The interaction of water with archeological birch bark and its effects on swelling, shrinking and deformations

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Abstract

In this paper we present results of water sorption of archaeological, ethnographical and contemporary birch bark and of the water-induced size and shape changes during humidification and drying. The analysis revealed that the equilibrium moisture content is higher if lenticels or inner bark are present and that the burial context influences the sorption behavior: the better the preservation condition of the archaeological birch bark, the lower the equilibrium moisture content. Compared to other organic materials like wood, the water uptake and the related swelling of outer birch bark is modest. This can be attributed to the cell structure and composition: outer birch bark is composed of closed cells made to a large extent of hydrophobic components (suberin, lignin). We show that warping of the bark takes place at high moisture content. This deformation is related to the plasticization effect of water and to the release of the built-in tension related to the stretching of the cells during the tree's growth. Our results provide a first guidance to conservators in the decision of water treatments on birch bark objects.

Introduction

Birch bark is a natural water-repellant organic material used for containers, fishing equipment and mats both in Neolithic times and nowadays by indigenous and hunter-gatherer populations.

Conservation treatments for birch bark objects involve water in a number of cases. Objects may be humidified in order to soften them during re-shaping. Craftspeople use mainly warm liquid water [1, 2] while conservators use mainly water vapour [3-6]. They might also be frozen for short-term preservation or they might be dried for exhibition and long-term preservation.

These treatments are known to possibly cause in archaeological wooden objects shrinkage and distortions and therefore there is a general hesitation in performing them on archaeological birch bark. How far these objects might swell, shrink and deform is expected to depend on the amount of water absorbed by the bark and on its preservation condition. The quantification of birch bark's sorption behavior and of the connected swelling and deformation is necessary therefore to provide guidance to conservators in the decision of water treatments.

The sorption isotherm of birch bark has been measured on freshly harvested samples but never on archaeological samples and the swelling and shrinking of outer birch bark during wetting and drying has never been published. In this article we fill this gap by presenting novel measurements of the sorption isotherm of birch bark on a number of archaeological and ethnographic samples and at different temperature. We also estimate the swelling and shrinkage during absorption and desorption processes and further deformations connected to an air-drying at room temperature.

Based on these results we provide recommendations for the performance of water treatments and for the relative humidity ranges for long-term preservation.

The material birch bark: macroscopic and microscopic structure and chemical composition

The portion of birch bark used to manufacture objects is the outer bark, in botany called the phellem layer. The outer bark prevents the birch tree from transpiration, it isolates it from heat, sun-radiation and cold [7, 432] and protects it from the penetration of parasites [8, 85f]. Both chemical composition and microscopic structure determine moisture absorption properties.

The phellem is made by cells formed each year from May to August [9, 27] by a thin layer of active cells, the phellogen or cork cambium, present between the inner and the outer bark. These cells are organized in bands of thin-walled cells and thick-walled cells that differ in the filling material of the cell lumen (Figure 1). Both thick-walled and thin-walled cell walls are composed of suberin, a biopolymer made of polyaliphatic and polyphenolic parts connected by glycerol via ester bonds. As consequence of the suberization of the cell walls these become impermeable and the cells die. Suberin makes 36.2 wt% of the phellem total composition and together with the closed structure of the cells is responsible for the water low permeability of the phellem [10]. Lignin accounts for 14.3 wt% of the phellem composition and is found in the middle lamella and between the suberin molecules in the secondary cell walls [11, 86f]. Polysaccharides account for 10.3 wt% of the phellem composition while extractives accounts for the remaining 32.2 wt% and are located in the cell lumen [12]. In particular the thin-walled cells are filled with betulin, a triterpene, while the thick-walled cells are filled with phenolic components.

The number of layers in each band depends on the phellem age and on the specie. The freshly formed phellem cells are pushed against the existing layers and, as the tree circumference grows, migrate outwards, stretch in tangential direction and compress in radial direction [13] and eventually come off the tree. Both phellem thickness [14, 209] and stiffness [15, 468f] increase with age: the thickest outer bark can be found on the bottom of a tree. When manufacturing objects, a portion of the white outlying, oldest, more brittle and more permeable to water and oxygen layers [16, 351] is usually removed.

Gas and water exchange between the internal tissues of the stem and the environment is allowed by the presence of pores in the phellem called lenticels that characterize the birch bark surface. Lenticels are fanned out bands of cell layers made of continuous bands of thick-walled cells and disrupted bands of thin-walled cells.

The phellem detaches easily in spring, the typical season when birch bark is harvested. If the bark is harvested in winter a part of the phloem, the layers of living cells internal to the phellogen, might be retained and used to manufacture objects with decorations carved in the inner bark surface. The phloem and the phellem differ radically in function, microscopic structure and chemical composition. The function of the phloem is to transport sugars from the leaves to the roots of the plant. It is produced not by the phellogen but by the vascular cambium outwards. The vascular cambium produces inwards the secondary xylem, in plain language the wood cells. The phloem is a complex tissue consisting of sieve tubes, open cells dedicated to the transport of nutrients, phloem parenchyma, sclerenchyma and rays parenchyma [17]. Parenchyma are living thin-walled cells of various functions, sclerenchyma are dead cells with a thick secondary lignified wall and support function and rays are the continuation of rays of

the xylem. Polysaccharides like cellulose constitute 43 wt% of the phloem chemical composition, lignin the 32.2 wt%, suberin the 13.2 wt% and various extractives the 8.1 wt% [12].

Interaction of water with birch bark

Different authors [18-23] investigated the sorption behavior of freshly harvested birch bark, mostly in studies on the influence of the addition of bark material to the properties of wooden particleboards.

Samples where the phloem is still attached to the outer bark are characterized by a high hygroscopicity, similar to wood [21] while samples where only the phellem is retained are characterized by a four times smaller moisture content that has little dependency on the specific birch specie (Figure 2). We report here only the adsorption isotherm as the authors reporting desorption results [19] did not let the samples saturate at 100% RH, measuring therefore scanning isotherms whose hysteresis cannot be univocally interpreted [24]. The difference in sorption behavior between samples with or without phloem can be explained by the fact that the secondary phloem is formed structurally by open cells, the sieves tubes, and chemically mostly by polysaccharides that are hygroscopic due to the large number of hydroxyl and carboxyl polar groups that can form hydrogen bonds with water molecules. The phellem on the contrary is formed by closed cells and mostly by suberin, a lipophilic biopolymer.

Kajita [23] measured the sorption isotherm at two temperatures, 20°C and 30 °C, and confirmed the well-known slight decrease of the equilibrium moisture content by increasing temperature observed in hygroscopic materials. Holmberg et al. [19] measured scanning isotherms in absorption and desorption for *Betula Papyrifera* samples. The time needed to reach equilibrium at each step of the sorption isotherm depends on the sample thickness, preparation method and presence of ventilation in the surrounding environment. Holmberg et al. [19] showed that for cuboid samples of 3 mm side in a ventilated environment it is of the order of 8 hours where half of the weight loss is attained in the first 30 minutes.

An understanding of which component in the phellem absorbs water is provided by the study of Schönherr and Ziegler [16, 387f] on samples of *Betula Pendula Roth.* The samples were infiltrated with water containing as indicator silver nitrate that was subsequently precipitated with hydrochloric acid. Electron microscopy revealed silver in the middle lamella and in the primary but not in the secondary, suberized, cell wall. It was significantly more concentrated in the radial middle lamellae than in the tangential ones. Further it was found in the lenticels, which have intercellular cavities in the tangential direction. This shows that the radial middle lamella and the lenticels are the components that mostly absorb water and build the pathway for the diffusion of water in the phellem.

The sorption isotherm is expected to increase therefore if the phloem is retained in the sample, if the sample contains lenticels, if the amount of cavities increases as it occurs in brittle degraded samples and by decreasing temperatures.

The adsorption and desorption of water is expected to cause swelling and shrinking of birch bark. While Gilberg [25] found no radial and tangential swelling on previously dried contemporary microtome sections

of outer birch bark exposed for 24 h to water vapor, Groh et al. [18, 799] detected 4 % swelling at 100 % RH of the surface of round birch phellem samples. Bhat [14] investigated the shrinkage of the inner and outer bark together and showed that it is anisotropic, being higher in radial direction then in tangential direction. The same anisotropic behavior is documented for Douglas fir cork [26, 98] and oak cork [11, 198]. The high radial swelling is attributed by both authors to the unfolding of corrugated lateral walls of thin-walled cells taking place upon moisture absorption. The stretching of the radial cell walls is retained also upon drying while further absorption of water vapor or liquid water causes a radial expansion of much smaller magnitude [27]. No data are available on swelling and shrinkage of outer birch bark neither for contemporary nor for archaeological and ethnographical material.

Beside swelling and shrinking water absorption may lead in birch bark to the warping of the bark with the outer side in. Water acts as a plasticizer allowing the phellem outer cells, that in the tree have been stretched tangentially to accommodate the growth of the stem and the creation of new cork layers, to return to their original dimension. The tangential length of the outer phellem cells is shorter than the tangential length of the inner phellem cells, as these have been produced when the stem had a larger diameter. The release of the tension through moisture plasticization cause a contraction of the outer layer and therefore a warping or rolling with the outer side of the bark inwards [13]. Deformations can also lead to delaminations related to failures in the thin-walled cell layers that are intrinsically weaker than the thick-walled cell layers [16, 348, 28, 251]. To avoid such deformations conservators may block the artefact in the desired shape using specially made capsules during both humidification and drying.

A clearer understanding of the swelling, shrinkage and of the deformations of birch bark upon moisture absorption and desorption can support conservators in their choices of conservation procedures.

Experimental

In order to get a better understanding of the risks connected with the drying or humidification of birch bark artefacts the interaction of water with both contemporary and archaeological birch bark was examined. Three types of experiments were performed: 1. Scanning sorption and desorption isotherms at different temperatures; 2. Measurement of swelling and shrinkage induced by water and observation of the connected decay patterns (deformation, cracking, delamination) 3. Simultaneous measurement of deformation by time-lapse photography and mass loss during water desorption.

Sample description and preparation

Different archaeological and contemporary birch bark materials were used to assess the water induced changes to outer birch bark.

Table 1 reports the characteristics of the materials and the experiments performed with them.

All materials except of C and B consisted only of the phellem layer. Material C has remains of the phloem visible as a brown-red layer. This was used to create a flower décor pattern, found typically on artefacts

from North America and Siberia. The phellogen and phloem stays attached to the phellem layer if the bark is harvested in Autumn or Winter. Material B was harvested from Neolithic waterlogged birch trunks found in the Burgäschisee. After vertically sectioning the bark on the trunk, this could carefully be detached and, contrary to what happens with contemporary bark, it remained in its original shape and with phloem, phellogen and phelloderm still adhering. The phloem was very brittle and could be removed mechanically by gently scraping it off with a wooden stick. The enzymatic treatment proposed by Orgell [29] and revised by Schreiber, Schönherr [30, 259f] and consisting in the immersion in a solution of a fungal pectinase and cellulase was tested but did not lead to the disintegration of the phelloderm within a time period of 4 and half weeks. Material B consists finally of phellem, phellogen and phelloderm and was stored in deionized water and kept refrigerated at 4 °C. The dry materials were stored in ambient conditions.

The sorption measurements were conducted on all materials by taking samples of size of about 15 × 5 mm. Measurement of swelling, shrinkage and deformation required rather large samples of length between 80 and 120 mm and was carried out on materials C, P and B. Samples taken from material P showed significant delamination, samples taken from material C and B had a bended shape, for C with the inner side outwards while for B with the inner side inwards.

Scanning isotherm at 25 °C

Scanning isotherms of contemporary and archaeological materials were measured with a multi-sample gravimetric vapor sorption analyser (SPSx-1µ Advance) from ProUmid. A drawing and description of the instrument has been published by Murr, Lackner [31]. Each type of material was analysed in duplicate samples. Before starting the sorption cycle the samples were dried over silica gel to 0% RH and reached a mass between 75 and 198 mg. Then the samples were exposed to increasing relative humidity from 0 to 95% RH at constant temperature of 25 °C and weighted every 15 min. If the rate of weight change was smaller than 0.01% in 80 minutes, the samples were considered in equilibrium with the relative humidity and the value of the relative humidity was changed. After reaching equilibrium with 95% the value of the relative humidity was decreased and the desorption cycle was recorded in order to gain information on the sorption hysteresis. The full sorption cycle took 600 h.

Scanning isotherm at 0 °C and − 20 °C

Adsorption isotherms at 0 °C and − 20 °C were measured on samples from material S. The measurements were performed at TH Wildau, Germany with a McBain-Bakr balance [32]. In this balance the sample holder is connected to a quartz spring within a quartz tube. The pressure in the tube is controlled by MKS Baratron pressure heads of high sensitivity in the range 10^{-5} to 10^{3} mbar. The elongation of the spring is measured with a KM6 cathetometer (workshops of the Soviet Academy of Sciences, Moscow) with an accuracy of ± 0.01 mm and a sensitivity of the spring of 0.04 mg/mm. Prior to the adsorption measurements the samples are equilibrated at room temperature in high vacuum atmosphere ($p < 10^{-5}$ mbar) for at least 12 hours and the dry mass is measured (accuracy ± 0.02 mg). The sample temperature is kept constant during the measurement by a thermostat (Lauda Ecoline RE107) with

fluctuations of ± 0.05 °C while the temperature of the spring is maintained at 30 °C to avoid temperature-related length changes. The equilibrium between the sample and the environment is reached when the pressure and the mass are constant for at least 30 minutes, which took about 24 hours.

Swelling and shrinking during humidification and drying

Percentage swelling and shrinkage in the three dimensions was calculated in reference to the dimension of the samples equilibrated at 50% and 97% RH. We have decided to use as a reference the 50% RH condition and not a dryer state as this is the most common condition in repositories. The dimensions in the swollen state were measured after the samples were equilibrated at 97% ± 3% RH for 28 days at 4 °C in a box containing a water saturated polyester fleece separated from the samples with a Tyvek® tissue. The dimensions at 50% RH was measured after the samples were equilibrated at 50% ± 3% RH for 8 days at 20 °C in air. For the waterlogged samples taken from material B only shrinkage measurements were performed.

Sample dimensions were measured with a digital caliper with an accuracy of ± 0.02 mm or, where not possible, by scanning the sample on a photocopy machine and analysing the images with the software ImageJ. For each type of sample material the measurements were performed on four samples and the average and standard deviation was calculated. The samples were also photographed from all sides to assess further changes like cracks and delamination.

Deformation and mass loss during desorption

The correlation between deformation during air drying and mass loss was investigated by time lapse photography of the samples placed on an electronic scale. A sample from material C, C_11, that was in good condition but initially bent as it was cut out from an object, was first conditioned to 97% RH at 4 °C for four weeks and then placed at ambient conditions (22 °C, 54% RH) on a Kern EG 600- C3 NM scale with an accuracy of 0.001 g and photographs of the cross section were taken with a Nikon DC 1500 for 26 hours in total.

Results And Discussion

Scanning isotherms

Scanning isotherms both in adsorption and desorption were measured for all samples confirming the well-known hysteresis effect, whereby the equilibrium moisture content attained through desorption experiment is higher than the one attained at the same relative humidity through adsorption. For the sake of simplicity the diagrams show only the adsorption isotherms. Data for the full scanning isotherms are in the supplementary materials (Figure S1, S2).

The influence of the presence of lenticels was investigated on samples from material WA, while the influence of the phloem on samples from material C (Fig. 3).

The equilibrium moisture content increases if the phloem is present on the outer bark, confirming the observations of Standke and Schneider [33]. This increase is related to the different structure and chemical composition of the phloem. The presence of the lenticels also increases the equilibrium moisture content, confirming the observations of Schönherr and Ziegler [16, 387f] on water deposition in the lenticels' intercellular cavities.

The influence of the burial environment and of the sample condition was investigated on samples from material B, Neolithic waterlogged, on samples from material S, Neolithic from ice fields and on samples from material P, Iron age from permafrost (Fig. 4). Each curve has been repeated on two samples and the difference among the resulting equilibrium moisture contents was in all cases less than 0.1 g/100 g. The cover, middle and lower part of the bow case are considered as three distinct materials as they differ in preservation condition and detailed manufacture characteristics. The bow case cover has been subjected to a harmful treatment with alcohol that extracted most of alcohol soluble components from the bark. The middle and the lower part of the bow case body are untreated, but the lower part is tapered and made of thicker bark.

These results show that the better the preservation condition of the archaeological birch bark, the lower the equilibrium moisture content. Brittleness, delaminations and waterlogging increase the number of bonding sites between water and birch bark.

The temperature dependence of the adsorption isotherm was investigated on two samples from the middle part and one sample from the lower part of material S, the Neolithic bow case from the Schnidejoch Pass (Fig. 5). While for one sample no temperature dependence was detected, the two remaining samples showed the usual increase of the equilibrium moisture content with decreasing temperature. In analogy to wood, based both on experimental studies and thermodynamic considerations [34–36] no freezing of bound water is expected at these subzero temperatures.

Swelling and shrinking during humidification and drying

Humidification led to swelling while drying to a shrinkage of the outer bark. Table 2 summarizes the results of the experiments.

Table 2
Swelling and shrinking values of sample C, P and B during humidification and drying.

Sample	Value	% swelling			% shrinkage		
		tangential	*longitudinal*	*radial*	*tangential*	*longitudinal*	*radial*
C	**av**	**0.2**	**1.4**	**2.2**	**0.3**	**2.1**	**4.8**
	SD	0.10	0.76	0.61	0.21	0.91	0.96
P	**av**	**2.2**	**1.8**	**-**	**0.8**	**0.7**	**-**
	SD	0.67	0.37	-	0.18	0.44	-
B	**av**	-	-	-	**1.85**	**3.35**	**15.35**
	SD	-	-	-	1.28	0.17	3.54

Swelling and shrinking data for the radial direction (thickness of the samples) could not be obtained for the P material as these samples were delaminated. The B material was waterlogged and therefore swelling could not be measured.

For all materials swelling and shrinking is higher in radial direction and generally small in longitudinal and negligible in tangential direction. The waterlogged material (B) has considerable shrinking especially in radial direction. This sample had also the highest mass loss (53.3 wt%), an indication that it sample contained liquid water in open void spaces.

The negligible dimensional changes in tangential and longitudinal direction of birch bark can be understood in analogy to wood. Wood is composed of long cells, the tracheids, oriented longitudinally. In this direction wood deformation is negligible. Birch bark is composed of elongated cells oriented tangentially (Fig. 1) and in this direction its deformations are indeed negligible.

The increased swelling and shrinking in radial direction can be explained by the unfolding of the radial cell walls as folded radial cell walls were detected by optical microscopy in birch bark (Fig. 6). This is in agreement with the observations in Douglas-Fir bark [26].

Deformation and mass loss during drying

While deformations and delaminations of the B and C samples took place both during water absorption and desorption, larger deformations were detected during the absorption process, accompanied by the warping of the bark towards the outer layers. These movements led to delamination and deepening of tangential cracks (Fig. 7, right and Figure S4, S6, S8 in the supplementary materials). The time lapse photography investigation allowed to monitor in details the deformation of one sample of the C material during desorption. Surface evaporation, revealed by a colour brightening of the surface, takes place in the first two minutes of the procedure. In the following twenty minutes of desorption, while the moisture

content decreases from 8.7–6.7%, the sample bent slightly in the direction of the inner bark and a crack formed (Fig. 8a). A more significant movement in the direction of the outer bark dominated the process in the following three hours up to a moisture content of 4.8%. After this time no movement was detected and the moisture content slowly decreased for about 24 hours to the value of 3.5% in equilibrium with the environmental 50% RH (Fig. 8b and video in the supplementary materials).

The desorption kinetic depends on the geometry of the sample, on the environmental RH and on the presence of ventilation. Nevertheless, it is possible to conclude that surface evaporation is a very fast process and that movements of the bark are possible at higher moisture content, in this case corresponding to relative humidities higher than 75%, a consequence of the fact that water is a flexibilization agent for birch bark. While we do not have an explanation for the inward warping of the bark, the outward and commonly observed warping is explained by the shortening of the stretched outer birch bark cells when they are plasticized at high humidity [13].

Conclusions And Implications For Conservation Measures

The equilibrium moisture content of archaeological birch bark is generally modest and the better the condition of the material, the lower the water uptake. Ice-preserved Neolithic birch bark from the Schnidejoch, Switzerland, has almost the same sorption properties as unaged bark. Degraded samples have higher moisture content at high humidities and a more pronounced hysteresis, in particular waterlogged Neolithic samples from the Burgäschisee, Switzerland, had the highest water sorption.

The sorption behaviour of birch bark does not depend on the specific birch specie, therefore there is no need in determining it when deciding on treatments for birch bark objects. When considering specific requirements for the storage of birch bark objects, these can be classified as mechanically stable and a broader humidity target range can be acceptable, provided it is lower than 75% RH. Long-term storage at sub-zero temperatures is acceptable for birch bark objects as long as they do not contain liquid water.

Water vapour can plasticize birch bark, despite the process is rather slow as water vapour needs to diffuse through the middle lamella. As birch bark gets flexible, it deforms towards the outside of the bark. The extent of deformation depends on the flexibility of the bark. Such deformations can lead, especially in brittle samples, to a tangential separation of the layers. Deformation and shrinkage take place also when air-drying the samples but to a minor extent. In order to prevent damages, both humidification and drying should be performed within a supporting capsule. Swelling and shrinkage is anisotropic and the highest shrinkage was found in the radial direction, corresponding to the thickness of the bark. These size changes do not have an impact on the appearance of the object. As a consequence, air-drying within a supporting capsule is feasible for all tested archaeological birch barks.

List Of Abbreviations

wt%: weight percent

mm: millimeter

°C: degree Celsius

mg: milligram

mbar: millibar

RH: relative humidity

g: gram

Declarations

Availability of data and materials

The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

Competing interests

The authors declare that they have no competing interests.

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Authors' contributions

JK and GDP conceived this study. JK prepared samples and performed the swelling and shrinking measurements and analyzed the deformation and mass loss during drying. GDP interpreted sorption isotherm and prepared graphs. JK interpreted swelling / shrinking and deformation and prepared drawings/images. JK and GDP wrote the paper.

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Note For Table 1

Due to technical limitations, Table 1 is only available as a download in the supplemental files section

Figures

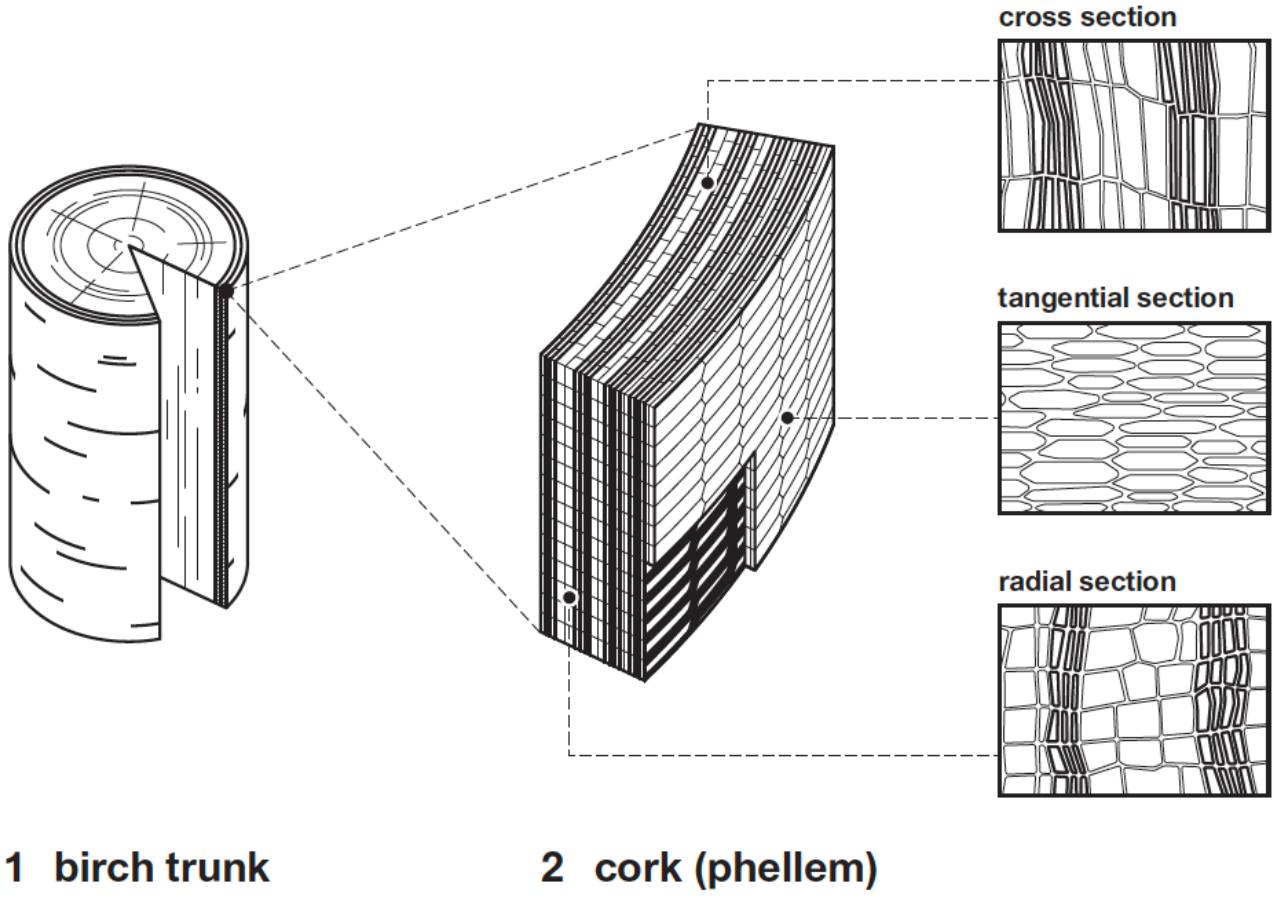

Figure 1

Illustration of the betula phellem from a macroscopic to a microscopic scale: left: location of outer bark (phellem) on birch trunk. Middle: microscopic illustration of phellem layer consisting of thin-walled cells and thick-walled tangential elongated cells. Right: Shape and size of the two cell-types in cross section, tangential and radial section.

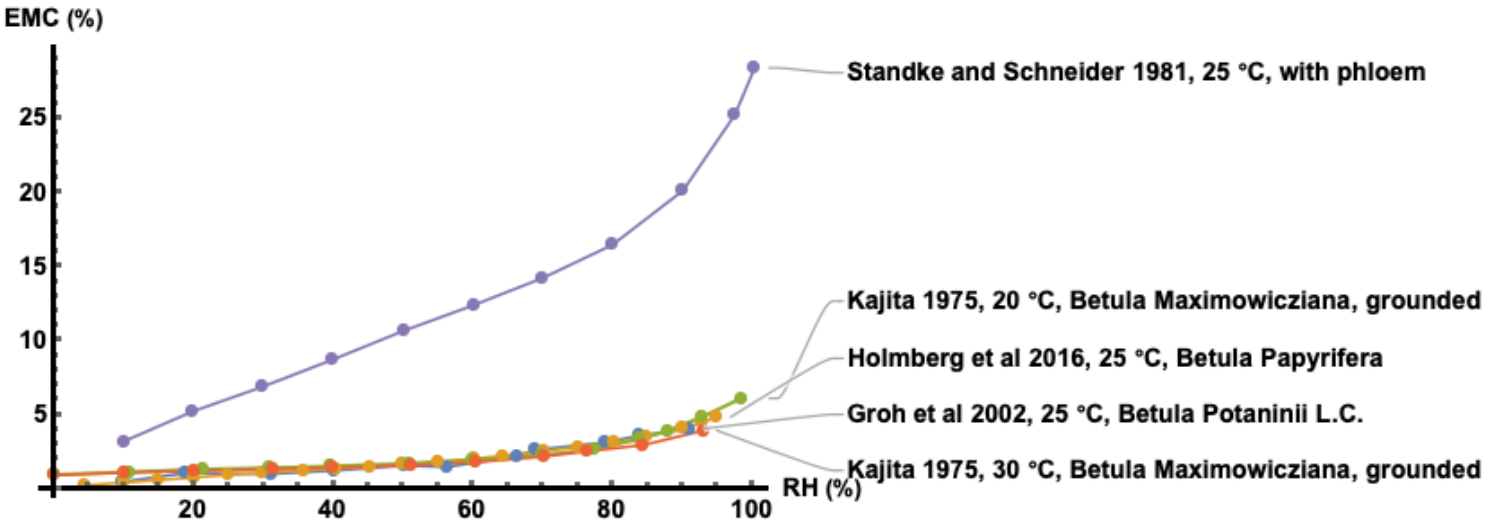

Figure 2

Sorption isotherms of different Betula barks. Data compiled from different sources quoted in the bibliography.

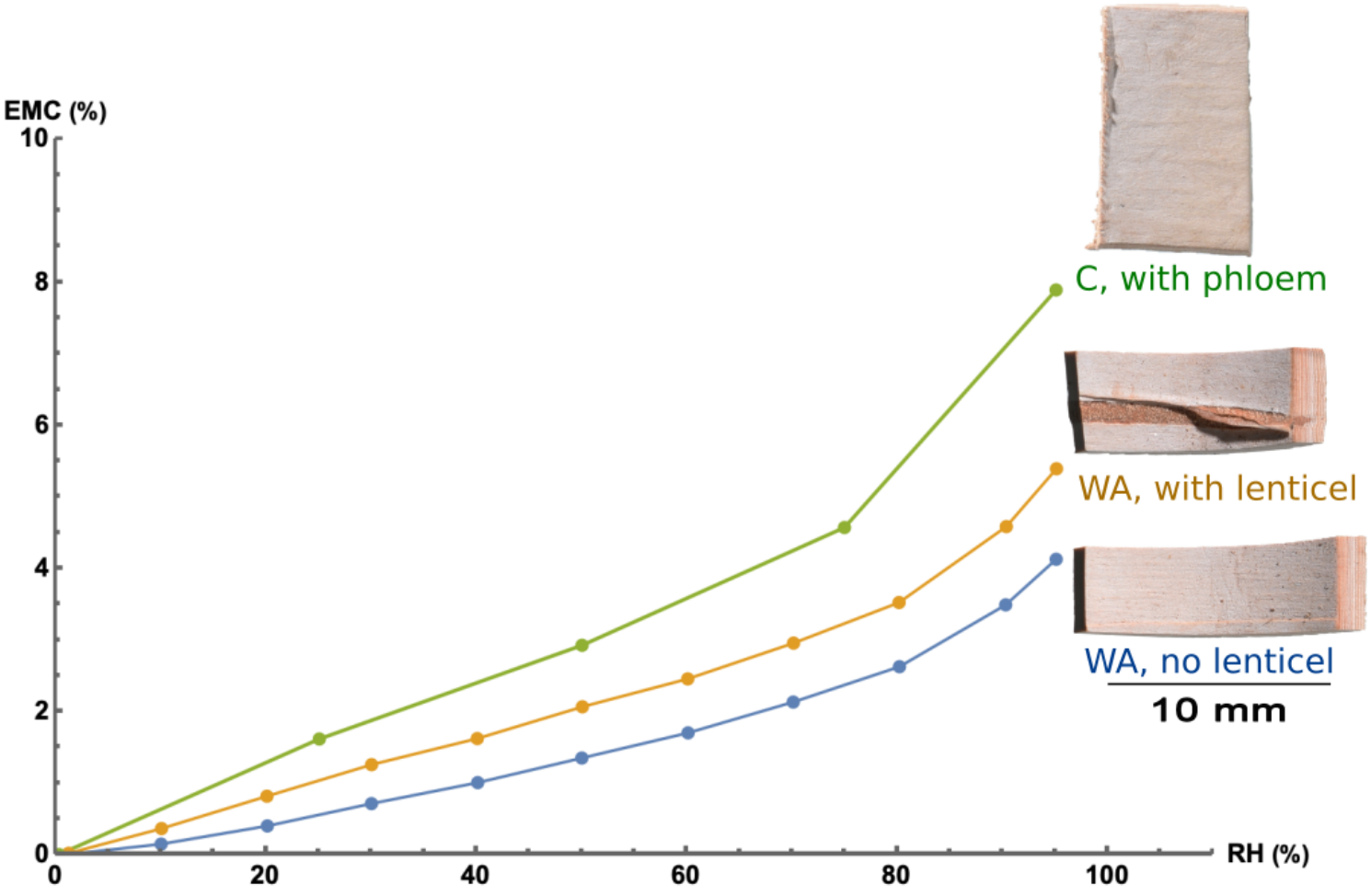

Figure 3

Sorption isotherms of contemporary samples, retaining the phloem and with or without lenticels.

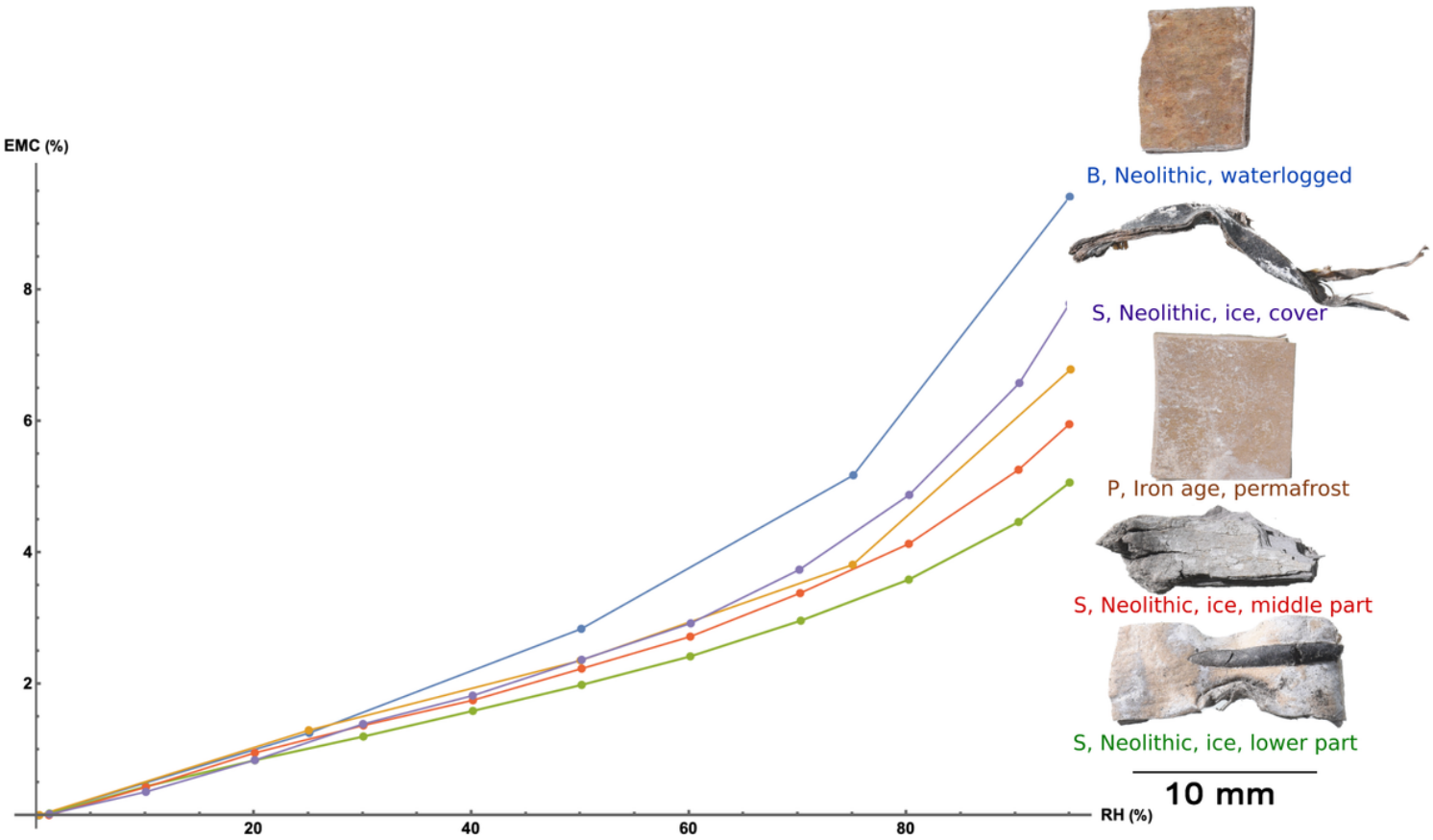

Figure 4

Sorption isotherms of archaeological birch barks excavated from different environments.

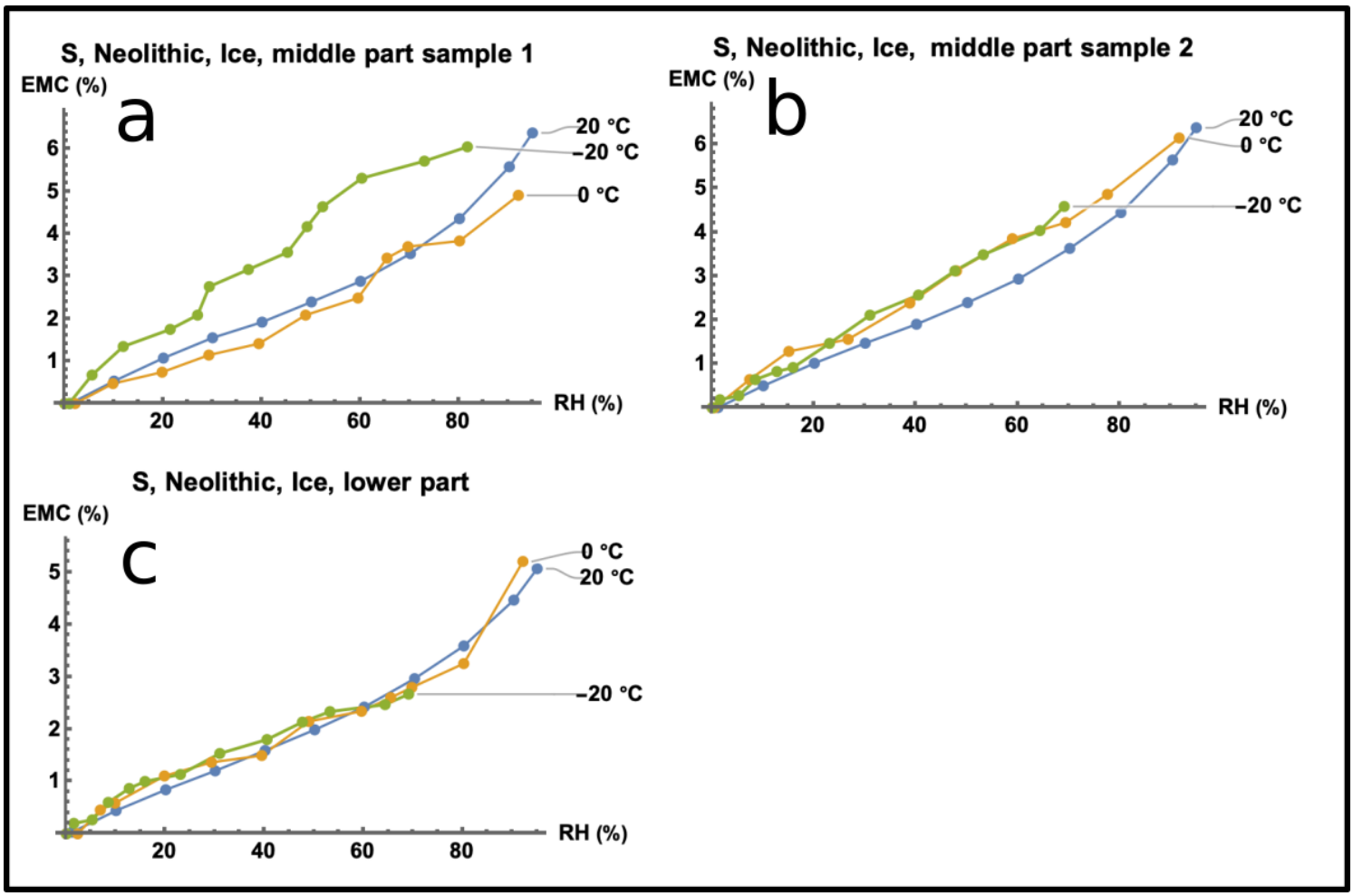

Figure 5

Temperature dependence of the sorption isotherm of three samples from the Neolithic Schnidejoch bow case.

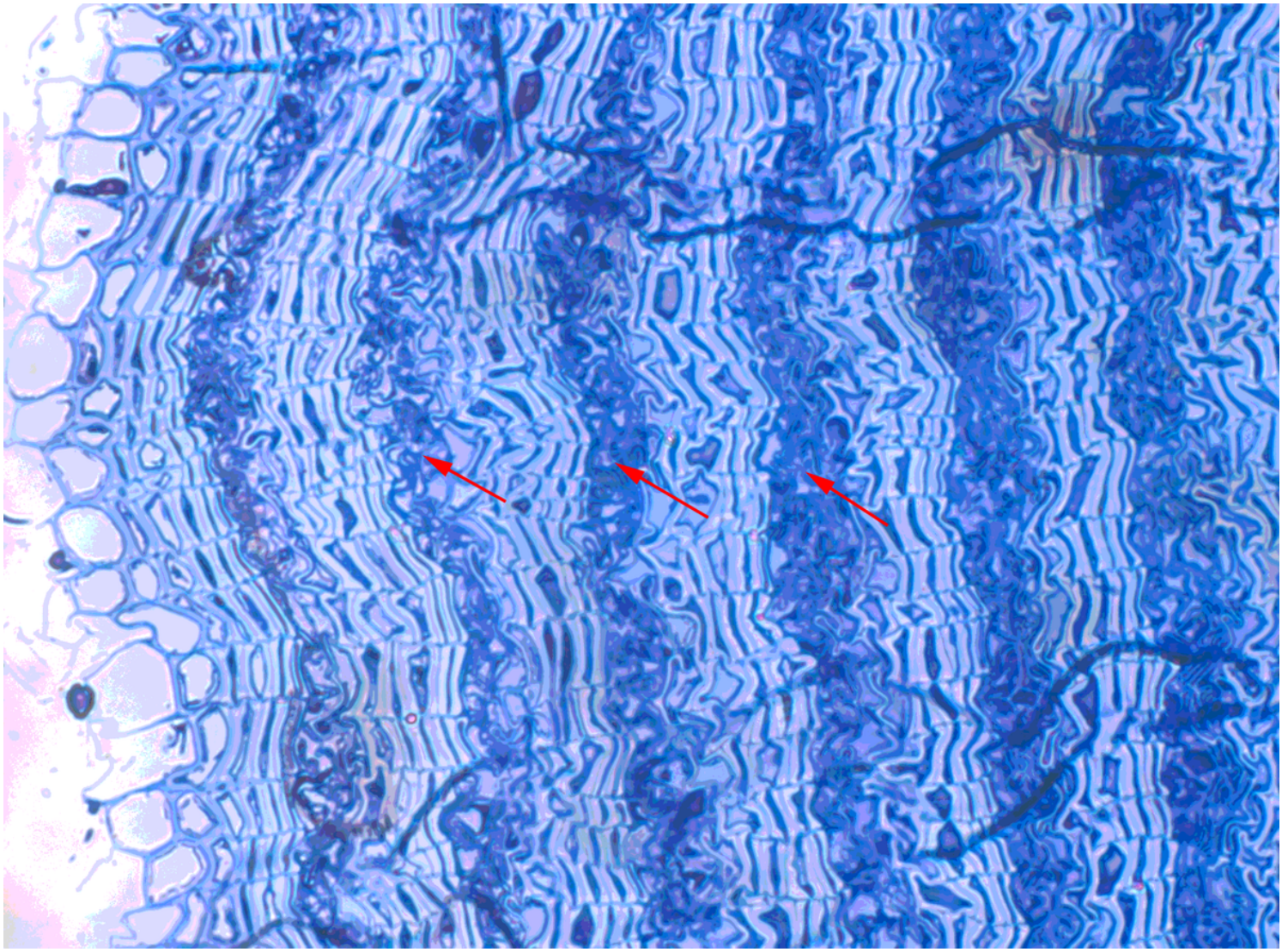

Figure 6

Radial section of contemporary birch bark LM 40x, stained with toluidine blue. The red arrows show the folding of outer thin-walled cells.

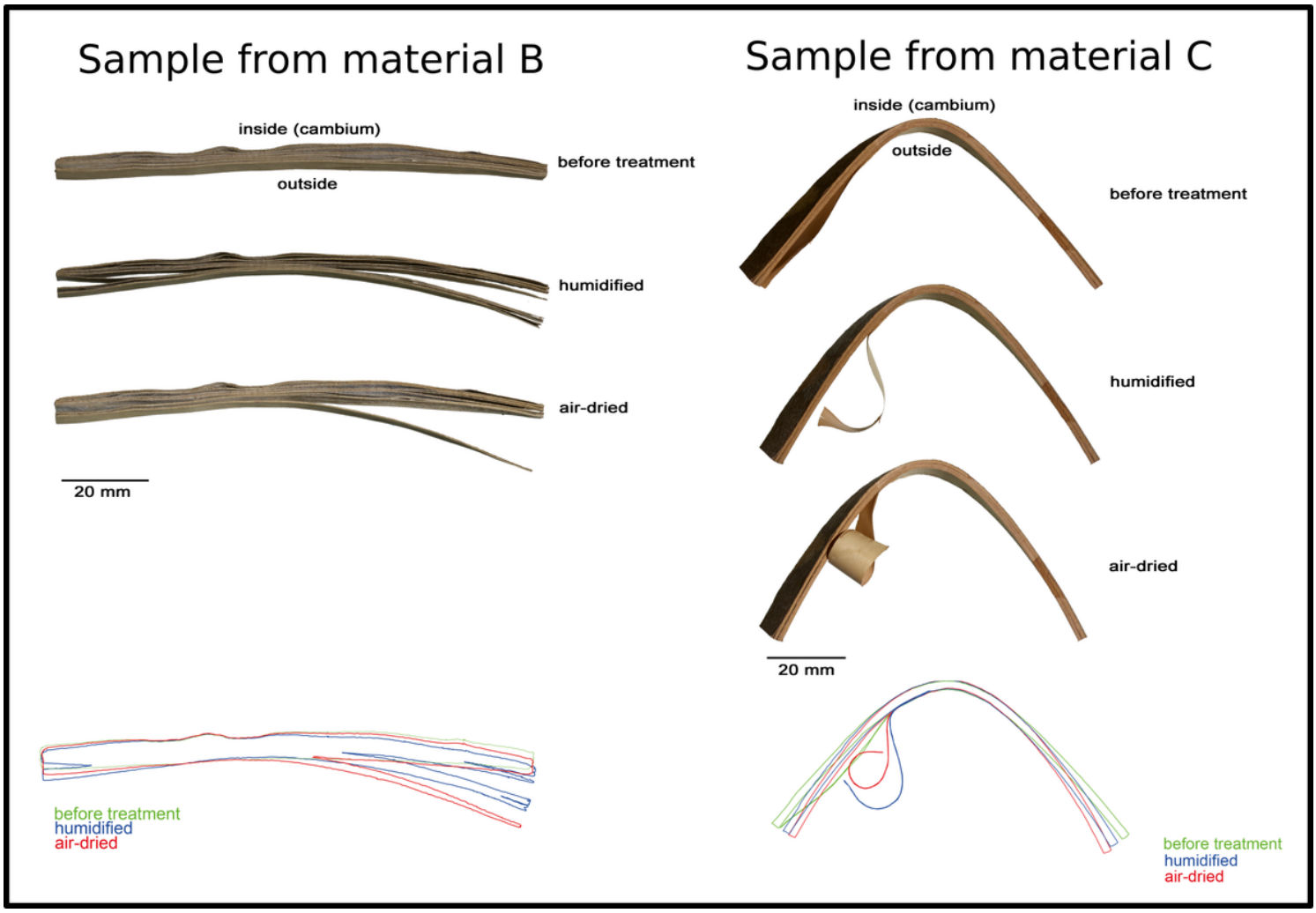

Figure 7

Above: Deformation and delamination during humidification and drying on samples from material B and C. Below: Superimposed contour illustrating the warping.

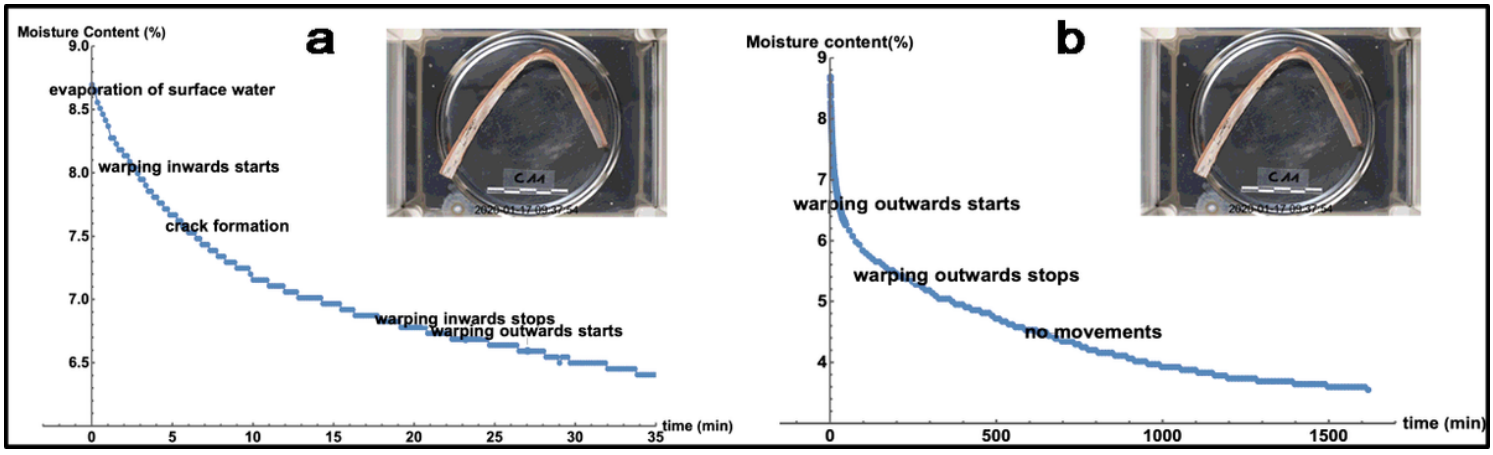

Figure 8

Mass loss and labelled deformations of a sample from material C.

Supplementary Files

This is a list of supplementary files associated with this preprint. Click to download.

- Table1.docx
- KluegldiPietro.mov
- KlueglDiPietroSupplementaryFigureS08.jpg
- KlueglDiPietroSupplementaryFigureS07.jpg
- KlueglDiPietroSupplementaryFigureS06.jpg
- KlueglDiPietroSupplementaryFigureS05.tif
- KlueglDiPietroSupplementaryFigureS04.tif
- KlueglDiPietroSupplementaryFigureS03.tif
- KlueglDiPietroSupplementaryFigureS02.png
- KlueglDiPietroSupplementaryFigureS01.png