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Towards a description of the degradation of archaeological birch bark

JOHANNA KLÜGL*
Archaeological Service of the Canton of Bern
Bümpliz, Switzerland
University of Bern/Bern University of the Arts
Bern, Switzerland
Johanna.Kluegl@erz.be.ch

ALBERT HAFNER
University of Bern
Bern, Switzerland
albert.hafner@iaw.unibe.ch

GIOVANNA DI PIETRO
Bern University of the Arts
Bern, Switzerland
Giovanna.DiPietro@hkb.bfh.ch

*Author for correspondence

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ABSTRACT

Archaeological birch-bark artefacts from ice patches are rare and little knowledge about their conservation exists. The degradation mechanisms are unknown and it is uncertain how they affect the mechanical properties and the cell structure. Due to this lack of knowledge, the treatments for archaeological birch-bark artefacts usually mimic those for waterlogged wood, which are tuned to the preservation condition of the object. This is assessed by measuring the maximum water content and, in some cases, the basic density and by microscopic examination of microscopic examination. In this paper, it is explored whether these parameters and techniques can be used to characterise the degradation of archaeological birch bark. Light microscopy examinations showed that cell wall deformations and fractures were present in both unaged reference material and archaeological birch bark and are not a distinct attribute of degradation. Cell collapse was not detected in ice-logged samples, while loss of birefringence is a potential tool to characterise degradation. Birch bark cells cannot be saturated with water, not even in the case of waterlogged archaeological samples. The authors conclude that maximum water content is not a diagnostic tool to quantify degradation.

INTRODUCTION

Thanks to its flexible and water-repellent nature, birch bark has been widely used throughout history for the production of a variety of objects, ranging from vessels, shoes and hats to canoes and manuscripts. Nevertheless, archaeological finds made of birch bark are unusual compared to the amount of waterlogged wooden finds, as wood served as a building and construction material. Thus, there is broader knowledge about the degradation and conservation of waterlogged archaeological wood. As a consequence, conservators tend to apply the same conservation solutions developed for waterlogged wood to birch-bark objects. However, birch-bark and wooden objects differ substantially in their morphology, chemical composition, degradation pattern and retrieval environment.

Birch wood is primarily composed of axially oriented wood fibres and vessels. The vessels are interconnected and allow water to flow from one cavity to the other. Birch bark, on the other hand, is made of closed, isolated, transversally oriented cells with different sizes and cell wall thicknesses arranged in layers. This structure ensures that the bark cannot be easily penetrated by water or gases (Jensen 1963, 595) and that gas and water exchange takes place uniquely through horizontal openings called lenticels.

Wood is made of cellulose, mainly located in the secondary cell walls, with hemicellulose forming the cell wall matrix and lignin binding the cells together through the middle lamella. Birch bark, on the contrary, is primarily composed of suberin (a lipophilic polyester), which forms the secondary cell wall, and Betulin (a pentacyclic triterpene), a non-structural component present in the lumen of the broader cells of the birch bark. Further structural cell wall components are lignin, forming the matrix of the middle lamella and present in the secondary wall, and small amounts of polysaccharides to constitute the primary and tertiary cell wall (Frey-Wyssling 1959, 61; Pereira 2007, 87; Pinto 2009, 128).

Degraded waterlogged wood suffers during uncontrolled drying from severe volume reduction and warping due to the collapse of the decayed cell walls. In birch bark, however, deformations and curling take place whenever the bark is plasticized (Gilberg 1986). Furthermore, delamination, a separation of layers developing along the border between the different cell types, is a common type of damage.

Finally, the retrieval environments are also different. While archaeological wood is mostly found waterlogged in sediments, birch-bark objects can be retrieved either from waterlogged sediments or from ice patches and permafrost. Due to the limited size and thickness of these objects, they often reach the conservation workshop already in a dry state (Goedecker-Ciolek 1996).

The conservation strategies developed for waterlogged wood are tuned to the condition of the treated object, characterised through its values of maximum water content (MWC) and basic density (BD) (Macchioni 2003, Jensen 2006). These measurements are often coupled with direct investigation of the micromorphology through light microscopy (LM) or transmission electron microscopy (TEM) and scanning electron microscopy (SEM) (Pedersen 2012). Given the use of MWC, BD and LM measurements in waterlogged wood conservation, it is often assumed that these parameters can be appropriate attributes for the characterisation of birch bark objects.

In this paper, the authors explore if and how LM, MWC and BD are practicable methods to determine the degradation of birch bark objects. First, an overview of the application of these techniques is given to characterise waterlogged wooden objects, and later it is shown how these methods have been adapted to the investigation of contemporary and archaeological birch bark and the results obtained.

LM has been widely used to describe and classify archaeological waterlogged wood degradation patterns (Blanchette 1990, Björdal 1999). Several authors have shown that bacterial metabolism of the cellulose-rich S2 cell wall is the main decay path, whereas the middle lamella and also often S1 and S3 cell wall layers are partly preserved (Pedersen 2012). The loss of the crystalline cellulose in the S2 cell wall has been confirmed by the reduction in the birefringence observed under polarised light (PL) (Björdal 1999, Pedersen 2012). To distinguish between unlignified and lignified cells, double staining with safranin O and astra blue has been used, while aniline blue in lactic acid has been used to stain fungal hyphae and bacteria (Björdal 1999, 64), and toluidine blue to stain decay features of erosion bacteria (Pedersen 2014).

LM has never been used to investigate the degradation of birch bark but has been used since the 19th century to study the anatomy and formation of birch phellem (Mohl 1836, Von Höhnel 1877, Moeller 1882) and later to study the cell shape and size (Jensen 1949, Chang 1954) and the transport of water through the bark (Schönherr 1980).

As the loss of cell wall material induces a reduction in the density of the samples and an increase in the volume of the cavities, MWC and BD are the attributes of choice to characterise wood degraded in an anoxic environment (Macchioni 2003, Jensen 2006).

MWC is defined as the percentage amount of water contained in a sample with respect to its dry mass when all cavities are completely filled with water. Classification of the degradation of waterlogged wood is based on MWC values (De Jong 1977, 324; McConnachie 2008) and these are

used to decide on the polyethylene glycol (PEG) treatment. To determine the MWC, the fully water-saturated and dry mass of the sample are measured gravimetrically. To fully saturate the samples, the methods used in conservation (Hoffmann 2013, 33) mimic classical methods used in forestry. They consist in submerging the sample in water and then either reducing the surrounding air pressure with a vacuum pump (Smith 1954, 3*f*) or boiling the sample (Keylwerth 1954, 78). Care is taken in removing the surface water when measuring the water-saturated mass (Keylwerth 1954, 78; Smith 1954) as excess surface water is a source of error if the surface area is large in comparison to the volume (Panter 1996, 187) or for sample sizes smaller than 0.5 cm^3 (Jensen 2006, 554). The dry mass is usually measured after drying at 103°C.

No investigation of the MWC of birch bark has been published as yet.

The BD of a wooden sample is defined as the dry mass of the sample divided by the fully swollen, waterlogged volume. From the literature data, the percentage difference between the BD of unaged and aged samples, the so-called residual BD, can be calculated. Despite the variability of the within-species wood density (calculated with respect to the dry or green volume) being of the order of ± 30% (Longuetaud 2016), the residual BD is commonly used to estimate the state of degradation of the sample (Jensen 2006).

The BD can be calculated from the MWC value and density of cell walls on the assumption that all wood cavities are filled with water and if the density of the cell walls is known (Hearmon 1958).

Few authors have measured the density of contemporary unaged outer birch bark (Bhat 1982, Groh 2000, Holmberg 2016), but the results differ largely and allow a range between 0.45 g/cm^3 and 0.77 g/cm^3 to be defined. Large differences in density have also been found for oak cork in a survey of 680 trees leading to values between 0.16 and 0.47 g/cm^3 (Pereira 2007, 191). The large variation in the density of unaged birch bark does not allow for the necessary reference for the calculation of the residual BD, the actual attribute characterising degradation. Therefore, BD was not investigated further in this study.

EXPERIMENTAL

LM and MWC have the potential to convey information about the degradation state of birch bark if they differ substantially among archaeological and contemporary samples. Both types of samples were therefore analysed (Table 1).

Light microscopy

To evaluate the state of preservation and to identify possible micro-morphological decay, small (5 to 10 mm) rectangular samples were cut from archaeological and contemporary birch bark using a razor blade. Thin sections (8 to 12 µm) in radial, transverse and tangential directions were produced using a Leitz 1208 sliding microtome and an N42 blade. Investigations were performed with an Olympus BH-2 light microscope using both transmitted and polarised light at different magnifications

(04, 10, 40 and 60). Photographs were taken with a Jenoptik ProgRes SpeedXT Core 3 3.0MP CCD digital camera. Helicon Focus 6 focus stacking software was used to produce a fully focused image.

Maximum water content

Portions of material from samples No. 1–3 (100 × 100 mm) and 5, 6 (Ø 40 mm) of a thickness ranging from 0.8 to 2.6 mm were submerged for 5 days in water by using appropriate weights as they otherwise floated. To achieve the removal of air and penetration of water into the specimens, air was evacuated in steps (150, 100, 50, 30 and 20 mbar) lasting 30 minutes each and interposed by pauses of the same duration at standard pressure. The integrity of the cell walls upon air removal was monitored by transmission LM at the different stages on sections of a sacrificial sample. The wet mass was then measured after removing excess surface water using a moisturised PE non-woven fabric (Dermotekt) and storing the sample for five minutes in a closed 800-ml container.

The dry mass was measured upon drying the sample for six weeks in a closed container with RH = 1% using a desiccant. Heating of the sample was avoided to prevent possible evaporation of volatile components.

Table 1.

Sample no.	Origin	Measurement	Size in mm	Condition	Dating
Contemporary					
1-2-3	Tomsk, Siberia, Russia, living tree	LM, MWC	100 × 100 × 2.6	air-dry	2012
4	Wallis, Switzerland, living tree	LM	10 × 10 × ~1.5	air-dry	2014
5-6	Wallis, Switzerland, living tree	LM, MWC	Circular Ø 40 × ~0.8	green	2016
Archaeological					
7	Lendbreen, Norway, permafrost, 1900 m.a.s.l.	LM	28.1 × 8.6 × 4.0	air-dry (uncontrolled)	1450 AD
8	Schnidejoch, Switzerland, ice patch, 2756 m a.s.l.	LM	21.8 × 4.5 × 3.3	air-dry (uncontrolled)	2800 BC
9	Moossee, Switzerland, waterlogged sediments, 521 m.a.s.l	LM	9 × 6 × ~0.6	Waterlogged (in distilled water and 4°C since 2011)	3800 to 4500 BC

RESULTS

Light microscopy

Optimisation of the preparation method

It would be desirable to develop a preparation method that allows the cell wall integrity to be investigated at the same time as the lumen betulin content, as both are expected to alter during degradation. However, betulin is opaque and obscures the cell walls and, moreover, being a crystalline resin, adheres to the blade and damages the following cell walls. Betulin was therefore removed from the sample with acetone, which is a solvent that is less effective with betulin than with ethanol but leads to limited swelling and softening of the cell structure (Gilberg 1986, 180). After brushing with acetone, the sample was sectioned

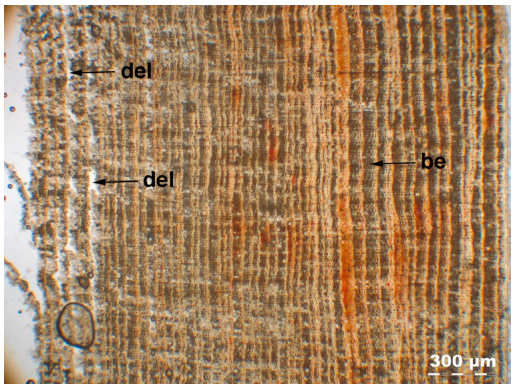

Figure 1. In the outer layers (left), fractures of thin cell walls cause delamination in the contemporary birch bark from Tomsk, Siberia. The remains of betulin and air (visible as the darker areas) are present. LM, 04× magnification, radial direction; *del* = delamination; *be* = betulin

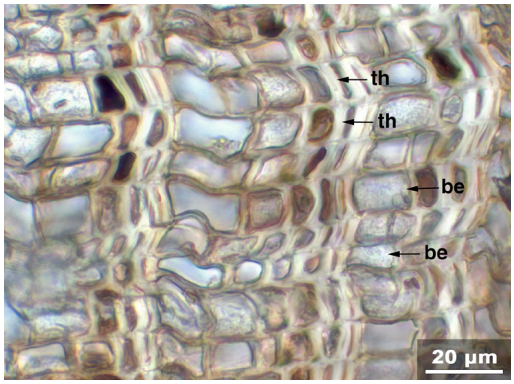

Figure 3. Well preserved cell structure with no signs of cell collapse in Neolithic Schnidejoch sample No. 8, ice patch. Thick cell walls look solid and betulin remains are visible. LM, 40× magnification, radial direction; *th* = thick-walled cells; *be* = betulin

with a sliding microtome and the sections were immersed in glycerol. Embedding in Technovit 7100 and paraffin was not successful as they do not penetrate the cell structure and provided only external stabilisation during microtome sectioning. Sectioning without embedding did not prevent occasional separation of the layers of thick- and thin-walled cells resulting in incomplete sections.

The lignified middle lamella could be highlighted by immersing the sample in a bleaching agent (sodium hypochlorite) followed by double staining with safranin O and astra blue but the bleaching removes birefringent components in the cell walls. Double staining without bleaching resulted in intensive staining of both thick-walled suberised cells and lumen content and did not allow lignin-containing portions to be highlighted, and therefore did not clarify the micromorphology.

Micromorphology of contemporary and archaeological birch bark

The reference sample from Tomsk, Siberia (Figure 1) shows the known composition of alternate layers of thick- and thin-walled cells. In the outer layers, cell fractures of the thin-walled cells can be seen. The fracture of the thin-walled cells along the junction between the cell types leads macroscopically to delamination. Fractures and cell deformations are observed in contemporary and archaeological samples retrieved from the ice environments of Schnidejoch and Lendbreen (Figure 2). No signs of cell collapse could be found in ice-logged archaeological samples despite having undergone uncontrolled air-drying (Figure 3). The thick-walled cells look solid and no signs of amorphous substance or erosion were detected. The integrity of the thin-walled cells could not be investigated within the magnification limits of LM.

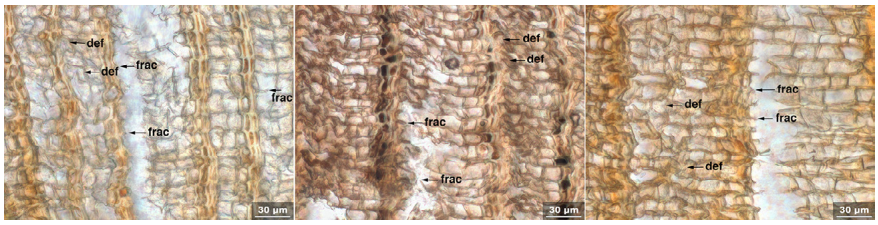

Figure 2. The fracture of thin-walled cells along the radial border of the two cell types and the deformation of broad cells is present in all investigated samples (left: reference birch bark, Tomsk; middle: Schnidejoch Neolithic ice patch, sample No. 8; right: Lendbreen, ice patch, sample No. 7. LM, 10× magnification, radial direction; *frac* = fracture; *def* = deformation)

The double birefringence of the thick-walled cells seems to decrease in archaeological samples, probably indicating a degradation of the secondary cell wall (Figure 4). As shown in Figure 5, air is present in the cell lumen of all samples, even in the waterlogged Neolithic samples from the Moossee. For the purpose of LM, air was removed by brushing the sections with ethanol or acetone.

MWC determination of contemporary samples

The aim of determining MWC was twofold: to get information on the variability of unaged material and to verify if the cell structure can be saturated with water.

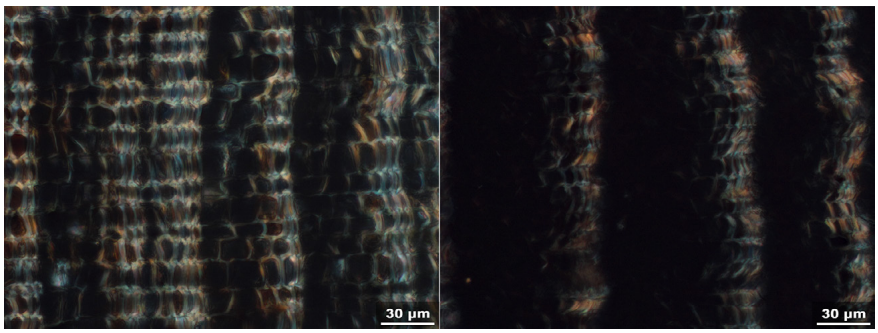

Figure 4. Left picture of reference material from Tomsk shows a high birefringence of the secondary cell wall in thick-walled cells and pronounced birefringence in thin-walled cells, while the Neolithic ice-patch sample No. 8 from Schnidejoch, Switzerland, lost all birefringence in the thin-walled cells. Thickness of section: 12 µm; PL, magnification 40×, radial direction

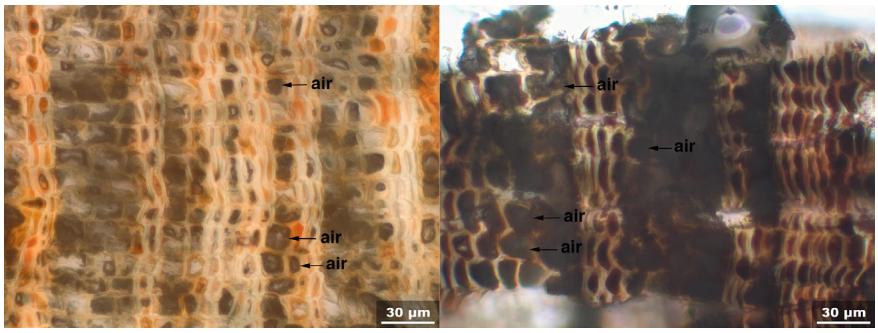

Figure 5. Air visible as a dark area is present in both the contemporary birch bark from Tomsk (left) and the waterlogged Neolithic birch bark from Moossee, sample No. 9 (right). LM, magnification 40×, radial direction

While the determination of the MWC on three samples of contemporary Siberian birch bark (sample No. 1–3) led to an average value of 25% ± 1%, the same measurement on contemporary Swiss samples (sample No. 5, 6) harvested at the same time from the same area of a trunk led to the values of 42% and 73%. This large difference may be related to the penetration of water among delaminated layers, to different extents of porous lenticels and to the presence of structural discontinuities.

Despite bubbles arising from the samples during the first stages of air evacuation, the LM investigation showed that, at all stages, all samples had a considerable amount of air in the cell structure, which only decreased slightly with air evacuation. As it was not possible to fill the cavities with water, the measured values do not actually correspond to the maximum water content and are not a measure of the void volume of birch bark.

DISCUSSION

LM is an appropriate method to investigate cell wall deformations and cell wall fractures in archaeological birch bark. However, these deformations are also present in contemporary materials and must be attributed to the formation process of the bark. Phellem is a dead tissue which cannot expand as the stem grows. This results in tangential tensile strain and leads to breakage of the thin cell walls and, macroscopically, to delamination. Deformation (corrugation) of cell walls is caused by radial pressure that arises from the formation of new cork layers. As a consequence, cell wall deformations and cell wall fractures are not attributes that can be

used to classify degradation. There are indications that ageing leads to a reduction in the birefringence of the secondary cell walls. More research is necessary to establish if the loss of birefringence is a valid microscopic attribute of degradation.

The cell wall structure of thick-walled cells in archaeological birch bark from the ice patches showed no signs of alteration or cell wall collapse. The absence of cell wall collapse, together with the presence of air within the cells in all samples, even in Neolithic waterlogged samples and in contemporary samples immersed in water and subjected to prolonged air evacuation, shows that, on the one hand, there is no increase in void volume with ageing and, on the other, that the voids cannot be filled with water anyway. MWC is not therefore an appropriate attribute to describe the degradation of birch bark.

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