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# The cork viewed from the inside

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ABSTRACT

Cork is the natural material stripped from the outer bark of cork oak. It is still the most used stopper to seal wine bottles and to preserve wine during storage. Cork stoppers are sorted in different classes according to apparent defects, named lenticels, which can be related to the cork macroporosity. The more lenticels there are, the worst cork quality is. The present work aims at investigating defects analysis of cork stoppers from two classes by comparing images recorded by digital photography and neutron imaging. Surface analysis of defects obtained from photography leads to more surface defects in class 4 (6.7%) than in class 0 (4.1%). Neutron radiography and tomography are powerful methods that really show the defects inside the material. From neutron radiography and tomography, class 4 contains 7.5% of volume defects and class 0 5.9%. Moreover, tomography also allows observing defects distribution along the whole stopper and possible interconnectivity.



## 1. Introduction

Cork was one of the first materials put under the microscope. The first depiction dates back to the years 1660, when Robert Hooke drew the scheme of its very characteristic cellular organization, giving the term cell to the basic biological unit (Hooke, 1664). More recently, Gibson et al. (1981) described the geometry of cork cells in three different sections: axial, radial and tangential (Fig. 1).

Cork was the prime candidate for sealing of beverages from amphorae at the Romanian age up to wine bottle with a marked increase since the industrialization of the glass processing in the 19th (Karbowiak et al., 2010). Nowadays, it is also used in a large range of applications such as floor covering, activated carbon, acoustic and thermal insulation (Gil, 2009; Silva et al., 2005). In its use as sealing, cork is sorted in different classes according to a main characteristic: the proportion of lenticular channels. These defects can be considered as the macroporosity. The more lenticels there are, the worst cork quality is. The knowledge of the structure is important to better understand the mass transfer properties in its use as a sealing material (Giunchi et al., 2008; Karbowiak et al., 2010; Lequin et al., 2010). Cork is generally sorted visually by hand or by optical analysis (Pereira et al., 1996; Prades et al., 2010) as a function of its overall outside general aspect, considering the defects viewed from the outside are a good estimation of the inside. Some other technics such as X-ray or Terahertz imaging were recently used to get a better understanding of the inner structure of cork samples or to perform 3D reconstructed images by tomography (Brunetti et al., 2002; Donepudi et al., 2010; Teti et al., 2011). Neutron imaging is another non-destructive and non-invasive method which allows characterizing materials structure and defects at the microscopic length scale (Lehmann et al., 2011). Whereas light can only probe the inside of transparent materials, neutrons and X-rays penetrate most materials to depths of several centimeters. X-rays are scattered by atomic electrons whereas neutrons are scattered by atomic nuclei. This results in a number of differences between the two methods, perhaps the most important being in the scattering from light elements. For example the hydrogen nucleus scatters neutrons strongly and aluminium only slightly. These last properties allow the observation of defects or fluid migration in biological materials (if one plays with the thickness of the samples). Structural studies inside usually opaque materials or during processes can also be easily performed (Tanoi et al., 2009). Classically, neutron imaging has

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been used for quality control purposes in industries that require precision machining such as aircraft, motor engineering, metallurgy or material science (Boillat et al., 2010; Kardjilov et al., 2009; Warren et al., 2013). However, to study the microstructure of complex materials with a higher resolution (few µm), X-ray and neutron imaging experiments have to be performed in large scale facilities.

Whatever the technique used, cork stoppers are grading in different classes. Generally there are 7 or 8 qualities (Ferreira et al., 2000; Benkirane et al., 2001; Fédération française des syndicats du liège, 2006; Natural cork users group, 2007), but there is no well-defined standard (Pizzurro et al., 2010). Class 0 represents the best quality while 6 or 7 (depending on the classification used) is the poorest. The aim of this study is to identify and quantify defects present in different classes of cork stoppers with two techniques: digital photography and neutron imaging (radiography and tomography).

## 2. Material and methods

### 2.1. Cork stoppers

Raw natural cork stoppers, from *Quercus suber* L. oak trees in the Mora (Portugal) production area, were supplied by Bouchons Trescases S.A. (Boulou, France). Two qualities of cork stoppers were chosen: high quality (class 0) and lower quality (class 4). Stoppers were neither washed nor surface treated (with paraffin or silicone) prior to use. Cork stoppers of 24 mm diameter were cut with a cutting machine, Mecatome T201 with resinoid cut-off wheels of 180 mm diameter and 0.5 mm thickness (Presi S.A, France). They were cut following the axial section, which means the section corresponding to the surface in contact with the wine, which is perpendicular to the axial axis. Cork wafers were of 24 mm diameter and 3 mm thickness. For each quality, 12 wafers from 12 different stoppers and 12 wafers from a single stopper were used, in order to compare the variability between stoppers and within a single stopper along its axial direction. For such a heterogeneous natural material, it is obviously not possible to consider only few samples, but it requires a large enough set of individuals to truly depict it.

### 2.2. Digital photography

The surface of cork wafers was observed by taking pictures of the axial section. The image acquisition was made with a Nikon D200 camera equipped with a micro Nikkor 55 mm f2.8 objective. Cork samples put on a black background were lit with two indirect optical fibers as a light source, giving an intensity of 70 lux at the surface of the samples. Aperture 4 was chosen, iso sensitivity was set to 100 and the picture was slightly underexposed (−1 IL) in order to achieve good image quality and good contrast. The original image size was 3872 × 2592 pixels, with 32 bits per pixel.

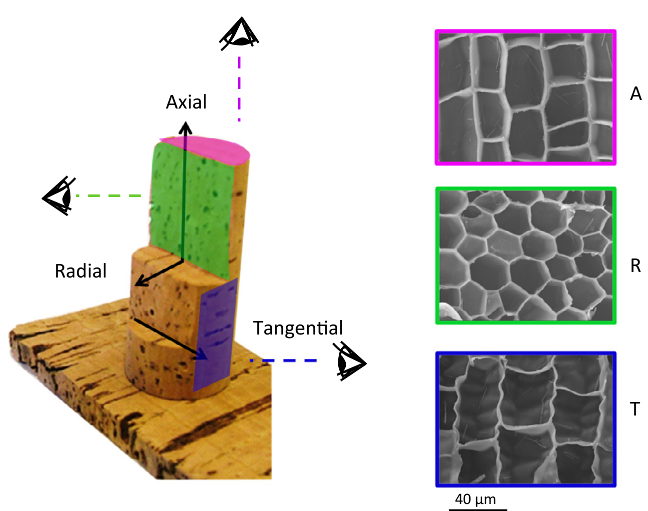


**Fig. 1.** Cork seen from the three directions, with the three corresponding scanning electronic microscopy pictures of the axial (*A*), radial (*R*) and tangential (*T*) sections.

### 2.3. Neutron radiography

The neutron radiography experiments were performed on the new cold neutron imaging station (IMAGINE) at the Laboratoire Léon Brillouin. The station is located in the neutron guide hall on the cold guide G3bis. The available neutron spectrum extends from 3 to 20 Å. The detector position was at around 4 m from the aperture of the source, and the diameter of the aperture was 18 mm. In this configuration, the field of view on the detector was around 70 by 70 $mm^2$ and the neutron flux was $2 \times 10^7$ neutron $s^{-1}$ $cm^{-2}$. A sCMOS camera from photonics science coupled with a 100 µm lithium scintillator has been used for image acquisition. Spatial resolution was around 200 µm/pixel. Exposure time was 7 s by image and an average of 32 images was performed to increase the quality of the image. The image size was 2048 × 2048 pixels. Background noise and open beam were measured at the beginning of each series of samples under the same experimental conditions.

### 2.4. Neutron tomography

The neutron tomography experiments have been performed on the thermal neutron imaging station NEUTRA at the Paul Scherrer Institute. On Neutra, the neutron flux on the detector is around $7.5 \times 10^6$ neutrons $cm^{-2}$ $s^{-1}$ $mA^{-1}$. The maximum field of view at the better resolution was 150 × 150 $mm^2$. Six tomographies were performed on three full cork stoppers for each of the two qualities studied. A lithium 6 scintillator (100 µm thickness) was used. The pixel size was 63 µm. 625 projections were taken all around the samples (360° rotation) and the exposure time was 10 s.

### 2.5. Image analysis

#### 2.5.1. From photography

Images from digital photography were firstly cropped to 1992 × 1992 pixels around the region of interest (cork surface) without resizing. Hough transform was used to detect the edge of the cork disc in order to crop a square centered on the cork disc. This image was then converted into gray scale in 8 bits. A threshold of gray level value of 65 was chosen for binarization. Such high value was used to only have lenticels appearing out of the image, without any false positive. False positives mean some darker parts of the cork (such as ring growths), which would appear as black areas and be considered in the following as lenticels. Image treatment of the binarized image was then done applying an algorithm based on morphological erosion with an elliptical kernel of 5 × 19. This operation enables a better filling of the lenticels regions by taking the information and texture of the surrounding area and gives a result very close to the original image. The "defects" of the material, defined as the percentage of lenticels present onto the surface, was expressed as the percentage of the number of black pixels over the total number of pixels in the full disc (corresponding to the initial cork surface). This last value is the sum of white and black pixels of the full image, the outside of the cork disc appearing as a grey mask in order not to be taken into account in this calculation (see Fig. 2).

This surface defects detection and quantification was determined for four cork series (class 0 or class 4, from a same stopper or from different stoppers). For each series, a Lilliefors test was first