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SYNCHROTRON RADIATION MICRO-TOMOGRAPHY: A NON-INVASIVE TOOL FOR THE CHARACTERIZATION OF ARCHAEOLOGICAL WOOD

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Abstract

Archaeological wood, and waterlogged archaeological wood in particular, are almost always characterised by a modification in the original properties and structure of the wood. Degradation agents can be of different origins (Biological, Chemical, Physical) and they are able to produce a large variability in the degradation of individual pieces, requiring their characterization for a proper conservation. X-ray computed Micro-Tomography (X-ray μCT) is a powerful tool, which provides three-dimensional information on the morphological characteristics of materials. Contrary to other analysis, it gives a full insight of the inner structure of the material, in a totally non-destructive way and without requiring a specific sample preparation. In this study the measurements were performed on archaeological and recent wood samples; its main aims were:

- to obtain two- and three- dimensional characterization of archaeological wood in order to evaluate if X-ray μCT can be a powerful tool for the investigation of anatomical features of wood;
- to obtain a quantitative description of the level of degradation by measuring the linear attenuation coefficient and material density.

The experiments were carried out at the SYRMEP beam line of the Elettra synchrotron light source at Trieste (Italy) and at TomoLab, a new X ray micro-focus generator located at the same Elettra site.

1. Introduction

Archaeological Wood is wood that has been excavated from different types of archaeological sites, including marine and submerged environments. In these last cases the time during which wood has been waterlogged is considerably long, often over one thousand years. The characteristics of these finds might be rather different from those of recent wood, as a result of the activity of different deterioration agents (biological, chemical, physical, mechanical, etc.). Furthermore the different conservation sites (type of water, soil composition, etc.) represent a further element of complexity that may affect wood degradation. In this context very important are methods and techniques that can be applied for the characterization of this particular type of wood.

X-ray computed Micro-Tomography (X-ray μCT) is a powerful tool that provides three-dimensional information on the morphological characteristics of materials, giving a full insight of their inner structure in a non-destructive way, without requiring a specific sample preparation. Furthermore, by means of suitable algorithms, starting from a set of planar images (projections) it is possible to reconstruct a volumetric map of X-ray linear attenuation coefficient.

In this study the X-ray μCT has been applied to the characterization of archaeological wood with the specific aims of evaluating its potentiality as a tool for 1) the investigation of anatomical structure of wood (i.e. determination of wooden specie), and 2) the assessment of wood degradation trough the measurement of residual density. The experiments were carried out at the SYRMEP beamline of the Elettra synchrotron light source at Trieste (Italy) and at TomoLab, a new X ray micro-focus generator located at the same Elettra site.

2. Material and methods

2.1 X-ray micro-tomography with synchrotron radiation light

The main characteristics of synchrotron radiation are the continuous spectrum, extending from infrared to hard X-rays, the high spatial coherence, and the intensity of photons emitted in a small solid angle. This makes it possible to work with short exposure times, allows for tuning the photon energy as a function of the sample characteristics, and, in some cases, makes it possible to apply digital subtraction techniques. Phase-sensitive imaging techniques, using highly coherent, hard X-rays from 3rd-generation synchrotron sources, have the additional advantage of allowing the imaging of samples with very low absorption contrast, such as light-element composites, and biological systems [Snigirev and others 1995] [Cloetens and others 1996]. The X-MCT investigations were carried out using the SYRMEP beamline [Arfelli and others 1995] of the Elettra Laboratory in Trieste, Italy.

The samples, mounted on a rotation stage, were illuminated by monochromatic radiation (E = 15 KeV). The distance between the sample and the detector was less than 50mm when the absorbing images were collected, and at 300 mm. for the image in phase contrast regime.

For each tomographic set 900 projections of the sample were acquired for equally spaced rotation angles and measurement times of 1 s, over a total rotation of 180 degrees.

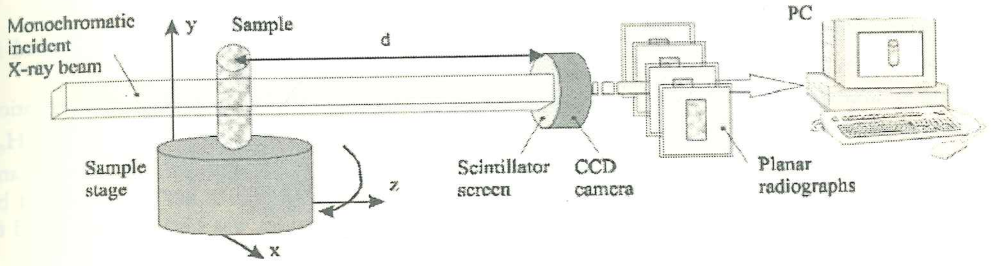

Fig. 1. Schematic view of the experimental setup used for the X-MCT at SYRMEP beamline of ELETTRA.

The detector used was a cooled charge-coupled device (CCD) camera coupled to a Gadolinium Oxysulphide scintillator (Photonic Science Ltd, Boulder, Colo., U.S.A.) placed on a straight fibre optic coupler. High dynamic range and low noise is achieved through 16-bit digitization and deep cooling, whereas the 4008 × 2672 pixels CCD is characterized by a pixel size of 9 μm.

The tomographic projections have been processed using a set of routines [Montanari 2003]. These routines allow the reconstruction of single slices; data can then be saved in several formats and subsequently loaded by other applications for visualization and analysis. The computer code is written in the interactive data language (IDL).

The 2D reconstruction is performed using a filtered back projection algorithm [Herman 1980], for each projection an intensity map is recorded in the xy detector plane; then each intensity map is back-projected along the normal to the projection itself. Projections are submitted to filtering procedures to eliminate noise and artefacts and, finally, the intensities are added for all the projections.

Then, the reconstructed slices can be visualized as stacks of 2D images, or 3D views of the sample can be obtained by volume rendering procedures. Rendering process was performed using the ImageJ software (version 1.37, Natl. Inst. of Health, Bethseda, Md., U.S.A. ImageJ software is in the public domain and is available from http://rsb.info.nih.gov/ij or ftp://rsbweb.nih.gov/pub/imagej) and Volume Graphics VGStudio 1.2.1 (commercial software).

2.2. Density determination

One of the critical problems in the application of CT to wood, and to archaeological wood in particular, is that of turning attenuation coefficient values, directly measured by the device, into density values. In fact, if for imaging and for morphological analysis, the knowledge of μ coefficient can be considered sufficient, density characterization needs a calibration process.

In order to achieve this aim two different steps are needed: the first consists in determining $\mu(x, y)$ in each pixel of the image, the second in the transformation of such values in density values.

By using known and homogenous material, a linear function for the normalization of slices, at 15 KeV, was found and then different methods for the determination of wood density were developed and tested.

According to a transformation of Beer-Lambert law the integral of μ along a considered path is:

$$\ln\frac{N_0}{N_1} = \int_{path} \mu(x, y_1)\,dx \qquad [1]$$

where the term $\mu(x, y)$ represents the values of the linear attenuation coefficient at the point (x, y).
The relation between $\mu(x,y)$, E, ρ and Z is:

$$\frac{\mu}{\rho} = k\frac{z^4}{E^3} \qquad [2]$$

where k is a constant.
If the value of linear attenuation coefficient has to be read directly from the images acquired in absorbing mode, a first calibration of the system is required.
In order to achieve that, samples in which density (ρ), atomic number (Z) and the linear attenuation coefficient $\mu(x, y)$ were known for a given X-ray energy were scanned [liquid Propane (C_3H_8), Acetone (C_3H_6O), distilled water (H_2O), Glycerol ($C_3H_5(OH)_3$), Chloroform ($CHCl_3$) and Aluminium (Al)]. The correlation between theoretical and measured values has been verified by fitting the data. A calibration curve of theoretical versus measured values was obtained, and used to directly measure the linear attenuation coefficient in each *(x,y)* point of the sample.
For the transformation of the determined value into density values two different method have been tested: the method of CT numbers [Lindgren 1991] and a new method, called method of "*average density*". In the first method the reference is represented by the density of water, while for the second method the average density ρ_w of the sample (at the same MC value that the sample will have during the X-μCT scanning) needs to be known.

2.3 Sample preparation

In relation with the size of the camera's field of view, samples were cut along the fibre direction with the shape of little parallelepipeds, approximately 30mm by 15mm, with maximum dimension in transversal direction. As the cell wall in waterlogged woods is completely filled of water and its density often heavily reduced by degradation, in saturation condition its residual density is very close to that of water, making very difficult to distinguish between the two phases (wood and water). Because of that, for the measurement of density samples have been dried in a controlled oven at 103 °C for 24 hours, and then enveloped with Parafilm in order to avoid water adsorption and desorption.

3. Results

3.1 Morphological results

Looking at the comparison between images obtained by optical microscope and the tomographic reconstruction (Fig.2), it is obvious that also on archaeological wood, as well as on recent wood, X-ray μCT allows for an easy detection of most part of the anatomical features useful for wood identification. The three-dimensional (3D) reconstruction of the whole samples (fig.3), made possible because the samples were exposed to the irradiation beam along their longitudinal axis, allows the visualization of morphological and structural aspects within the entire volume of the specimens. After that the virtual image is reconstructed, the sample can be cut in any plane or direction, and all the typical sections (cross, radial and tangential) can be virtually obtained.

3.2 Density measurement

In figure 4 is reported the fitting of the relationships between theoretical values of linear attenuation coefficient and the experimental ones, obtained from the scanning of the various substances chosen as reference for different values of density.

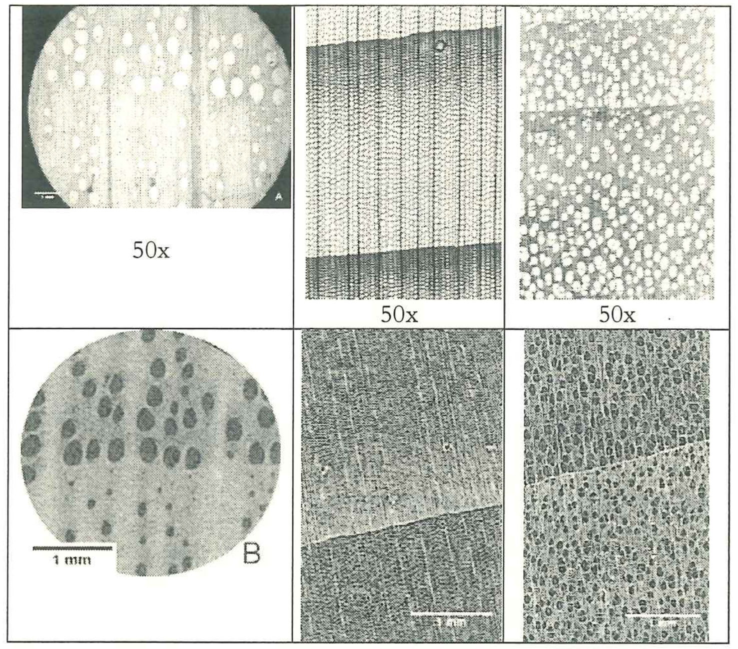

Fig. 2. Comparison between images of cross sections with optical microscope (up) and X-MCT (down). From left to right: oak: early wood porous vessels and parenchyma rays; spruce: early and late wood, presence of resin ducts; poplar: diffuse porous distribution of vessel.

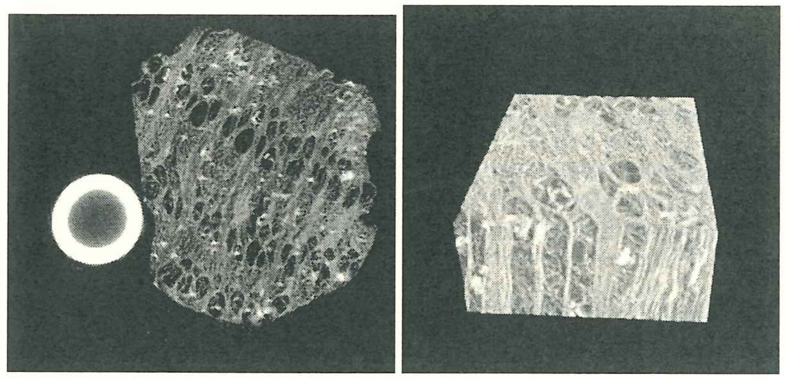

Fig. 3. Left: Cross section of archaeological oak wood (on the left of the wood sample a plastic pipe filled with distilled water); right: the same sample after 3D reconstruction. In both 2D and 3D images are clearly visible, within the structure, mineral concretions (whitish elements) that are largely present in waterlogged archaeological wood.

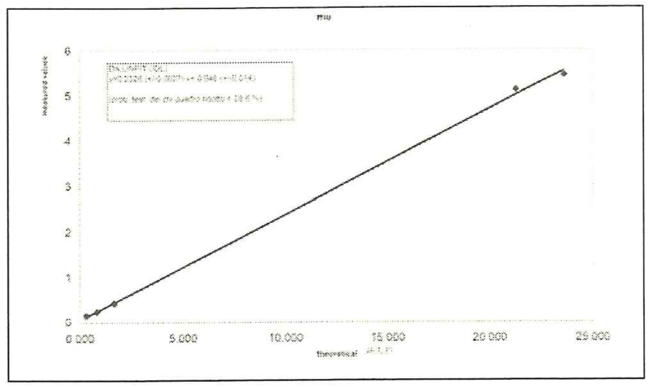

Fig. 4. Theoretical versus measured values linear attenuation coefficient.

The parameters obtained from the model are the following:

$$y = 0.2328(\pm 0.0027)x + 0.046(\pm 0.014) \qquad [3]$$

In order to directly measure the values and the punctual variation of linear attenuation coefficient, all the slices reconstructed were normalized applying equation [3].
In each sample μ was measured along single line of pixels, and as average values in selected areas, chosen avoiding parts too close to the edges of the sample.
Measurements performed on samples of recent wood belonging to Oak, Spruce, Poplar and Larch have given the average results of μ reported in Table 1.

Table 1. Measured values of linear attenuation coefficient on different species.

Sample	**Average μ**	**Density at MC% 12**
Oak	1.259 cm^{-1}	0.80 g/cm^3
Larch	0.490 cm^{-1}	0.65 g/cm^3
Poplar	0.445 cm^{-1}	0.50 g/cm^3
Spruce	0.327 cm^{-1}	0.42 g/cm^3

According to equation [2] variations of linear attenuation coefficient are strictly dependent from the chemical composition and the density of the examined material. As in wood chemical composition (that affect the atomic number Z), can be considered almost the same in all species, the variation of μ is due to the different values of density typical of each species. This has been confirmed by the experimental results obtained, where μ increase its value as the density of the relevant wood species increase (Table 1). The higher values of μ in Oak can be also explained considering the large amount of extractives present in the heartwood of this species. Even their percentage in chemical composition is little, they have an important influence on the Z number of the material and considering that Z is raised to the fourth power in equation [2]. For the same reason the high content in mineral elements (Iron salts mostly) typical of waterlogged wood (see figure 3), may affect quite a lot the value of attenuation coefficient.
Applying the two methods of conversion (i.e. CT number and average density) it has been possible to read the profile of punctual variation of ρ (variation every each 10 microns) along a pre-selected profile within the scanned samples. An example of comparison between the two methods is reported for a sample of oak wood in figure 5.
The values are in the range typical of the specie, and they show a good agreement between the two methods, especially for the lower values of density (i.e. inside the porous vessels). In the parts of the sample characterised by higher values of density (presence of fibres or smaller cellular elements) the method of average density seems to systematically give higher values.

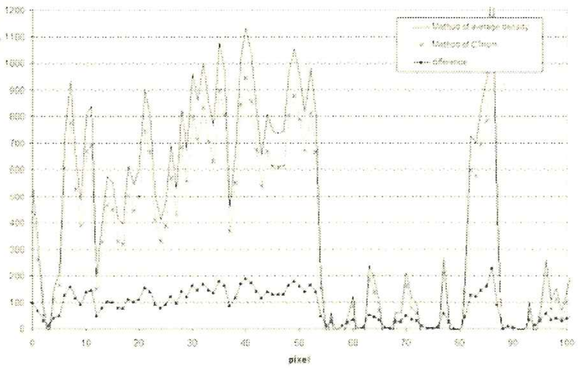

Fig. 5. Density profiles within an annual ring of oak wood. The two curves have been obtained applying the two conversion methods proposed.

4. Conclusion

Results obtained with this preliminary work have shown that X-ray μCT can be a powerful tool for the analysis of waterlogged archaeological wood at least for morphological studies. The two methods proposed for turning values of attenuation coefficient into density seem to be promising, even if a further and deeper experimentation is needed, in order to better understand the differences noticed for the higher values of density within annual rings.

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