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The potential of X-ray computed tomography for 3D anatomical analysis and microdensitometrical assessment in wood research with focus on wood modification

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ABSTRACT

Studying structure and chemistry of wood and wood-based materials is the backbone of all wood research and many techniques are at hand to do so. A very valuable modality is X-ray CT, able to non-destructively probe the three-dimensional structure and composition. In this paper we elaborate on the use of Nanowood, a flexible multi-resolution X-ray computed tomography (X-ray CT) setup developed at UGCT, the Ghent University Centre for X-ray Tomography. The technique has been used successfully in many different fields of wood science. It is illustrated how 3D structural and microdensitometrical data can be obtained using different scan set-ups and protocols. Its potential for the analysis of modified wood is exemplified, a.o. for the assessment of wood treated with hydrophobing agents, localisation of modification agents, pathway analysis related to functional tissues, dimensional changes due to thermal treatment, etc. Furthermore, monitoring of transient processes is a promising field of activity too.

Keywords: microdensitometry; multi-resolution X-ray computed tomography; modified wood; quantitative wood anatomy; structural changes; time-dependent processes

INTRODUCTION

Many techniques are at hand to investigate wood properties, ranging from very simple methods for point measurements down to in-depth three-dimensional characterization. Most of these are either limited to surface or sub-surface examination and/or destructive in nature. A very powerful addition to the existing modalities is X-ray computed tomography (X-ray CT). Tomography is an extension of radiography, i.e. the recording of a shadow image of an optically opaque object using penetrating radiation and a recording medium. For a detailed history of tomography one can read Webb (1990). The purpose of tomography is to obtain a three-dimensional representation of the composition and morphology of the object in a non-destructive way. Basically the 3D object under study is reconstructed based on a set of 2D projections (radiographies) taken from different angles by rotating the sample around a defined axis. In the case of X-ray tomography, X-rays are used as a probe to study objects. X-rays typically have a wavelength below 10 nm and they are attenuated less easily than electrons and visible light. More importantly, their penetrating power is largely dependent on their energy, which is directly related to their wavelength, making it possible to adjust the energy of the source to the type of sample under investigation, depending on size, composition, etc. (Vlassenbroeck 2009). Furthermore, the ability to extract the chemical and densitometric properties of an object from X-ray projections is therefore related to the nature of the interactions between the radiation and the material. These interactions are heavily dependent on the density and composition, allowing to use the transmitted radiation to reveal these properties. Over the years the resolution of CT imaging systems has steadily improved, thanks to the significant progress both in hard- and software. Current state-of-the art lab-based systems are clearly coming of age. Depending on the resolutions required, different manufacturers deliver different systems for non-medical purposes. In addition to the commercial available systems, many groups build their own scanners for specific research purposes, differing in the type of material that has to be scanned, the required specimen size, energy level, etc. X-ray tomography has become a well-established technique and its multi-purpose use is widely recognized in wood science and technology. The number of papers with focus on lab-based X-ray tomography for fundamental research as well as its use as a tool for structural analysis is steadily increasing. A selection of different topics for which lab-based X-ray tomography can be used, shows the versatile employability in both static as dynamic experimental set-ups: wood anatomy (a.o. Van den Bulcke *et al.* 2008; 2009a), structural wood vessel analysis (a.o. Hass *et al.* 2010; Brodersen *et al.* 2011), moisture dynamics (a.o. Lazarescu *et al.* 2010; Derome *et al.* 2011), analysis on from wood derived materials (a.o. Faessel *et al.* 2005; Walther 2006; Walther and Thoemen 2009), etc. The non-destructive internal probing of a material at different scales, ranging from macroscopic down to microscopic level, is a significant advantage. Moreover, thanks to the non-destructive nature of the scanning process, one can monitor and quantify any time-dependent process such as structural changes owing to modification, fungal growth, water movement, weathering processes, etc. Such a technique can, therefore, play an important role in the characterisation of modified wood products. Wood modification has been a field of research for over a century (Rowell 2006; Esteves and Pereira 2009), covering a large range of methods that can be either chemical or thermal in nature, or a combination of both, aiming at an improvement of wood properties. The general awareness of limited natural wood resources as well as an incremental use of domestic, less durable wood species, has been a trigger for a recent increased interest in

wood modification. Modifying wood mainly aims at reinforcing resistance against physical weathering and biological degradation by changing its chemistry and/or structure. By doing so there is a measurable change of the basic wood characteristics such as strength, colour, texture, structure, chemical composition, etc. It is essential to map these property changes before and after treatment to assess both the level of change and the effective resistance to come to a proper dose-response interpretation of the treatment, as well as a quality indicator for established treatments. X-ray tomography can be the pre-eminent technique to do so.

This paper elaborates on the wide range of possibilities of X-ray tomography as a tool for advanced static and dynamic analysis for wood research in general and more specifically on modified wood in its broadest sense. Examples on unmodified wood are presented too, yet all concepts and approaches discussed here are considered transferable to and most useful in the field of wood modification.

EXPERIMENTAL METHODS

The scanner used at Woodlab-UGent is developed at UGCT, the Ghent University Centre for X-ray Tomography (www.ugct.ugent.be). The scanner is similar to the one as described in Masschaele *et al.* (2007) and used in Van den Bulcke *et al.* (2009c; 2011a; b; 2012) and consists of two X-ray tubes and two X-ray detectors, specifically designed to obtain very high resolution scans as well as scans of larger objects. The system offers a large range of operational freedom, all combined in versatile acquisition routines (standard or fast scanning, tiling, helix, etc.). It has a generic in-house developed CT scanner control software platform (Dierick *et al.* 2010) that allows full control of the scanner hardware. Reconstruction of the scans is performed with Octopus, a tomography reconstruction package for parallel, cone-beam and helical geometry (Vlassenbroeck *et al.* 2007) and includes phase-retrieval and correction algorithms (Paganin *et al.* 2002; Grosso *et al.* 2006; De Witte *et al.* 2009). As the system can be used in many fields of wood research, several examples of scans performed with Nanowood are given on different types of materials, including different sizes of wood specimens, wood cores, plywood, etc.

RESULTS AND DISCUSSION

In general high resolution X-ray tomography can contribute considerably to research and development in wood science and technology in general and wood modification more specifically. Depending on the mode of action of the treatment, structural and/or chemical changes are under investigation on different levels. Treatability aspects are valid when comparing wood species (e.g. the role of refractory wood species and heartwood zones) and when discussing the modification processes for engineered wood products and wood based panels. Main interest is however in assessing both qualitatively and quantitatively the impact of different parameters on the modified product as well as characterisation in relation to performance. In this respect 4 focus areas can be identified: treatment level, dimensional stability, biological durability and wood anatomical changes. These are discussed and exemplified below in a framework of both static and dynamic tomography.

Static tomography

Straightforward analysis focuses on the visualization and qualitative investigation of modified wood at different spatial scales. Thanks to the flexible set-up of the scanner, various sample sizes and resolutions can be obtained, enabling broad distribution patterns of modification throughout blocks of treated wood as well as probing the modification down to the cell wall level. The system is capable of scanning samples of 37 cm in diameter and a maximal length of approximately 20 cm with a resolution of 0.2 mm down to approximately 400nm for objects that have about the size of a splinter. Scan times range from several minutes up to several hours depending on the required quality and resolution. One is always confronted with a trade-off between the signal-to-noise ratio and necessity of stability during scanning, as such scan settings (source-detector distance, source-object distance, tube power, exposure time, #projections, etc) are attuned to the research question at hand. A multi-resolution top-down approach for tomographic wood microscopy (Mannes *et al.* 2010) is exemplified for an untreated wood species, ipé (*Tabebuia* spp.), at different scales. This is illustrated in Figure 1 using a cross-section of the scanned volumes. These images were taken with standard cone-beam tomography. For very high resolution scans, such as the rightmost image of Figure 1, phase contrast must be taken into account. Although conventional tomography is based on the attenuation of X-rays, both the amplitude and phase change when passing through an object. At very high resolution this effect is visible and can, in some cases, be beneficial to visualise small details, yet often the mixture of phase and amplitude hampers proper processing. Therefore, to mitigate this effect, phase contrast filtering is often applied (Boone *et al.* 2009; De Witte *et al.* 2009; Wernersson *et al.* 2013).

Structural analysis allows for analysis of *treatability* as can be deduced from the work of Svedström *et al.* (2012) on aspen and a series of examples given by Van den Bulcke *et al.* (2009a). The image processing chain is illustrated in Figure 2 for labelling of the vessel lumina of oak (*Quercus* spp.). Although only a single cross-section is shown, all algorithms run in three dimensions as shown in the resulting 3D rendering in Figure 3a. Similar results could be obtained with images acquired with standard light microscopy, SEM or another technique, yet these are mostly limited to visualization of the (sub-)surface while internal features can only be revealed through sectioning or any other process of material removal, possibly creating artefacts as well. X-ray tomography on the other hand enables a one-to-one analysis of a sample before and after treatment in three dimensions, as such not limited to the surface. All analyses are performed with the in-house developed software package Morpho+, containing an extensive set of high-performance 3D operations to obtain object segmentation, separation, and parameterization or to extract a 3D geometrical representation for further modelling (Brabant *et al.* 2011). First the original volume is pre-processed, applying bilateral filtering to remove noise without extensive smoothing, i.e. in order to keep sharp transitions such as edges. In a second step the optimal threshold level is chosen to segment vessel lumina. The latter can be used to map different objects and label them separately. Subsequently, a distance transformation can be calculated to classify objects according to their size, shape, etc. and to separate them optimally when connected. The final result of such a process is given in Figure 3a, showing a 3D image of the labelled vessel lumina of the oak sample under investigation. Figure 3b is an example of fibre labelling on aspen (*Populus tremula*). The same principle is of course feasible for treated wood. Treating chemicals or derived polymers can be present mainly in the cell lumen or cell wall penetration can be envisaged.

Anatomical pathways can be primarily through axial tissues consisting of vessels in hardwoods or earlywood tracheids in softwoods, but some complementary involvement of ray pathways is required to obtain adequate distributions. Additionally it is relevant to check on envelope or matrix treatment by assessing treatment gradients linked to *treatment level* for products like resins, oils, coatings and even for hydrophobation agents like siloxanes.

Chemical distribution is elaborated on in the paper of De Vetter *et al.* (2006), describing the use of X-ray tomography for the distribution analysis of organosilicon compounds in wood. Scholz and co-workers (2010a) explore the impregnation of waxes related to the anatomy of Scots pine sapwood (*Pinus sylvestris*) and beech (*Fagus sylvatica*) complementary to SEM. This kind of top-down approach gives the opportunity to investigate the structure in depth in a fast and non-destructive way, offering the possibility to combine with other analysis if necessary. Especially the latter one-on-one relation is highly important to anticipate the inherent biological variability. Similarly, the follow up research of Scholz and co-workers (2010b) uses X-ray tomography for quantification of the pore size and wax distribution, with comparison of these results with other techniques, showing the complementary functioning of non-destructive tomography. Also, Kucerova (2012) illustrates the use of X-ray tomography to measure the penetration of consolidant solutions into wood and Ahmed and co-authors (2013; in press) show the potential of CT scanning for studying preservative distribution in thermally modified wood.

An example of resin deposits on the walls of Scots pine sapwood tracheids is given in Figure 4a. The wood was treated with an aqueous solution of a methylolated melamine formaldehyde (MMF) resin, cured in a laboratory oven. The aim of this investigation was to localize the cured resin droplets and assess how the specific anatomy of a wood species influences the impregnation behaviour. Visualization and analysis of coatings can also be considered as a form of surface modification, resulting in full structural characterization (Van den Bulcke *et al.* 2010). Figure 4b illustrates the visualization of an opaque coating on Scots pine.

Assessment of *dimensional stability* is mainly linked to a dynamic analysis however differential scanning can allow verification of bulking effects of e.g. acetylation on specific parts of wood and the refractory nature preventing chemical penetration, e.g. poor treatment of Scots pine heartwood. As Boonstra and co-authors (2006) illustrated it can be useful to check the *wood anatomical alterations* of modified wood. Treating processes to obtain TMT products can initiate collapse or related features that can be quantified on anatomical structure level.

More advanced analysis includes connectivity before and after treatment, e.g. for *fungus pathway analysis* as illustrated in Van den Bulcke *et al.* (2008). High-resolution imaging of penetration routes in oak, beech, bamboo etc. are under investigation as well as glue barrier analysis. This enables verification of non-toxic treatments allowing fungi to grow into the wood matrix however without inducing rot. Pathway analysis was also developed by Hass and co-authors (2012) to study adhesive penetration in wood using synchrotron-based tomography.

The above-mentioned analyses derive quantitative structural information from the volumes but without the use of the data resulting from the underlying principle of a

direct relation between greyscale and density. Although this relation is not straightforward and depending on the chemical composition as well, there is an opportunity of extracting densitometrical data from these volumes for wood as composition in most cases can be considered more or less constant. Briefly, the basic principle relies on the use of a reference material with a density approximating cell wall density and recalculating all 16 bit greyscale values accordingly. As such the scanned 3D volume is converted to actual densities. More information on the methodology and validation of the technique can be found in the paper by De Ridder and co-workers (2011), using helical X-ray tomography on limba (*Terminalia superba*) wood cores. Helical tomography is similar to standard cone-beam tomography, but instead of a circular movement, the sample describes a helical trajectory. Helical tomography has several advantages, especially when scanning elongated objects such as wood cores, but comes with a higher mechanical and mathematical complexity; reconstruction is based on the Katsevich algorithm (De Witte 2010). Helix scanning is used complementary to cone beam scanning enabling gradient analysis over cross-sections of trees (Van den Bulcke *et al.* 2012) and this can be used for verification of *microdensitometric differences* as a result of modification processes. An example to illustrate this potential is given in Figure 5 for non modified limba and Scots pine. It should be noted that the 1D profile given is an average of each cross-sectional slice taken in radial direction, i.e. the density is derived from actual 3D density data.

A similar principle can be used in the field of wood modification, when evaluating density changes of treatment, visualization and assessment of moisture, etc. by either sub-sampling of core-like samples of larger material, region of interest scanning or by tomography of the entire specimen if small enough.

Time lapse and dynamic tomography

Time lapse tomography is the analysis of the same sample at different times, such as the analysis of density changes during fungal attack (Van den Bulcke *et al.* 2011a), high-resolution fungal growth monitoring (Van den Bulcke *et al.* 2009b), etc. In fact, the analysis of the effect of thermal treatment can be considered an example of time lapse tomography, when the same sample is scanned before and after treatment. An example of such possible changes is given in Figure 6 illustrating the shrinkage of thermally treated aspen. The treatment was carried out in a device made by wood thermal technology (WTT). Samples were placed in an autoclave and subjected to a specific thermal treatment consisting of three main stages: (1) temperature increase up to the modification temperature (160 °C); (2) holding at the modification temperature (1 h); (3) cooling. In the course of the process, as the water evaporated at a rate dependent on the treatment temperature in the chamber, the pressure increased, reaching 5–9 bars. For more information on the treatment process and microstructural, the reader is referred to Biziks *et al.* (in press).

Quantification of change in porosity, even on a tissue level, can help interpreting the treatment process. 3D image registration can also map the actual shrinkage of the sample on a voxel-by-voxel basis, as such also inferring on the stress-strain fields. The work of Forsberg and co-authors (2008; 2010) is pioneering in that respect.

Furthermore time lapse tomography is key when analysing the impact of water and *moisture content* both related to dimensional changes and time of wetness (ToW) analysis. An example of combining moisture monitoring using a CMM (Continuous Moisture Monitoring) approach (Van den Bulcke *et al.* 2009c) and the effect of

weathering thereon is given in the paper by Van den Bulcke and co-workers (2011b). Analysis revealed glue cracking and as such some plywood samples reached high moisture contents.

Dynamic tomography on the other hand refers to the actual monitoring of a dynamic process during scanning. For very fast processes (< 1 min), full tomographical scanning is currently difficult if not performed at synchrotron facilities. Synchrotron radiation has some unique properties, making it highly suitable for X-ray imaging. Since its high flux is concentrated into a cone beam with an extremely narrow opening angle, originating from a very small source area, imaging can be performed far away from the source and at very high speed, therefore ideal for dynamic tomography, such as is elaborated on in the papers by Derome *et al.* (2011) for the hysteretic behaviour of wood and Zauner *et al.* (2012) for the observation of plastic deformation of uniaxially compressed Norway spruce samples. Radiographies, however, can also be useful. Figure 7a shows an example of a set-up for contact angle measurement and water penetration in a sample of MDF (Medium Density Fibreboard). Through image processing, only the water is made visible and penetration can be followed in time. For processes that are slower than 1 minute or for which a high image quality is not required, full 3D analysis of a time-dependent phenomenon is feasible. Figure 7b illustrates the penetration of water in plywood veneers. Water penetration was monitored for several hours and separated from the plywood by image processing.

Currently, faster scanning protocols are developed that will enable full tomographical scans at high-resolution in less than a minute to follow dynamic processes in 3D. Such fast scanning is also beneficial when working with living biological tissue in which stress and possible damage due to radiation has to be reduced to the minimum (Van den Bulcke *et al.* 2009b). Clearly, implementation at lab-based systems is a continuing task.

CONCLUSIONS

The use of high-resolution X-ray computed tomography in wood science is increasing considerably. Early research with this technique was mainly focusing on its applicability as an advanced imaging tool to reveal the internal structure non-destructively, but the last decade the wood science community clearly has discovered it as a modality for quantitative research, not the least owing to the significant progress in hardware, the massive computational resources available and the commercial availability of user-friendly table-top systems. Wood is especially challenging because of its hierarchical nature, demanding a multi-scale approach with regard to three-dimensional imaging. The enormous potential of flexible X-ray tomography scanners such as Nanowood can result in fast 3D characterization of the same material before and after treatment for modified wood, following dynamic processes with time-lapse scanning, etc. leading to new insights and improved treatment procedures. The abovementioned examples are only a small part from a long list of possible experimental set-ups and scanning techniques and this is expected to increase substantially in the near future. Together with other equipment for semi- to non-destructive testing, it offers wood research with the possibility to go beyond current state-of-the-art. New scanning set-ups such as dual-energy and phase-contrast, as well as reconstruction algorithms such as the family of iterative techniques, will lead to faster scanning and faster and improved reconstruction

and visualization of different, difficult to follow and discern phenomena and phases. Furthermore, the hierarchical nature of wood necessitates its study on different spatial scales. As phenomena emerging on a high resolution have an impact on all other resolutions, multi-resolution imaging of a complex material such as wood in general, and modified wood more specifically, is one of the major challenges.

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FIGURE CAPTIONS

Figure 1: Cross-sections of *Tabebuia* spp. at different scales (length of image given below each image)

Figure 2: Vessel segmentation and labelling in oak, a: original, b: bilateral filtered, c: thresholded vessel lumina, d: labelled vessel lumina, e: distance transform, f: overlay of size-labelled vessel lumina on the original cross-section (diameter of sample approximately 12 mm).

Figure 3: Vessel lumina segmentation in oak (a; diameter approximately 12 mm) and fibrefoc lumina segmentation in aspen (b; cross-sectional dimensions approximately 0.4 mm x 0.5 mm).

Figure 4: Resin deposits on Scots pine tracheid walls (a; longest edge of volume approximately 0.25 mm) and an opaque coating (red) on Scots pine (b;). For the latter the wood is virtually removed to illustrate the rough penetration profile of the coating in the tracheids.

Figure 5: 3D volume of a core of limba (a; diameter of core approximately 5 mm) and of Scots pine (b; cross-section approximately 3 x 3 mm) and corresponding microdensitometrical profile with ring width and age (c).

Figure 6: Aspen before (greyish) and after (brownish) thermal treatment (cross-section approximately 0.4 x 0.5 mm).

Figure 7: Water droplet penetration in an MDF (Medium Density Fibreboard) panel (a1 to a3); penetration of water in plywood in 2D: b1 illustrates the cylinder containing the water on top of the plywood panel (darker lines = glue) and b2 illustrates the visualization of the water in the first veneer; a 3D version of water penetration (blue) in the first veneer of plywood is shown in (c).

Figure 1

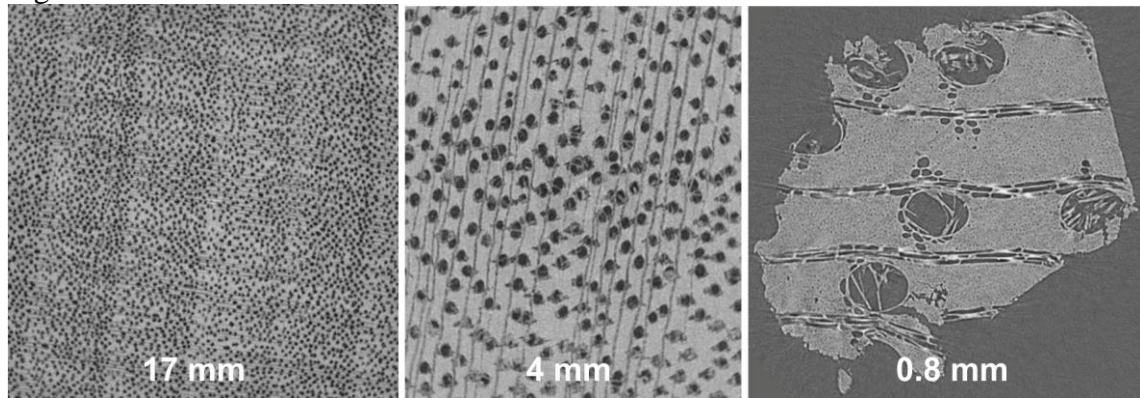


Figure2

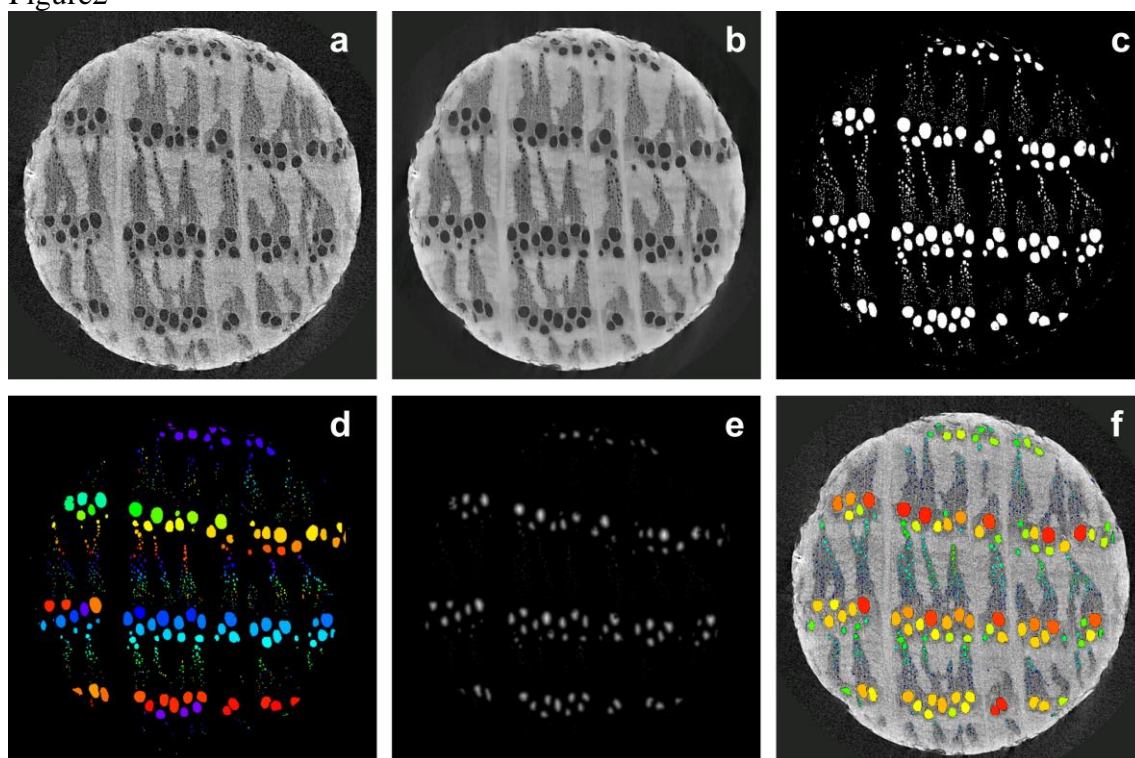


Figure3

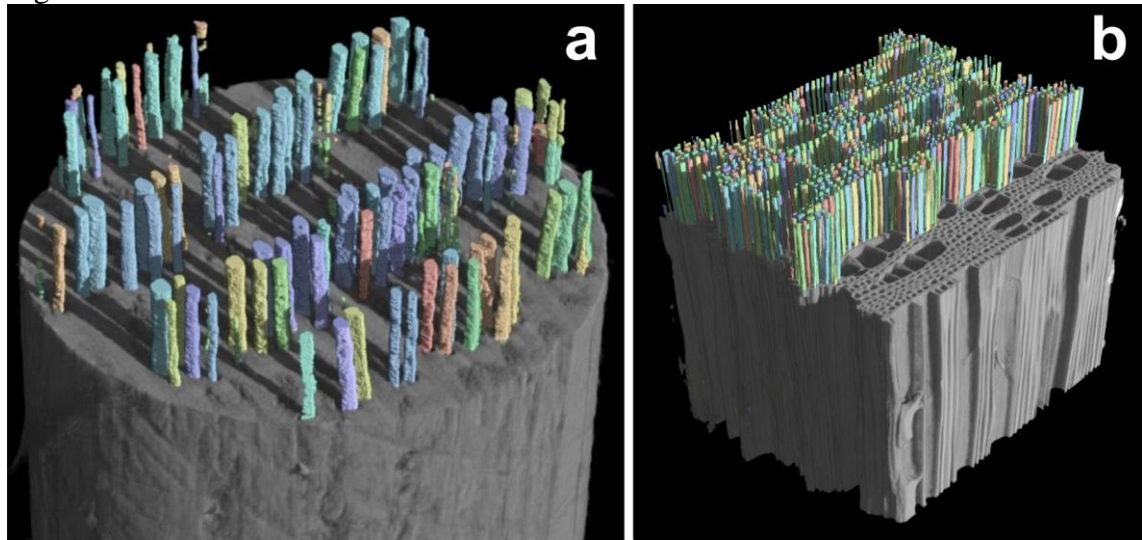


Figure4

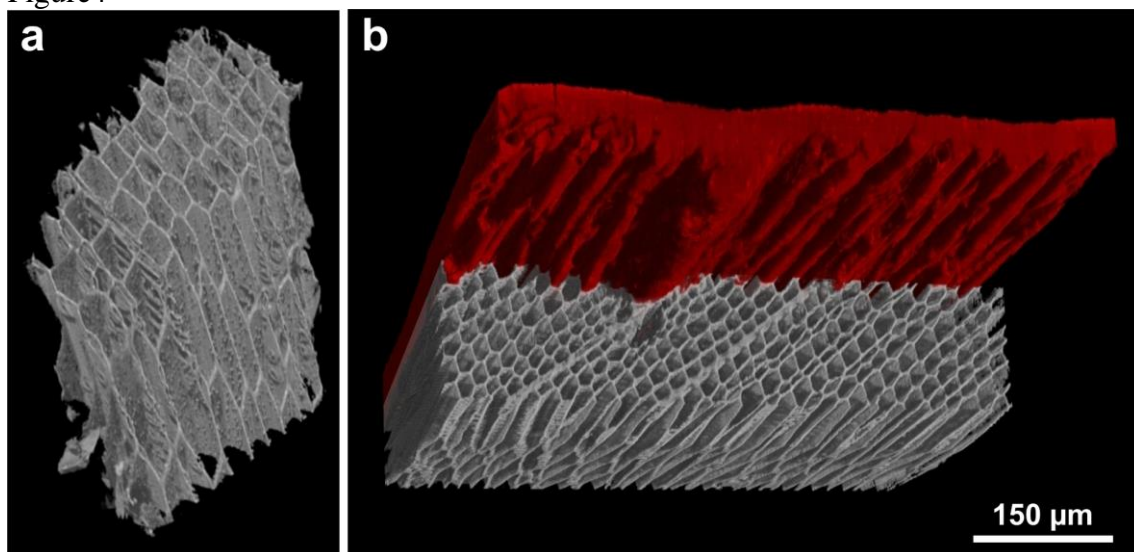


Figure5

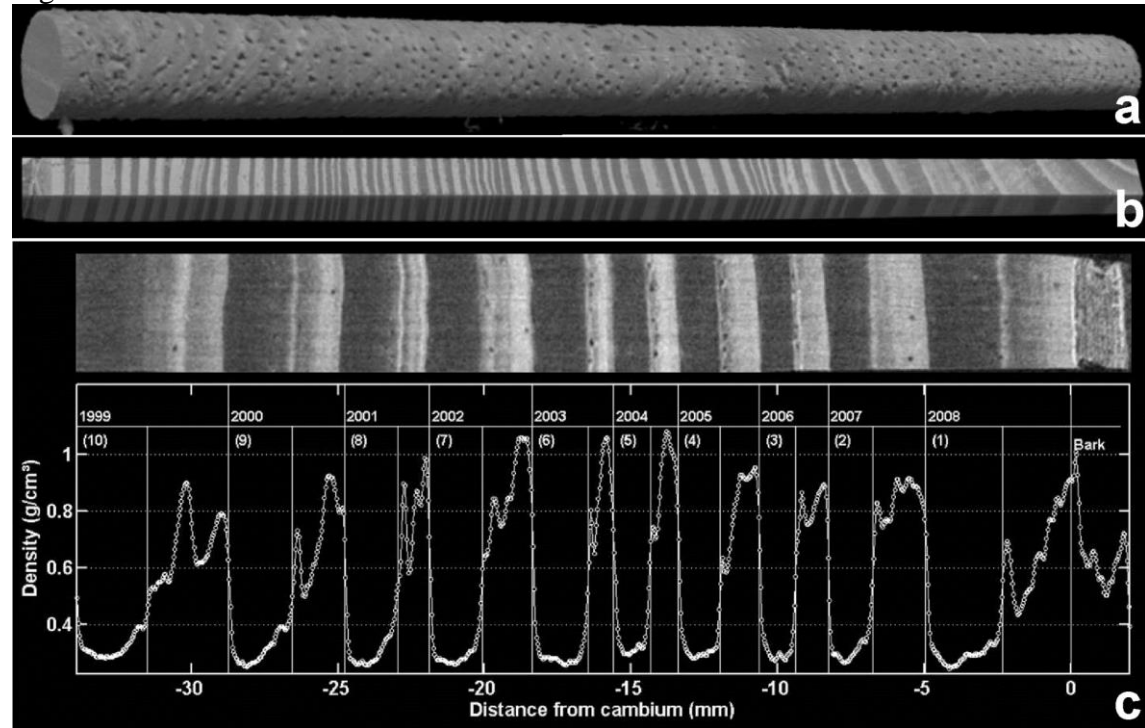


Figure6

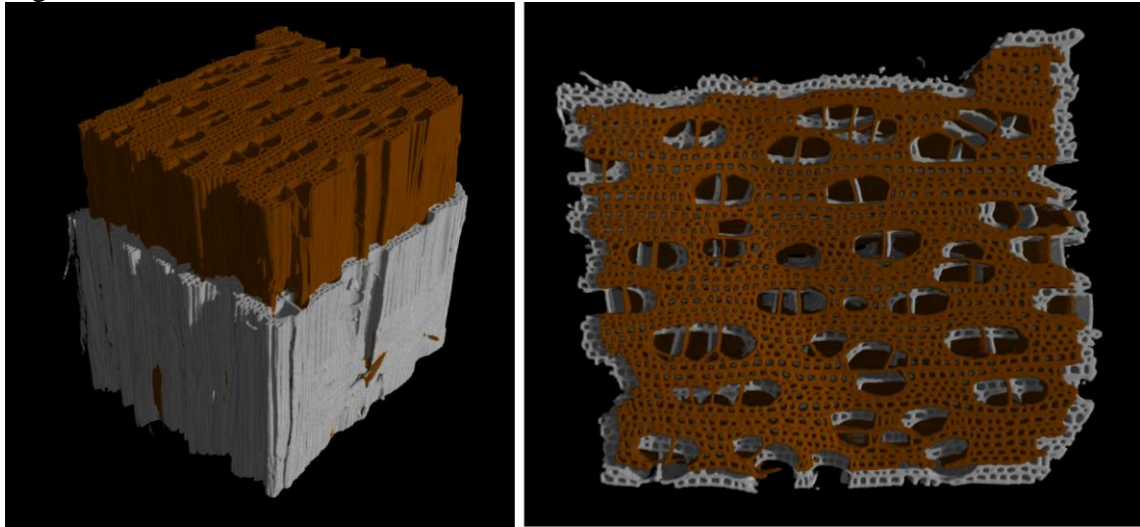


Figure7

