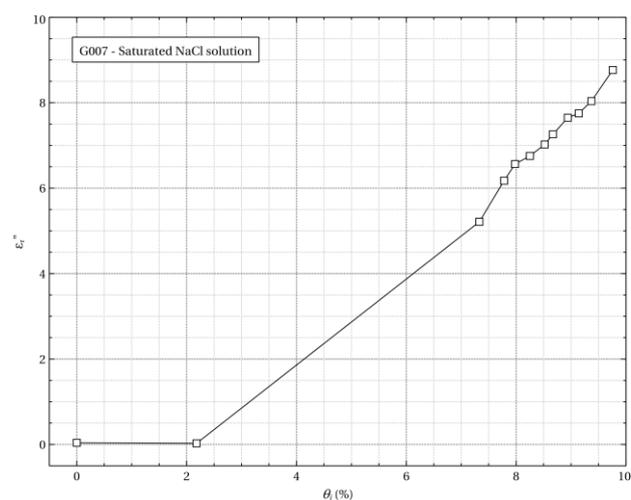


Fig. 10. G007 sample – ϵ_r'' as function of θ_i @ 5 GHz

Plotting the imaginary part ϵ_r'' vs. the real part ϵ_r' for both samples, it is possible to see how by measuring

complex permittivity it should be possible to discriminate the moisture and salinity of material under study. Even if the method seems promising, it is obviously necessary to extend the study to other materials and proceed to build calibration curves by independently measuring the moisture and salt content, with a more rigorous and systematic experimental procedure and more refined techniques.

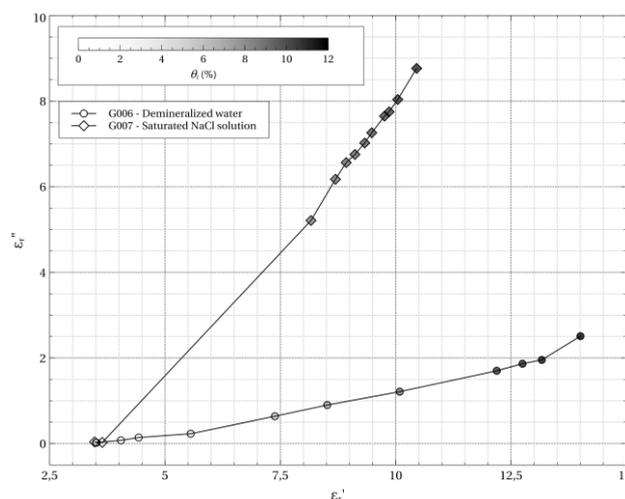


Fig. 11. ϵ_r'' vs ϵ_r' for both G006 and G007 samples at the frequency of 5 GHz. The different values of θ_i are highlighted by grayscale of markers.

V. CONCLUSIONS

In this paper, preliminary results of complex permittivity measurements, between 3.95 and 5.85 GHz, for different values of gravimetric moisture content, for clay-brick samples immersed in demineralized water and saturated NaCl solution, are presented.

Data show that it should be possible to discriminate the content of moisture and the presence of salt, from permittivity measurements, so demonstrating the suitability of microwave measurement techniques to detect moisture and salinity degrees of construction materials, and consequently their usefulness for the cultural heritage preservation.

REFERENCES

- [1] S. Carrà, "Introduzione" in "La diffusione in Italia delle metodologie scientifiche per lo studio e la conservazione delle opere d'arte", Acc. Naz. dei Lincei, Roma, 1989 (in Italian).
- [2] B. M. Smith, "Moisture Problems in Historical Masonry Walls", U.S. Dept. of the Interior, National Park Service, Preservation Assistance Division, 1984
- [3] E. Doehne, "Salt Weathering: A Selective Review", in Geological Society, London, Special Publications, Natural Stone, Weathering Phenomena, Conservation Strategies and Case Studies, Vol. 205,

- pp. 51-64, 1 January 2002
- [4] E. Doehne, C. Selwitz, and D. Carson, "The damage mechanism of sodium sulfate in porous stone", EC/GCI SALTeXPert Workshop, November 2002, Prague.
- [5] T. Lombardo, E. Doehne, S. Simon, "The response of NaCl and Umm Ishrin Sandstone to humidity cycling: mechanisms of salt weathering", 2004 Proceedings of the 10th International Congress on Deterioration and Conservation of Stone, Stockholm, June 27th -July 2nd p. 203-210.
- [6] D. Spennemann, "Urban salinity as a threat to cultural heritage places", Charles Sturt University, Johnstone Centre Albury, N.S.W 1997.
- [7] D. Spennemann, "The creeping disaster: dryland and urban salinity and its impact on heritage", Cultural Resource Management, 24(8), pp. 22-25, 2001.
- [8] E. Piuzzi et al., "A comparative assessment of microwave-based methods for moisture content characterization in stone materials", Measurement, in press. Available on line 22 April 2016.
- [9] J. Krupka, "Review article: Frequency domain complex permittivity measurements at microwave frequencies", Measurement Science and Technology, Volume 17, Issue 6, pp. R55-R70, 2006.
- [10] Keysight Technologies, "Measuring Dielectric Properties Using Keysight's Materials Measurement Solutions", Document 5991-2171EN, June 21, 2016.
- [11] A. M. Nicolson and G. F. Ross, "Measurement of the intrinsic properties of materials by time domain techniques," IEEE Trans. Instrum. Meas., vol. IM-19(4), pp. 377-382, 1970.
- [12] W. W. Weir, "Automatic measurement of complex dielectric constant and permeability at microwave frequencies," Proc. IEEE, vol. 62 pp.33-36, Jan 1974.
- [13] J. Baker-Jarvis et al., "Transmission/Reflection and Short-Circuit Line Methods for Measuring Permittivity and Permeability", NIST Technical Note 1355-R, December 1993.
- [14] Keysight Technologies, "Models in the 85071E Materials Measurement Software", Available at: <http://na.support.keysight.com/materials/docs/85071Emodels.pdf>