PAPER • **OPEN ACCESS**

Real-time detection of salts content in waterlogged archaeological wood by evanescent field dielectrometry (EFD): preliminary results

To cite this article: S. Longo *et al* 2022 *J. Phys.: Conf. Ser.* **2204** 012058

Real-time detection of salts content in waterlogged archaeological wood by evanescent field dielectrometry (EFD): preliminary results

S. Longo[1], E. Fazio[2], B. Pizzo[3], C. Riminesi[1]

[1]CNR-ISPC, Via Madonna del Piano 10, 50019 Sesto Fiorentino (FI), Italy.
[2]Department of Mathematical and Computational Sciences, Physics Science and Earth Sciences (MIFT), University of Messina, 98166 Messina (ME), Italy.
[3]CNR-IBE, Via Madonna del Piano 10, 50019 Sesto Fiorentino (FI), Italy.

Corresponding author: Sveva Longo (email: sveva.longo@ispc.cnr.it)

Abstract

Monitoring moisture content and detecting the presence of soluble salts in archaeological waterlogged wood remains is a key issue for their conservation. Evanescent field dielectrometry (EFD), originally applied to frescoes and mural painting investigation is proposed as a novel tool for the real-time detection of soluble salts inside waterlogged wood samples. Preliminary measurements on archaeological samples from Denmark were performed to provide the basis for on-site monitoring of the rinse process required before consolidation treatment.

Keywords: Microwaves dielectrometry; archaeological waterlogged wood; salts detection

1. Introduction

Moisture and salts are some of the main factors that cause deterioration in cultural heritage artefacts made of porous materials [1]. It is essential to determine moisture content (MC) and salinity index (SI) to understand the state of preservation and choose the best conservation treatment. In this context, different in situ non-invasive techniques for water - and salt - transfer dynamics monitoring has been developed in the last years such as thermography [2], electrical resistance measuring [3, 4] and Nuclear Magnetic Resonance (NMR) [5, 6]. However, they were applied mostly on wall painting or porous stone materials and buildings (concrete and reinforced concrete), rarely for monitoring moisture content in organic porous materials such as wood [7, 8].

In this regard, the archaeologically excavated wood generally has a very high moisture content (400-800%) thus proving to be an ideal investigative subject for water-transfer studies. In particular, waterlogged archaeological wood seems to be very well preserved if left immersed. However, its level of decay appears once extracted from the water, when profound shrinkages and even collapse may occur, making evident the need for conservation. The conservation of waterlogged wood is a two-fold process that involves the incorporation of material (e.g., PEG- or sugar-bulking treatments) into the wood. This material consolidates and confers mechanical strength to the wood while the water is being removed by methods preventing any shrinkage or distortion of the wood (e.g., solvent- or freeze-drying) [9]. Another important aspect is the necessity to remove soluble salts coming from marine environments. Depending on their composition, these can oxidise when exposed to air, and even leading to subsequent hydrolysis of cellulose or the same consolidating agent [10]. Moreover, if they are not removed before treatment, they will cause the appearance of efflorescence on the conserved wood surface [11]. Therefore, studies on conservation products efficiency and salts detection are desirable to better preserve the wooden remains.

For this purpose, it is possible to investigate by evanescent field dielectrometry (EFD) the presence of salts before the treatment process and quantify chemical agents that replace the water in the wood. In this regard, the system was recently applied for measuring moisture and salts inside wall paintings [12] and concrete [13].

Several studies are focused on dielectric properties of wood measured by microwave frequencies, but they are limited to the moisture content determination [14, 15, 16]. In the case of archaeological wood samples MC measurements, are not so crucial because they are conserved underwater and MC values are constant because of the full imbibition of wood cells. On the contrary by SUSI technique (it is the Italian acronym for integrated instrument for measuring moisture and salts content), it is possible to determine the salts content (salinity index, SI). SI is a parameter introduced recently in the field of microwaves dielectric measurements [12]. By this methodology it is possible to assess the quantity of salts inside waterlogged wood microstructure which is the main deterioration cause [17].

In this paper, we show, for the first time to our knowledge, that the SUSI technique can be a useful tool for monitoring salt content by salinity index equation in archaeological waterlogged wood, without any damage to the samples and in a very quick time (only 30 seconds for each measurement).

2. Experimental

2.1 Materials

Four samples (x68, x73, x88 and x430) of archaeological waterlogged oaks planks found in 2018 in Køge (Denmark), during the excavation of an XVI century wreck of an ancient ship, were investigated (Fig. 1). Dielectrometry results were compared with data acquired on two reference contemporary oak samples (6x6x1,25 cm*, Quercus sp*) boiled in water to improve the imbibition process. One sample was imbibed with plain water (Contemporary) and the other one with a solution of water and NaCl (35g/L) to simulate seawater (Contemporary + NaCl).
Measurements were carried out on the radial section for modern and archaeological samples because it was the most extended surface and SUSI technique requires a contact surface with a diameter of 2.5-3 cm.

Figure 1 On the left: some remains during the archaeological excavation; on the right: one of the archaeological samples analysed.

2.2 The Evanescent Field Dielectrometry technique

The Evanescent Field Dielectrometry (EFD) system operates in the microwaves range (1 – 1.5 GHz) and it consists of a resonant probe and a network analyser that collect the probe frequency response. The relevant electrical parameters, resonance frequency (Δf) and the quality factor (Q) of the open-ended resonant cavity can be extracted from the registered data [18]. Resonance properties are directly related to the permittivity of the material analysing the dielectric constant ε' and losses ε''. A modification of these two parameters in the investigated materials will cause a modification of Δf and Q. In this way, using the electric field-material interaction electromagnetic model [19], the moisture content (MC) and salinity (SI) are in real-time calculated from the measured values of Δf and Q by the relations:

$$MC = \alpha \Delta f + \beta \quad (1)$$

$$SI = \frac{f_r f_0}{2\Delta f^2} \Delta\left(\frac{1}{Q}\right) \quad (2)$$

where α and β are constant obtained by fitting gravimetric data of samples with known porosity, while fr is the resonance frequency measured on the material, $f0$ is that in air and $\Delta(Q^{-1})$ is the variation of the reciprocal of Q among air and material conditions.

The investigation probe area is approximately a half-sphere of 2 cm radius and depends on the aperture of the resonant cavity towards the material [20].

Measurements on archaeological samples were carried out following a map with spots of 2x2 cm (Fig. 2). For each sample, 9 measurements points were acquired.

On contemporary reference specimens, data acquisition was performed at the centre of the sample. Measurements were taken after 2h, 24h, 96h and 120h of the sample's sinking process.

MC and SI data were elaborated with Origin software.

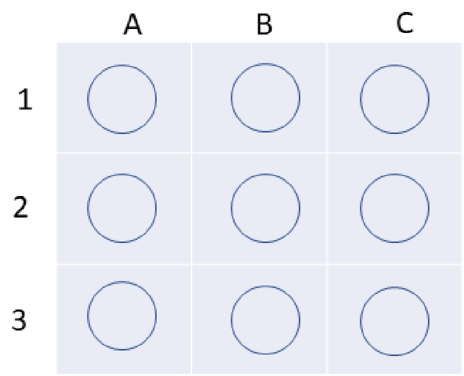

Figure 2 Measurements schema on waterlogged archaeological samples. In each spot, one data acquisition was performed.

2.3 Conventional conductivity meters evaluation

Conductivity tests with a real-time datalogger meter were performed. The measure was made at room temperature (25° C) by a PCE with TDS probe.

3. Results and discussion

SUSI measurements on two contemporary imbibed specimens and four archaeological waterlogged samples were performed. SI and MC data from reference measurements were compared with the ones obtained on archaeological samples.

Results reported in fig. 3a, show that archaeological samples x68, x73, x88 and x430 average values of SI index have similar range-values (SI ≈ 1 ± 0.25) to the sample imbibed in plain water (Contemporary) after 120h (for this reason they were aligned with the point related to this time in fig. 3). On the contrary, the measurements performed on the salty sample (Contemporary + NaCl) shows SI values between 2.32 and 2.61. For mortar samples, a value of 1 means that the conductivity of the electrolyte is approximately equal to 0.4 $S \cdot m^{-1}$ (plain water), while a value of 9 corresponds to a conductivity of the electrolyte of 10 $S \cdot m^{-1}$. In our case, archaeological waterlogged wood samples are not more affected by salts (SI ≈ 1 ± 0.25). The SI is assumed only as an estimation of the amount of salts inside the material, because we are not able to distinguish between salts species from this kind of measurements.

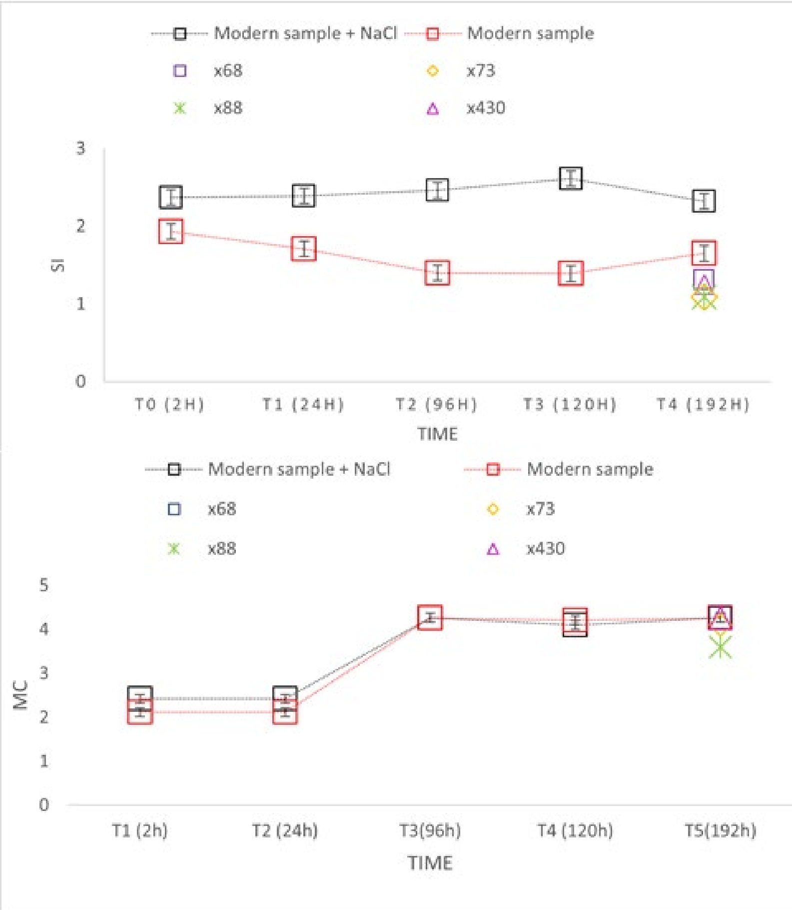

Figure 3 SI and MC values of contemporary samples compared with archaeological waterlogged wood samples.

As regards moisture content values of the contemporary specimens, between 96h and 120h, they agree with the ones from archaeological wood (MC ≈ 4.27) (Fig. 3b). X88 sample has lower values, but it is probably correlated to wood microstructure, which it is slightly different for each sample. Moreover, MC results show that contemporary samples reached full imbibition after 96h. MC values between 4.21 to 4.27 are obtained carrying out all the measurements following the schema in fig. 2.

The SI measurements by SUSI instrument were compared with the conductivity measurements performed on water where samples were stored. This method is usually utilized by restores to evaluate the residual presence of salts after washing.

Generally pure water conductivity is ≈ 0.05 µS/cm; tap water between ≈0.5 to 0.8 mS/cm and seawater ≈ 53 mS/cm. Our results are: modern samples water respectively ≈ 55 mS/cm for salty water and ≈ 0.2

mS/cm for freshwater; instead archaeological samples water has values of ≈ 0.13 mS/cm for x88, ≈0.13 mS/cm for x430, ≈0.2 mS/cm for x68 and ≈0.14 mS/cm for x73. Values of salty modern sample water agree with seawater values, same results are between archaeological samples water, modern sample water and tap water.

4. Conclusion

In this preliminary work, the main goal was to validate SUSI technique as a possible novel methodology for salts detection inside waterlogged wood.
This issue was overcome and shows how the evanescent field dielectrometry is a valuable and promising diagnostic tool for monitoring waterlogged wood microstructure during the conservation process. Conductivity measurements on water where samples are stored were conducted to confirmed that SI values obtained by SUSI are real.
In conclusion, the resonant approach guarantees an excellent sensitivity on the measurement of the moisture content and allows the detection of the presence of soluble salts simultaneously and independently. The method is going to be refined and applied to more prospective studies.

Acknowledgements

Dr Aoife Daly of the University of Copenhagen for providing archaeological samples.

References

[1] Siegesmund S, Snethlage R 2011 *Stone in Architecture: Properties, Durability*, Editor Springer Heidelberg Dordrecht London New York
[2] Grinzato E, Cadelano G and Bison P 2009 *J of Modern Optics* 57 (18) 1770-1778
[3] Valero L R, Sasso V F, Vicioso E P 2019 *Case Studie sin Construction Materials 10* 1-13
[4] Consoli G, Mobili A, Tittarelli F, Revel G M, Chiarotti P 2020 *Appl. Sci* 10 (9152) 1-42
[5] Oligschlager D, Waldow S, Haber A, Zia W and Blumich B 2015 *Magnetic Resonance in Chemistry* 53 (1) 48-57
[6] Proietti N, Calicchia P, Colao F, De Simone S, Di Tullio V, Luvidi L, Prestileo F, Romani M, Tatì A 2021 *Minerals* 11(4) 1-23
[7] Longo S, Capuani S, Corsaro C, Fazio E 2020 *IOP Conf. Ser.: Mater. Sci. Eng.* **777** 012004
[8] Capuani S, Stagno V, Missori M, Sadori L, Longo S 2020 *Magnetic Resonance in Chemistry* 59 (9) 860-869
[9] Antonelli F, Galotta G, Sidoti G, Zikeli F, Nisi R, Pietraggi Davidde B, Romagnoli M 2020 *Frontiers in* Chemistry 32 (8) 1-12
[10] High K E and Penkman K E H 2020 *Heritage Science* 83 (8) 1-33
[11] Almkvist G and Persson I 2011 *New J. Chem.* 32 1491-1502
[12] Olmi R, Bini M, Ignesti A, Priori S, Riminesi C, Felici A 2006 *Measurement Science and Technology* 17 (8) 2281-2288
[13] Riminesi C, Marie-Victoire EM, Bouichou M, Olmi R 2017 *Measurement Science and Technology* 28 (1), 014002
[14] Daian, G., Taube, A., Birnboim, A., Daian, M. and Shramkov, Y., 2006 *Wood science and technology*, 40(3), pp.237-246;
[15] Daian, G., Taube, A., Birnboim, A., Shramkov, Y. and Daian, M., 2005 *Wood Science and Technology*, 39(3), pp.215-223;

[16] Olmi, R., Bini, M., Ignesti, A. and Riminesi, C., 2000 *Journal of Microwave Power and Electromagnetic Energy*, 35(3), pp.135-143 ().
[17] Boichou M, Marie-Victoire EM, Jourdan H, Thauvin B, Queguiner R, Olmi R, Riminesi C 2018 *Journal of Cultural Heritage* 34 237-246
[18] Olmi R, Nesti R, Pelosi G, Riminesi C 2004 *Journal of Electromagnetic Waves and Applications* 18 (2) 217-232
[19] Olmi R, Riminesi C 2008 *Nuovo Cimento della Società Italiana di Fisica C* 31 (3) 389-402