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EVALUATION OF CONSERVATION TREATMENTS FOR ARCHAEOLOGICAL WATERLOGGED WOODEN ARTEFACTS

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ABSTRACT
In favourable conditions of low temperature and low oxygen concentration, waterlogged wooden artefacts, such as shipwrecks, can survive underwater in relative good conditions. Nevertheless, as a result of the action of water and anaerobic bacteria on the cellulose and hemicellulose of the cell walls, waterlogged wood is very fragile and its consolidation is necessary. These conservation treatments are very often expensive and technically difficult, as they involve the replacement of the waterlogging water with the consolidants, filling the cell walls and all cavities in order to prevent stresses during the drying. Since the effectiveness of the treatment strongly depends on its distribution, the penetration depth and the degree of cavities-filling have been evaluated by micro x-ray tomography (μ-CT). Both laboratory and Synchrotron Radiation-based μ-CT experiments have been performed on different samples coming from findings excavated in the San Rossore archaeological site (Pisa, Italy), in order to study in detail how the different application parameters can influence the manner in which the conservation products fill the cavities of the wooden structure (i.e. complete or partial volume filling, complete or partial impregnation of the wooden artefact).

INTRODUCTION
In conditions of low temperature and low oxygen concentration, wood artefacts can survive for centuries below the water table in terrestrial environments or underwater and can surprisingly show a good appearance when brought to light [1]. Nevertheless, the cellulose and hemicellulose of wooden cell walls may be degraded by the action of biota and replaced by water [2]. In fact, waterlogged wooden artefacts are characterised by high water contents, which can reach 1000 % of the wood weight, depending on the preservation state of the wood. Therefore, wood becomes a spongy and weak material and needs appropriate conservation treatments. The recovery and the conservation of waterlogged wood represent an extremely arduous problem, which is still far to be completely solved [3, 4]. The conservation treatments very often involve impregnation with substances which, replacing the imbibition water, fill the cell micro-cavities and/or cell *lumina*, in order to prevent drying stresses. Moreover, the particular requirements of the substances and methodologies which can be used to reach this goal make the choice of the restoration treatments of archaeological waterlogged wooden artefacts a very complicated issue. Different polymers, both natural and synthetic, have been experimented as consolidants.

The evaluation of the conservation methodologies is generally done by measuring physical parameters, such as the weight of the impregnation substance in the wood after treatment, the acquired dimensional stability and the water absorption (or desorption) in different hygrometric conditions [5, 6]. The penetration depth and distribution of the impregnation products into wood structures are also relevant parameters in the assessment of the efficacy of the treatments [7]. These latter are normally estimated by means of optical and electron microscopy, preparing cross sections of the wooden material at different depths from the external surface of the object or sample.

An alternative to this destructive method, which can be considerably influenced by the sample preparation, can be provided by X-ray computed tomography (CT), which has been successfully applied for the characterization of waterlogged archaeological wood [8], and for the study of protective/consolidants in porous media, such as rocks and woods [9, 10].

CT is based on the acquisition of a set of radiographs recorded at different rotation angles, so that the horizontal section of the scanned object can be reconstructed by means of an appropriate mathematical algorithm without physical cutting or complicate sample preparation.

The penetration of conservation substances can be thus investigated in a non-destructive way and, furthermore, the samples can be analyzed by other techniques. Moreover, recent desktop instruments (µ-CT) can achieve (sub)micro-resolution, allowing to visualize and study in detail the three dimensional structures of the specimens.

On the other hand, in the case of wooden samples, this technique has some limitations:

- The resolution cannot achieve the standards of the electron and optical microscopy
- The contrast between materials of similar density and composition (e.g. wood and organic conservation products), being normally low, doesn't allow an easy visual distinction between the two phases.

Whereas the resolution is limited by experimental factors, mainly instrumental performances and sample dimensions, the contrast can be improved performing computed tomography using Synchrotron Radiation (SR-CT) as X-ray source. If compared to conventional X-ray tubes, Synchrotron produces a monochromatic X-ray beam (band width up to 10^{-4}) in a wide range of energy (from few to more than hundred keV) which allows obtaining higher quality and improved contrast in the reconstructed CT images.

In the present work µ-CT and SR-CT have been applied with the aim to:

- Evaluate different conservation treatments for waterlogged wooden artefact by observing their penetration and distribution;
- Compare performance and results obtained with µ-CT system and SR-CT

MATERIALS AND METHODS

Two types of samples have been analysed:

- Specimens of *Quercus caducifolia*, coming from San Rossore archaeological site (Pisa, Italy), impregnated with a mixture of polyethylene glycol with molecular weight of 1500 amu (PEG 1500) with concentration of 20% in weight and polypropylene glycol with molecular weight of 425 amu (PPG 425).
- Specimens of the same wood species, coming from the archaeological site of Marseille (France), impregnated with isoeugenol, subsequently polymerized *in situ*.

Cylinders of suitable size for µ-CT and SR-CT analyses, i.e. 9 mm and 1.5 mm in diameter respectively, have been cut from the cuboid samples of dimensions 50x50x30 mm For laboratory tomography scans a SkyScan 1172 system has been used. This instrument mounts an X-ray tube (tungsten target) and a CCD camera 4000x2048 pixel. The radiographs have been acquired at a 4.5 µm resolution, using a rotation step of 0.4°. The voltage and current of the X-ray source have been kept at 60 kV and 170 µA respectively, applying an aluminium filter (thickness = 0.25 mm) in order to reduce beam hardening artefacts and to improve the signal to noise ratio. The tomographic cross sections have been reconstructed by means of NRecon software package, based on a modified Feldkamp algorithm.

The tomography experiments with Synchrotron Radiation have been carried out at ID 19, one of the beamlines dedicated to X-ray imaging of ESRF (European Synchrotron Radiation Facility) in Grenoble (F). At this end station it is possible to use a monochromatic beam, obtained either with double Si (111) crystals (band width = 10^{-4}) or a multilayer mirror (band width = 10^{-2}), in a wide range of photon energies (6 – 120 keV) which contribute to considerably improve the contrast between the different phases present in the sample.

A FRELON (Fast Readout Low Noise) camera 2048 x 2048 pixel, developed by ESRF, has been used as detector, allowing at the same time fast acquisition of radiographs, good image quality and high magnification thanks to the optics system.

The samples have been scanned at 19 keV with a 0.12° rotation step and resolution of 700 nm.

RESULTS AND DISCUSSION

The cross section of the sample impregnated with PEG+PPG (fig.1), obtained by µ-CT data, shows that the major part of the vessels has been filled by the mixture until 15 mm from the surface. In the same image we can easily notice the presence of a high X-ray attenuating material, identified, by energy dispersive X-ray analysis (EDX), as iron oxide.

The contrast between PEG+PPG and the wooden structure is low, so that it is difficult to map exactly the impregnating products and to distinguish them from the material. Moreover at 4.5 µm resolution only vessels with *lumina* larger than 20 µm are clearly visible, while the wooden micro structure, such as *lumina* of tracheids cannot be investigated.

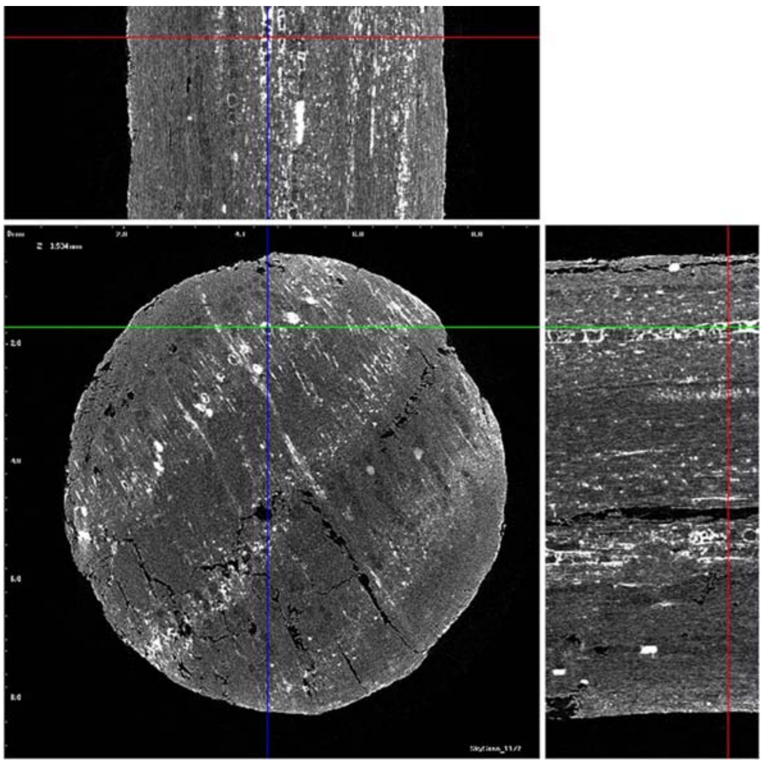

Figure 1 – Cross section of a sample of Quercus caducifolia *impregnated with a mixture of PEG 1500 al 20% in PPG 425, obtained by µ-CT*

The sample impregnated with isoeugenol shows a different impregnating product distribution in the wooden structure if compared to PEG+PPG: the polyisoeugenol fills the vessels close to the surface (fig. 2a), while already at the depth of 250-300 μm the polymer is not present or uniformly distributed in very thin films, coating the cell walls (fig. 2b).

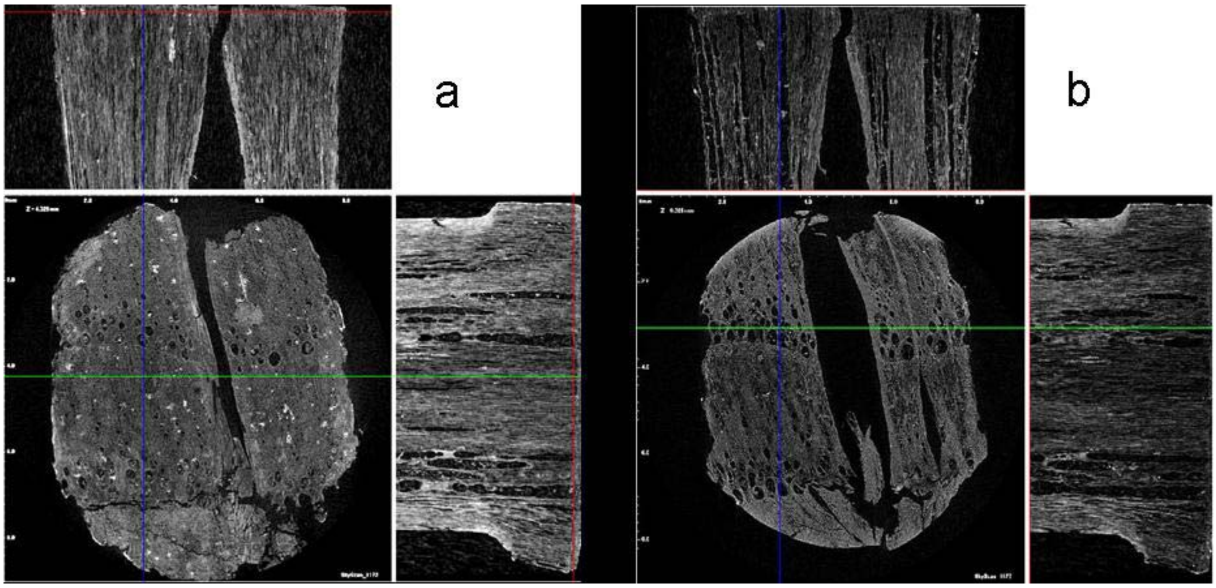

Figure 2 – Cross sections of a sample of Quercus caducifoglia impregnated with isoeugenol polymerized in situ, obtained by μ-CT: (a) surface; (b) 4.5 mm from the surface

The images reconstructed from Synchrotron Radiation tomography data (fig. 3), thanks to the high resolution (700 nm), allow evaluating the penetration of PEG + PPG and observing the diversified state of preservation of the wood cells. The glycol mixture fills almost all the vessels (fig. 3), but in the enlargement, few empty vessels, together with the iron oxide deposits mentioned above, can be detected.

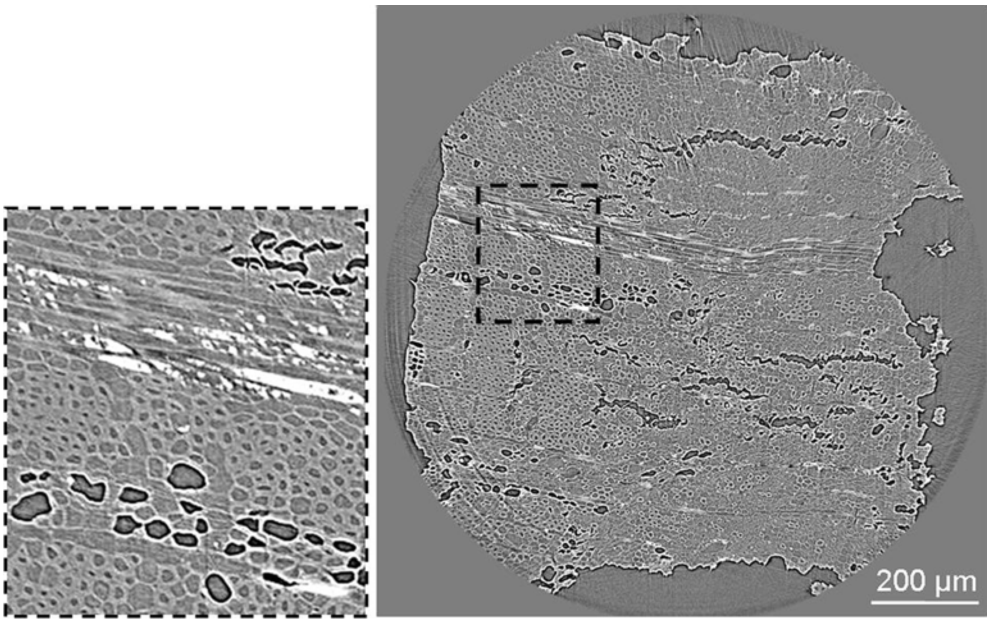

Figure 3 - Cross section of a sample of Quercus caducifolia *impregnated with a mixture of PEG 1500 al 20% in PPG 425, obtained by SR-CT.*

The three dimensional rendering of the sample impregnated with isoeugenol (fig. 4) scanned by SR-CT, reveals a different behaviour, if compared to PEG+PPG. In fact, polyisoeugenol does not block the lumen of vessels, being probably distributed on the cell walls. Despite the high resolution (700 nm), the mapping of this polymer is very difficult because of the limited contrast and the uniform distribution of the consolidants in very thin films.

Fig. 4 also shows how three dimensional renderings can be useful and representative of the anatomic wooden structure. Moreover, the high quality and resolution of the tomographic images allow observing in high detail the morphological features of the samples, highlighting the relevant advantage of this technique if compared to microscopy.

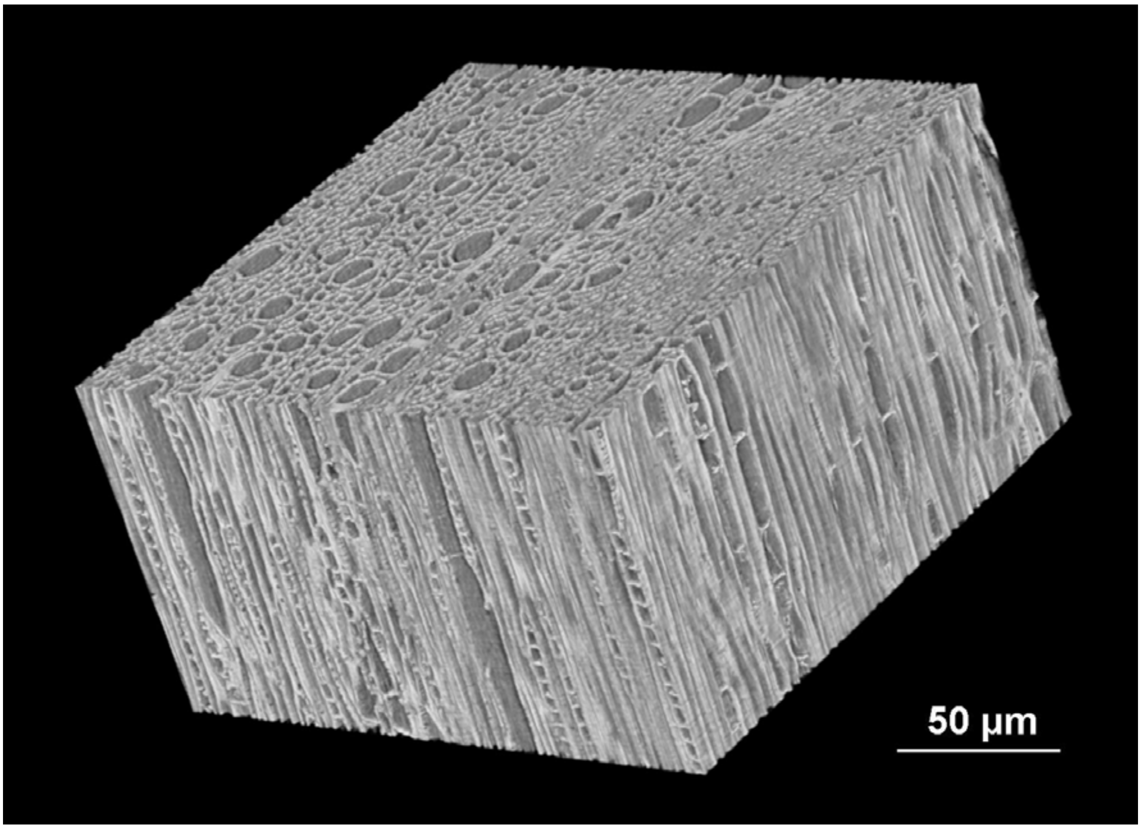

Figure 4 – Three dimensional rendering of a sample of Quercus caducifolia *impregnated with isoeugenol polymerized, obtained by SR-CT.*

CONCLUSIONS

Computed tomography proved to be a powerful tool to study the penetration and distribution of polymers used in conservation treatments of waterlogged wooden artefacts. This technique does not require particular sample preparation and allows to investigate the three dimensional structure of the wood in a non-destructive way, preserving precious specimens which can be analysed by other methods.

µ-CT systems allow to visualize the conservation products into samples, even if the limited contrast between wood and polymers and the resolution adopted in this research make the mapping of PEG+PPG and polyisoeugenol difficult.

Synchrotron Radiation tomography, using a monochromatic X-ray source, can be successfully applied in order to improve resolution and contrast and to study more in details the conservation treatments. The results show that PEG+PPG mixture efficiently fills the vessels, while isoeugenol is preferentially distributed in thin film in the wooden structure.

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