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PROPERTIES AND DIMENSIONAL STABILITY OF 12 500-YEAR-OLD SUBFOSSIL PINE WOOD

At the Koźmin Las site, in Central Poland, in the middle section of the Warta River valley, a series of well-preserved tree trunks and in situ stumps, as well as organic deposits, have been found. The tree remains are dated back to the period between 13,000–12,600 cal BP, i.e. to the Alleröd/Younger Dryas transition. The forest consisted predominantly of pines (Pinus sylvestris L.) of a maximum age of approx. 140 years and an average age of 68 years, and the river valley floor was overgrown. The forest was destroyed ca. 12,600 cal BP by deteriorating hydrological conditions or a sudden catastrophic event. The aim of the study was to assess the degree of degradation in terms of selected macroscopic, physical and chemical properties of a subfossil pine log. On this basis, a conservation process was developed, using aqueous solutions of polyethylene glycols (PEG) with varying concentrations of low- and high-molecular polymers. Treated and dried samples were compared in terms of their tangential and radial dimensional stability, as well as their hygroscopic properties.

Keywords: waterlogged wood, Scots pine (*Pinus sylvestris* L.), chemical composition, physical properties, conservation, Uniejów Basin, Central Poland, Alleröd/Younger Dryas transition

Introduction

The discovery of well-preserved pine wood objects several thousand years or tens of thousands of years old is relatively rare. More frequent discoveries are those

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of subfossil tree remains in deposits from the Holocene, including the early Holocene [Kalicki, Krąpiec 1995; Kalicki 2006]. The research presented is the first dendrological study of tree remains of the Late Weichselian age in Central Poland [Dzieduszyńska et al. 2014]. The *in situ* pine-birch forest, although in a bad state of preservation, growing during the final centuries of the Younger Dryas on the floodplain of the Spree River, was excavated in the lignite area of Cottbus in eastern Germany [Friedrich et al. 1999; Spurk et al. 1999].

The subject of this interdisciplinary study undertaken by scientists from the Department of Geomorphology and Palaeogeography of the University of Łódź is the horizon of a subfossil riparian forest buried by alluvial (flood) sediments (fig. 1). Apart from geochronological studies (radiocarbon dating of the tree remains and the enclosing organic material, and optically stimulated luminescence dating of the inorganic deposits overlying the organic series) the following analyses were carried out: dendrochronological analyses, analyses of pollen and plant macrofossils, palaeopedological analyses and analyses of sedimentological properties and grain size composition. The study undertaken aimed to reconstruct the environmental conditions in the river valley during the time of rapid global natural climate changes at the end of the Late Weichselian (14,700-11,550 cal. BP), based on a reconstruction of the evolution of the forest ecosystem [Kittel et al. 2012; Dzieduszyńska et al. 2012, 2014].

Fig. 1. Tree remains visible within successive levels of the test pit at the Koźmin Las site (Photo by J. Petera-Zganiacz, 2011)

Wood may be preserved under the surface of the ground for thousands of years thanks to the advantageous environmental parameters in which it was deposited.

The most important of these include for example, a high and stable ground water level, no oxygen access, a low redox potential and an optimal pH. Despite favourable conditions, the lignocellulose material is degraded over the course of time. However, it is not the length of time, but the intensity of the destructive processes occurring in the environment which determines the volume of degradation of the wood remains. Environmental pressure causes irreversible changes in wood at each level of its structure. Progressive wood degradation occurs under the influence of enzymatic, chemical and physical factors, leading to a decline in the numerical values defining wood properties [Helińska-Raczkowska 1999]. A classification of degradation factors affecting archaeological wood was presented by Ważny [1999] and an in-depth discussion of these factors was given by Witomski [2009]. By assessing the physical and chemical properties of wood, one may estimate the degree of destruction as well as deduce the environmental conditions under which it was deposited. Analyses of the physical properties of wood, such as the maximum moisture content or basic density, are also standard procedures in the assessment of the degree of the degradation of archaeological wood. The parameters of conservation processes may be determined based on these properties [Cook, Grattan 1991]. Due to differences in the macrostructure of examined wood, its chemical composition needs to be considered when estimating the degree of its degradation. Literature sources contain information on the chemical composition, physical properties and conservation of wood dated back several thousand years [Organ 1959; Coles, Coles 1986; Jover 1994; Ward et al. 1996; Kokociński 1999]. However, there are no available studies describing similar findings of pine trees. Apart from the pure science aspect of this study, the aim of the investigations conducted was also to select an adequate conservation method for the excavated objects. Chosen wood material in the best state of preservation will be exhibited after their conservation in the Brudzew commune, in Turek county, west-central Poland, where it was discovered.

Material and methods

Regional setting

The Koźmin Las site (N 52°04'51", E 18°40'3", 97.5 m a.s.l.) is situated in the village of Koźmin in the lowlands of Central Poland. The studied site is located in the middle part of the Warta River valley on the western side, approx. 2 km from the present river channel.

At present, the mean annual temperature in the study area is 8°C, with monthly variations from 18.2°C in July to –2.7°C in January. Average annual precipitation is 515 mm [Kłysik 1993]. The present Warta River is a transitional river, gathering tributaries of various regimes [Maksymiuk 1993].

The area of study was covered with ice sheets several times during the Quaternary period. The last ice cover was present during the Wartanian Stage glaciations of the Odranian Glaciation. The Weichselian Cold Stage was an ice-free period. The closest position of the ice sheet front, approx. 20 km to the north, occurred during the Last Glacial Maximum (LGM) [Marks 2011].

The Warta [Warthe] River valley is incised into a morainic plateau and a fluvioglacial plain. The main valley elements include the remains of erosional terraces of the Warsaw-Berlin ice-marginal valley [the Warsaw-Berlin Urstromtal], the Middle Weichselian high terrace (3.5–8 m above the Warta channel level) and the low terrace of the Late Weichselian/Holocene age (1–4.5 m above the Warta channel level). In terms of its geomorphology, the site lies on a low terrace elevated 1–2 m above the valley floor. The lower terrace is formed by organic deposits with the tree remains covered by mineral deposits, and the organic sediments were the subfossil Late Weichselian floodplain [Petera 2002; Petera-Zganiacz 2007]. The geological position of the area was presented in detail by Dzieduszyńska and Petera-Zganiacz [2012]. The relief of the present-day valley is dominated by several anthropogenic features of the post-exploitation area, because the Koźmin Las site is situated in an area of Miocene lignite exploitation by the Adamów Lignite Mine.

Remains of tree trunks of different ages are common in the Weichselian deposits of the Adamów Lignite Mine [Petera 2002]. Well-preserved subfossil tree trunks were recorded *in situ* approx. 2 m below the present-day surface within organic deposits (mainly organic mud and peat). The organic series is covered by 2–3 m thick inorganic sediments, mostly sandy and sandy-silty overbank alluvia, reaching the present-day surface [Petera-Zganiacz, Dzieduszyńska 2007; Dzieduszyńska et al. 2012].

A detailed study was undertaken in 2010 and 2011 in an open test pit of approx. 160 square metres (fig. 1). Altogether the forest remains were documented in 7 levels, at every 5 to 10 cm. Over 300 objects were registered in the form of stumps, collapsed trunks and branches [Dzieduszyńska et al. 2012]. According to macroscopic identification, these were mostly pine (*Pinus sylvestris* L.) remains and fragments of birch. Most stumps were in the *in situ* position within the organic unit, with well-preserved root systems. Clusters of cones were found between braided roots. Some wood remains were coated with bark. The length of the trunks reached up to several metres (i.e. 2–5 m). Their diameters were locally over 0.2 m (rarely over 0.3 m) with clearly visible annual rings. Dendrological and dendrochronological analyses (by M. Krąpiec) of 114 samples showed that the forest consisted predominantly of pines of an average age of 50–70 years, and had probably existed no longer than approx. 150 years. Narrow-ringed wood and the morphology of trunks were indicative of poor edaphic conditions of the forest community. The forest consisted of trees of a maximum age of approx. 140 years and an average age of 68 years, which overgrew the valley floor [Dzieduszyńska et al. 2014].

The age of the organic unit with tree remains was determined by radiocarbon dating and pollen analysis [Turkowska et al. 2004; Kittel et al. 2012; Dzieduszyńska et al. 2011, 2014]. Most of the tree remains were dated back to the period from 13,000 to 12,600 cal. BP. The forest was destroyed by deteriorating hydrological conditions or a sudden catastrophic event, such as strong wind [Dzieduszyńska et al. 2014]. In general, there were no sedimentological traces of high energy fluvial conditions. Some trunks, especially those from the bottom of the flood deposit layer, were probably transported in a fluvial environment [Kittel et al. 2012].

The multidisciplinary palaeoecological study of the core of the organic layer and tree remains provided an insight into the complexity of the Weichselian late glacial environment, from the late Alleröd up to the end of the Younger Dryas or the early Holocene. The registered events occurred for approx. 2000 years (between ca. 13,000 and 11,200 cal. BP). Organic deposition started during the late Alleröd (12,900–12,600 cal. BP) and possibly finished in the late Younger Dryas and early Holocene (11,600–11,250 cal. BP). In the beginning, the organic deposition could have occurred in a very shallow periodic flood basin. Afterwards, stable conditions on the floodplain were sufficient for the development of the soil horizon and for forest growth. These processes lasted until ca. 12,700–12,600 cal. BP. The upper part of the organic layer showed traces of shallow water or a pool. The increase in flooding activity dated back to ca. 12,600–12,100 cal. BP. The fluvial activity recorded in the inorganic overbank deposition occurred after ca. 11,600–11,250 cal. BP [Dzieduszyńska et al. 2014].

The forest persisted during the Alleröd/Younger Dryas transition under potentially good growth conditions in the floodplain of the Warta River, despite the generally open landscape in Central Poland. The river valley offered protection from the extreme conditions of environmental change of the Late Weichselian period and a refuge for vegetation associations against climate extremes [Madeyska 1998].

The outstanding features of the investigated site are connected with the well-preserved wood, mostly due to the geological properties of the deposits lying over the tree horizon (a silt admixture) and the environmental processes, e.g. rapid flooding of the forest. Besides, as a result of erosional tendencies in the river valley during the Holocene, the morphological fragment, on which the forest developed, became morphologically separated and it nowadays stands 1 – 2 m above the valley floor as the lower terrace. Such a position resulted in a high groundwater level and water saturation of the tree trunk horizon possibly throughout the 12,000 year period. The relatively close proximity of a large lowland river resulted in the stabilisation of the ground water level at a depth of only 0.5 m b.g.l. This is recorded at the site by a distinct horizontal layer of iron admixture (illuvial soil horizon).

The trunks for the present study were made available thanks to the Adamów Lignite Mine drainage works. In the last 20 years of deep drainage within the site area, no drying or destruction of tree remains occurred, which is most prob-

ably the result of the remains being covered with hygroscopic organic mud and inorganic silts, and those deposits prevented the occurrence of the suspended the water table. In turn, without prior drainage of the Koźmin Las site area, the field work at the site would have proved impossible.

Chemical composition and physical properties

Analyses were conducted on a wood sample from the entire cross-section. Concentrations of major and minor components were determined according to the methodology described in the Polish Standard [PN 92/P-50092]. Ethanol was used as a solvent during extraction in a Soxhlet extractor.

The wood for analyses of selected macrostructure characteristics, physical properties and dimensional stability was collected from a 3-m oval trunk of 0.36 to 0.44 m in circumference. Cross-sectional samples showed missing fragments of outer and inner trunk zones and sites of non-centric ring distribution (fig. 2a, 2b). The missing loose fragments were not taken into consideration in the conducted analyses. The log was cut into cross-sectional samples of 10 mm in width. The width of the late and early wood zones was measured on dried control samples using an Epson V33 scanner and Photoshop graphic software accurate to 0.05 mm. Measurements were taken in places in which shrinkage was measured (fig. 2a). These samples were also used to measure the maximum moisture content – μ_{max} (1) (the absolute wood moisture content after saturation with water in a vacuum chamber) and basic density according to formula (2).

$$\mu_{max} = \frac{m_{max} - m_0}{m_0} \cdot 100\% \qquad (1)$$

where: μ_{max} – maximum moisture content [%],
$\quad\quad m_{max}$ – maximum weight of waterlogged wood [kg],
$\quad\quad m_0$ – weight of oven-dried wood [kg].

$$d = \frac{100}{\mu_{max} + 66{,}7} \cdot 10^3 \qquad (2)$$

where: d – basic density [kg·m^{-3}].

Due to the high variation in the wood structure of the cross-sectional samples, the physical properties were also measured within individual zones (fig. 2a).
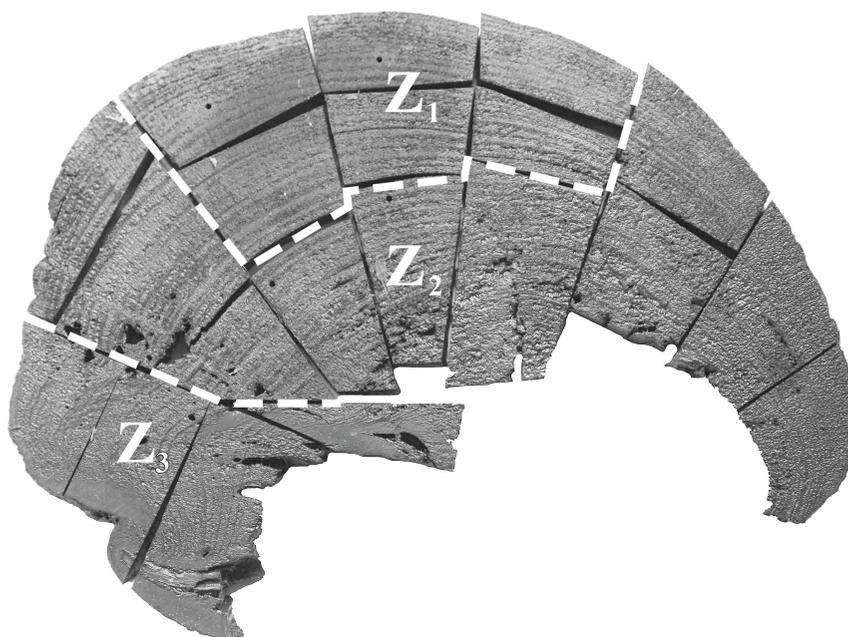
Fig. 2a. A cross-sectional sample divided into zones. Z_1 – outer zone with concentric uniform ring arrangement, Z_2 – zone with numerous missing fragments, Z_3 – deformed wood zone.
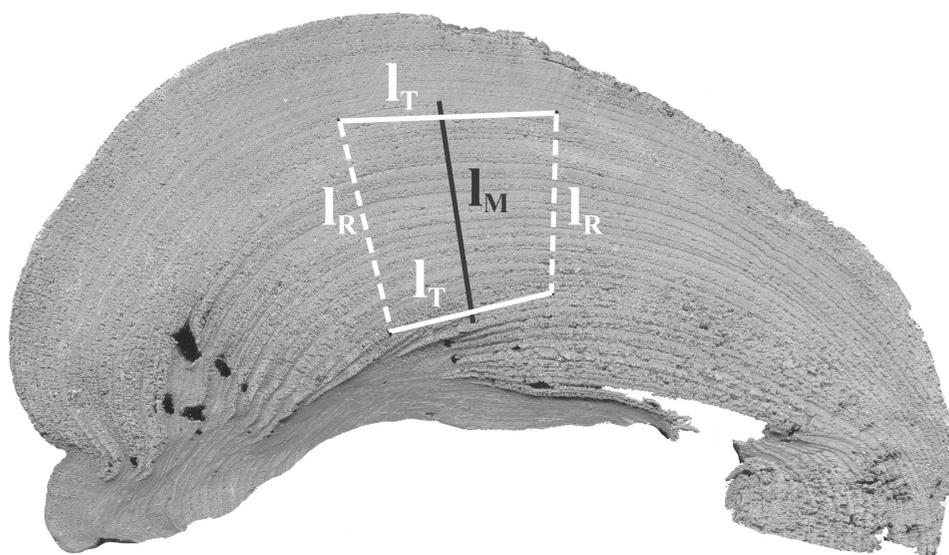

Fig. 2b. A cross-sectional sample of a subfossil log with measured sections marked, l_T – tangential sections, l_R – radial sections, l_M – section for measurements of macroscopic characteristics.

To analyse the dimensional stability, four pins were stuck in each sample. The distances between the pins ranged from 19 to 33 mm, measured in the tangential and radial directions using a slide caliper accurate to 0.01 mm (fig. 2b). The tested wood was saturated with aqueous solutions of polyethylene glycols (PEG) with mean molecular weights of 300, 600 and 4000, as well as mixtures of these. Four wood samples were tested for each impregnation variant. The treatments were initiated using 10% aqueous PEG solutions. Every 4 weeks the concentration was raised by 10% to provide solutions with a total PEG content of 30%. Impregnation baths in PEG solutions at such concentrations may provide very good results in the conservation of archaeological pine wood [Babiński 2011]. In the variants with mixtures of high- and low-molecular weight PEGs the wood was first treated with a PEG of a lower molecular weight. Upon completion of condensation, the samples were immersed in the final solutions for four weeks. The effect of the water contained within the wood on the final concentrations was taken into account for the prepared solutions.

After the samples were taken out of the solution, the wood was dried at a relative humidity of 67%, and then 40% at a temperature of 19–21°C. Humidity ranges were selected based on reports from literature [Brunning et al. 2010; Kozakiewicz, Matejak 2006] and experience gained by the authors of the study. Upon reaching constant mass at 40% humidity, the distances between the pins were again measured.

Wood shrinkage in the tangential, radial and transverse directions was calculated using the following formulas:

$$\beta = \frac{l_1 - l_2}{l_1} \cdot 100 \qquad (3)$$

where: β – linear (tangential or radial direction) shrinkage [%],
l_1 – dimension of the sample at the maximum moisture content [mm],
l_2 – dimension of the sample after drying and seasoning at 40% RH [mm],

$$\beta_{CS} = 100 - \frac{(100 - \beta_T)(100 - \beta_R)}{100} \qquad (4)$$

where: β_{CS} – cross-sectional shrinkage [%],
β_T – tangential shrinkage [%],
β_R – radial shrinkage [%].

The effectiveness of the dimensional stability procedures performed was also assessed, irrespective of the wood species or degree of its degradation. For this purpose, the anti-shrink efficiency (ASE) was applied (5).

$$ASE = \frac{\beta_1 - \beta_2}{\beta_1} \cdot 100 \tag{5}$$

where: ASE – anti-shrink efficiency [%],

β_1 – shrinkage of untreated wood, dried and seasoned at 40% RH [%],

β_2 – shrinkage of treated wood, dried and seasoned at 40% RH [%].

If upon completion of dimensional stabilisation, the ASE = 100%, it means that the wood underwent no deformation. ASE values over 100% are caused by wood swelling, while lower values are equivalent to shrinkage. In conservation practice, ASE exceeding 75% is considered acceptable [Grattan et al. 1980].

The last stage in this study was connected with the drying of the impregnated wood to absolutely dry mass in order to verify the hygroscopic properties of the modified wood. The wood moisture content was expressed as a ratio of the mass of water contained in the modified and dried wood to the absolutely dry mass of the modified wood.

Results and discussion

Table 1 presents the percentage proportions of the major and minor chemical components of the pinewood subjected to the experiment. The concentrations of the major constituents, cellulose and lignin, in the samples of the subfossil wood were approx. 30% and 55%, respectively. A comparison of these results with the percentage proportions of the main components in contemporary wood (around 50 and 27%, respectively) reveals a significant degradation of polysaccharides and an apparent increase in lignin content. A smaller C/L ratio in the case of the subfossil pinewood confirms these changes. However, a comparison of the recorded results with other archaeological wood from other archaeological sites and other historical times shows that the differences are not so significant (Waliszewska at al. 2007.) It means that the time of deposition is not as important as environmental conditions. The analysis of minor wood constituents indicated differences in the content of these substances in the compared materials. On the other hand, it is difficult to indicate a common trend for these differences. Therefore, these results are not particularly useful in the interpretation of degradation processes.

The macroscopic analysis of the tested wood included a determination of the annual ring widths and shares of the late wood. The results recorded and data from literature are given in table 2. On this basis, it may be stated that pine wood of 12 500 years of age exhibits macroscopic characteristics typical of contemporary specimens of this species.

Table 1. Percentage contents of main components, extractives and mineral compounds in pine wood

Wood components	Deposition time of wood remains [years]		
	12 500	600–700[a]	Contemporary wood[b]
Cellulose (C)	29.4	36.8	48.7
Lignin (L)	55.5	51.8	27.0
C/L	0.5	0.7	1.7
Extractives	3.4	8.7	2.6
Substances soluble in cold water	0.9	0.4	1.9
Substance soluble in hot water	1.1	1.0	1.2
Substances soluble in NaOH	16.5	10.7	–
Ash	0.2	2.2	1.1

[a] Data from [Babiński 2005]
[b] Data from [Zborowska et al. 2007]

Table 2. Selected macroscopic features of pine wood

Feature	Deposition time of wood remains [years]		
	12 500	500[a]	Contemporary wood[b]
Width of annual rings [mm]	1.3	1.2	1.5
Percentage of latewood [%]	39.8	55.0	37.6

[a] Data from [Fejfer et al. 2013]
[b] Data from [Zborowska et al. 2007]

Based on the analyses of the physical properties, it was determined that this wood had a mean maximum moisture content of 305%, while the mean basic density was 270 kg·m^{-3}. In view of the physical properties of contemporary pine wood, amounting to 141% and 483 kg·m^{-3}, respectively [Zborowska et al. 2007], it may be concluded that the examined characteristics changed markedly during approx. 12 500 years of deposition in peat. They showed the activity of degradation factors, as did the results of the analyses of the percentage shares of the major and minor components of this material, presented above. However, in view of the age of the objects analysed, their preserved condition was surprisingly good. A similar or lower density was recorded for the archaeological wood coming from pine water pipes from the 16th century, amounting to 220 and 340 kg·m^{-3} [Kokociński 2005], wood from the 14th century with 190 kg·m^{-3} [Babiński 2005] or wood from Biskupin from the 8th century BC, whose density was 136 and 148 kg·m^{-3} in the outer sapwood layer and 409 kg·m^{-3} in the inner layer [Zborowska et al. 2007]. The division of the examined subfossil wood into zones also showed a variation in the physical properties within a single sample. The best

properties were found in the wood from zone Z_1 in which the maximum moisture content was 249% and basic density was 316 kg·m^{-3}, while the wood in the deformed wood zone Z_3 had a maximum moisture content of 274% and basic density of 291 kg·m^{-3}. Zone Z_2 was least preserved, with a maximum moisture content of 338% and basic density of 237 kg·m^{-3}. These results may indicate a faster degradation of inner pith wood zones and changes caused by the pressure of the soil layers covering the remnants of a former forest.

Table 3 presents the shrinkage of the treated and untreated wood along with the ASE values for all the treatment variants. The slight shrinkage of the control samples, amounting to 6.2% in the tangential direction and 3.5% in the radial direction, is comparable to the mean values of shrinkage of contemporary pine wood β_T – 7.5…8.0% and β_R – 3.3…4.0% [Wagenführ, Scheiber 1989]. The low shrinkage of the examined subfossil wood may have been caused by earlier drying. The site in which the wood was discovered is located in the immediate vicinity of an open pit lignite mine. The drained area could have caused the drying and shrinkage of the wood *in situ* within the last ten - twenty years of the mine operation. This thesis is confirmed by cross-checks in the excavated logs. For the treated samples, the best dimensional stabilisation was obtained when applying 30% solutions of PEG 300 and PEG 600, when only minimal wood swelling was observed. Sample saturation with a 30% solution of PEG 4000 resulted in tangential wood shrinkage amounting to 2.2% and radial shrinkage of 0.8%. These relatively low shrinkage values for the conserved archaeological pine wood were not reflected in the calculated dimensional stability indexes (ASE), which amounted to 63.2% in the tangential direction and 76.5% in the radial direction, respectively. Such low values of ASE indicated poor dimension stabilisation by the PEG 4000 solution. Variants with mixtures of PEG 300 and PEG 4000, as well as PEG 600 and PEG 4000, assumed intermediate values at low shrinkage and good ASE levels.

Table 3. Tangential, radial and cross-sectional shrinkage (β_T, β_R, β_{CS}) and anti-shrink efficiencies (ASE_T, ASE_R, ASE_{CS}) of untreated and treated wood after seasoning at 40% RH and at a temperature of 18–21°C

Impregnator	Shrinkage [%]			Anti-shrink efficiencies [%]		
	β_T	β_R	β_{CS}	ASE_T	ASE_R	ASE_{CS}
Control	6.2	3.5	9.5	–	–	–
PEG 300 30%	-0.3	0.0	-0.3	104.9	100.6	103.5
PEG 300 20% + PEG 4000 10%	0.6	0.2	0.8	89.2	94.2	90.7
PEG 300 10% + PEG 4000 20%	1.4	0.5	1.9	76.2	85.2	78.8
PEG 4000 30%	2.2	0.8	3.0	63.2	76.5	67.0
PEG 600 30%	-0.3	0.0	-0.3	104.6	101.1	103.4
PEG 600 20% + PEG 4000 10%	0.6	0.4	1.0	89.8	88.7	89.1
PEG 600 10% + PEG 4000 20%	1.5	0.5	2.0	75.3	83.6	77.6

The measured wood moisture contents after drying are given in table 4. The lowest moisture content was recorded in the wood treated with the PEG 4000 solution. In turn, the highest moisture content was detected in the samples treated with 30% solutions of PEG 300 and PEG 600. These results confirm the hygroscopic properties of low-molecular weight polyethylene glycols. As expected, intermediate values were recorded for the mixtures of PEGs with different molecular weights.

Table 4. Water content in treated wood after seasoning at 40% and 67% RH

Impregnator	Water content [%]	
	RH 67%	RH 40%
Control	14.5	11.0
PEG 300 30%	13.4	7.4
PEG 300 20% + PEG 4000 10%	10.0	5.5
PEG 300 10% + PEG 4000 20%	8.2	4.6
PEG 4000 30%	7.1	4.1
PEG 600 30%	12.0	5.0
PEG 600 20% + PEG 4000 10%	9.1	4.4
PEG 600 10% + PEG 4000 20%	7.8	4.4

Conclusions

1. The tested pine wood of 12 500 years old had chemical and physical properties comparable to many findings of archaeological wood dated back several hundred years.
2. The good preservation condition of the wood resulted from the deposition of the tree remains within organic and silty sediments, their being rapidly covered by organic material in 2 m thick sandy-silty flood deposits, as well as the hyperhydration of the deposits with the tree remains.
3. The consistent results of the analyses of the chemical and physical properties facilitated an identification of the changes within the structure of the major wood components and a determination of the degree of wood degradation. This made it possible to indicate the appropriate conservation technique.
4. The 30% solutions of PEG 300 and PEG 600, as well as their mixtures with PEG 4000, provided good stabilisation for the tested material.
5. Due to the hygroscopic character of PEG 300 and PEG 600, pure solutions of these polymers should not be used to conserve wood with similar properties. A better option would be to impregnate the wood with a mixture of PEG 4000 with low-molecular polyethylene glycols, whose concentration in the impregnating solution does not exceed 10%. In the examined pine wood, this solution caused only a slight increase in hygroscopic properties.

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