#### G Model CULHER-3116; No. of Pages 9

### **ARTICLE IN PRESS**

Journal of Cultural Heritage xxx (2016) xxx-xxx



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#### Original article

# Study on dimensional stabilization of 12,500-year-old waterlogged subfossil Scots pine wood from the Koźmin Las site, Poland

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#### ARTICLE INFO

#### Article history: Received 20 March 2016 Accepted 22 August 2016 Available online xxx

Keywords: Waterlogged subfossil wood Scots pine Dimensional stabilization Polyethylene glycol Sugar alcohol Freeze-drying

#### ABSTRACT

The study examines the efficiency of selected conservation treatments proposed for dimensional stabilization of 12,500-year-old waterlogged subfossil Scots pine (Pinus sylvestris L.) tree trunks found at the Koźmin Las site, Poland. The research was done on 198 samples drawn from various parts of the same trunk. Experimental material was characterized on the basis of annual ring width, percentage of latewood, maximum moisture content, density at maximum moisture content, basic density, and wood porosity. Waterlogged wood samples were pre-treated with aqueous solutions of polyethylene glycol (PEG) 300, mixtures of PEG 300 and PEG 4000, a mixture of lactitol and trehalose or a mixture of mannitol and trehalose (nine variants of impregnation) and then freeze-dried or air-dried. Dimensional changes in the wood samples were measured after seasoning to reach equilibrium moisture content at 50% relative humidity and 20 °C. In the majority of the tested conservation treatments, tangential and radial shrinkages or tangential and radial swelling of wood did not exceed 1%. However, differences in dimensional changes depended on the conservation method used and sampling location. Regardless of the state of wood preservation and macroscopic structure features, the best dimensional stability, evaluated on the basis of average anti-shrink efficiency indices, was observed in wood pre-treated with aqueous solutions of 10% PEG 300 + 5% PEG 4000, 10% PEG 300 + 10% PEG 4000 or 10% lactitol/trehalose mixture, and then freeze-dried.

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#### 1. Introduction

The Koźmin Las (Koźmin Forest) site (N 52°04′51″, E 18°40′03″, 97.5 m a.s.l.) is situated in the Central Poland village of Koźmin, in the mid-Warta River valley of the Koło Basin. It was discovered in an area of Miocene lignite exploitation by the Adamów Lignite Mine. A study at the Koźmin Las site was undertaken in 2010 and 2011 in 160 square meters open test pit. The period of organic deposits accumulations with the forest remains is dated from 13,000 to 11,600/11,250 cal. BP [1] and their associated inorganic sediments were deposited at the very end of the Younger Dryas [2] or in the Early Holocene [3]. Over 300 fragments of stumps, collapsed trunks and branches, mostly of Scots pine and birch wood were found [1]. The lengths of trunks were up to 7 m (usually 2–3 m) and their diameters seldom exceeded 0.2 m. The

http://dx.doi.org/10.1016/j.culher.2016.08.002

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Late Weichselian forest remains were preserved by a thick (up to 2.5 m) cover of silty organic deposits and silty sandy overbank alluvium and a high ground water table. Dendrochronological analyses show the forest consisted predominantly of pines of an average age of 50–70 years and probably existed no longer than 150 years. The time of forest development has been estimated from 13,000/12,900 to 12,700/12,600 cal. BP [1]. Similar pine-birch forest from the Younger Dryas, but in a poorer state of preservation, was excavated on the Spree River floodplain in the Cottbus area (Eastern Germany) [4,5]. In Central Europe, Holocene subfossil tree remains are more common [e.g. 6–9].

The state of preservation of unearthed archaeological or subfossil wood depends mainly on deposit conditions [10]. In a wet environment with limited oxygen content, organic material is decomposed by a limited number of destructive factors, predominantly bacteria and soft rot fungi [e.g. 11–13]. Archaeological finds show that wood can survive in wet environments for several thousand years. Nevertheless, the above-mentioned destructive factors can cause minor or even major changes in physical, chemical and mechanical properties of wood [e.g. 14–17]. Degraded, waterlogged

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wood can shrink more than fresh wood [14]. Wood shrinkage may lead to deformation and/or cracking of an object during drying. The extent of the above damages is strictly related to the degree of wood decomposition but also to differing shrinkage of different wood zones [18].

In order to reduce dimensional changes, degraded waterlogged wood requires an appropriate conservation treatment. A part of the water filling the cell walls and lumens is replaced with an appropriate chemical compound, which reduces wood contraction and deformation on drying and strengthens the object being conserved [19]. The main chemicals used for dimensional stabilization of archaeological wood are polyethylene glycols (PEGs) [20,21]. Mannitol and lactitol, with the addition of 10% trehalose (mannitol + 10% trehalose, lactitol + 10% trehalose), are other chemicals applied for such treatment [22–24]. Drying may also have substantial influence on wood conservation. Taking into consideration dimensional changes in wood, freeze-drying seems to be one of the most effective methods. It also allows for achieving satisfactory results with lower uptake of an impregnant as compared to air-dried wood.

The choice of wood conservation method is based on the evaluation of wood degradation advancement. In turn, a state of wood preservation is evaluated on the basis of the degree of cell wall decomposition and some wood physical properties and chemical composition [e.g. 15,17,25,26]. In spite of the fact that conservators perceive the necessity for providing a complex evaluation of wood decay, in practice they mainly use physical properties of wood related to its weight and volume (e.g. maximum moisture content, basic density) for that purpose. Alternatively, there are no procedures enabling selection of the method of conservation on the basis of chemical composition or microscopic analysis of wood.

The wood degradation process often proceeds in an irregular way. The appearance of various zones of degradation is known particularly for oakwood and/or large-size objects. Multi-quality wood requires different conservation treatment than homogenously decayed wood [20,21,27].

However, wood properties used for evaluation of wood degradation are not related only to the advancement of destructive processes. They also depend on the examined wood zone (softwood and heartwood, juvenile and mature wood, normal and reaction wood) as well as annual ring width and percentage of latewood (particularly in the case of ring-porous and coniferous species). Dimensions of anatomic elements and annual ring width as well as the percentage of latewood in archaeological and subfossil wood were examined by Scheiber and Wagenführ [28], Horský and Reinprecht [6], and Kokociński [8].

Many previous studies on subfossil wood were done on a well-preserved oak trunk material found in Central Europe. The research, apart from determining wood's macroscopic features, also included physical and mechanical properties and chemical composition of wood [e.g. 6,8,9,28–31]. Unfortunately, little attention has been paid to the possibilities of its conservation.

Subfossil pinewood is more rarely discovered and studied than oakwood and literature on the conservation of subfossil pinewood is limited. Meanwhile, research on much younger archaeological material has proved, regardless of wood decay advancements, that softwoods require quite different chemical solutions and conservation processes than hardwoods [32–34].

Research on 12,500-year-old waterlogged subfossil pinewood of Koźmin was performed by Fejfer et al. [35]. It sought to determine the dimensional stability of wood treated with polyethylene glycols and then air-dried. However, that study did not consider differences in wood properties from various zones of the trunk and the presence of reaction (compression) wood. Differences in structure and state of preservation of individual wood layers can lead to an increase in tensions during drying. That increase should be taken

into consideration particularly in the case of round wood where reaction wood develops.

#### 2. Research aims

This study seeks to determine the state of preservation of Koźmin pinewood in various areas of the unearthed trunk (evaluated on the basis of selected physical properties with regard to basic features of macroscopic structure of wood) as well as determining the effectiveness of dimensional stabilization of examined material pre-treated with polyethylene glycols or a mixture of selected sugar alcohols and trehalose prior to freeze-drying.

#### 3. Material and methods

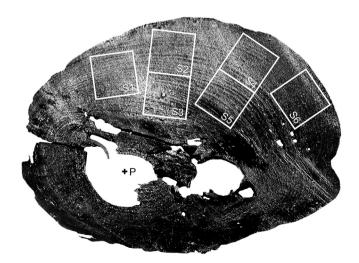
#### 3.1. Wood

The experimental research was performed on waterlogged subfossil wood of the Scots pine (*Pinus sylvestris* L.) excavated from the Koźmin Las site. The wood was dated using the radiocarbon method at ca. 12,500 years ago. Samples for the research were drawn from a trunk section with a length of 120 cm and a diameter of 12–15 cm (asymmetric cross-section, eccentric pith).

#### 3.2. Samples

The trunk fragment was cut into 10-mm-thick discs (D) and marked from 1 to 88 (D1–D88). Three discs were used for determining basic parameters of the macrostructure (annual ring width and percentage of latewood) and selected physical properties of wood related to its weight and volume (maximum moisture content, density, and porosity) while 30 discs were used for determining efficiency of the conservation treatments. Six samples (S1–S6) with dimensions  $20 \times 20 \times 10$  mm (T  $\times$  R  $\times$  L) were cut from each of 33 discs. The samples from individual discs were taken from the same trunk cross-section areas (Fig. 1).

Before measurements, wet samples were impregnated with water to reach maximum saturation. The samples, immersed in water, were put into the vacuum chamber. Air pressure was reduced to 50 hPa and retained for 30 minutes. Next, the samples were kept immersed in water for two hours at atmospheric pressure. Impregnation of wood according to the above-mentioned procedure was repeated for five times.



 $\begin{tabular}{ll} \textbf{Fig. 1.} Samples distribution on the cross-section of the Koźmin Scots pine trunk (+ P indicates pith). \end{tabular}$ 

Please cite this article in press as: L. Babiński, et al., Study on dimensional stabilization of 12,500-year-old waterlogged subfossil Scots pine wood from the Koźmin Las site, Poland, Journal of Cultural Heritage (2016), http://dx.doi.org/10.1016/j.culher.2016.08.002

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