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Evaluation of the state of preservation of waterlogged archaeological wood based on its physical properties: basic density vs. wood substance density

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ABSTRACT

The state of preservation of waterlogged archaeological wood was evaluated on the basis of the maximum moisture content (MMC), the basic density (BD) and the wood substance density (WSD) determined in water and helium. The degree of wood degradation was compared under the criteria: the loss of wood substance (LWS) and the loss of wood substance density (LWSD). Studies were conducted on the wood samples differing in species, degree of degradation, age and place of origin. The physical properties of wood were determined for the material containing mineral compounds and the material without minerals. The properties of the latter, in which the minerals are replaced by water, were calculated from the mass and volume of the wood containing minerals as well as the content and density of the ash obtained after burning the sample.

The study revealed the effect of minerals on the tested parameters and wood degradation indices. A strong relationship between BD and MMC was confirmed for both the wood containing minerals and without them, by contrast a substantially weaker correlation between BD and WSD was observed. It was found that the assessment of the state of wood preservation conducted on the basis of LWS and LWSD yielded different results. In addition, it was revealed that both indices of wood degradation might be unreliable. The main drawback of the LWS-based assessment is associated with a wide range of basic density of fresh wood. In turn, the LWSD mainly indicates the changing ratio of the carbohydrates/lignin content, but fails to provide information on the loss of wood substance. This may hinder the comparison of the wood sampled from different sites and subjected to different decay mechanisms. Nevertheless, the WSD-based assessment of the state of preservation of waterlogged archaeological wood might be a valuable complementary method to the BD- and/or MMC-based assessment, which is routinely carried out in many conservation centers.

1. Introduction

The state of preservation of waterlogged archaeological wood is evaluated mainly for the selection of an appropriate method for its conservation. Such a test is performed also in the case of monitoring the ongoing rate of degradation of wood tissue. The degree of wood decomposition can be determined on the basis of its physical and chemical properties as well as by microscopic observations (Babiński, 2005; Capretti et al., 2008; Gregory and Jensen, 2006; Hoffmann et al., 1986; Hoffmann and Parameswaran, 1982; Macchioni et al., 2012; Passialis, 1997; Pizzo et al., 2013; Schniewind, 1990). In many conservation laboratories, evaluation of the state of preservation of archaeological wood tissue is most often based on the physical properties of the wood, related to its mass and volume. This is because it is a simple research method and considerable repeatability of the results can be obtained. The fact that the choice of a method of conservation of waterlogged archaeological wood is mainly based on its physical properties should not be underestimated. Such a procedure has been widely described in many publications, which makes it a valuable source of information for conservators of monuments. On the other hand, no detailed procedures have yet been worked out to enable choosing an appropriate method for dimensional stabilization and strengthening of wood on the basis of the results of its chemical and/or microscopic analyses.

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Usually, in order to evaluate the degree of degradation of waterlogged wood its maximum moisture content and basic density are considered. To a much lesser extent, decay of the objects to be conserved is evaluated on the basis of wood density in waterlogged condition or porosity (Donato and Lazzara, 2012; Hoffmann and Blanchette, 1997; Mikolaychuk, 1997). However, the application of wood substance density for that purpose has not often been observed.

It is widely acknowledged that the mass and volume of wood substance forming the cell walls decreases as the wood decay develops. This process results in a decrease in basic density and wood density in wet condition, as well as an increase in porosity and maximum moisture content. There are strict dependences between all those properties, described in many handbooks on wood science. Practical guidelines for determining the physical properties of waterlogged archaeological wood and the dependences between them were presented in detail by Jensen and Gregory (2006).

However, wood is a material the properties of which vary not only with the degree of decomposition of its cell walls. The physical properties of fresh wood depend not only on its species, but also on the sampling place (trunk, branches and roots; sapwood and heartwood; juvenile and mature wood). Significant variation of the properties under discussion was also noted depending on the conditions pertaining to the tree growth, including its biosocial position in the woodland stand (Fabisiak, 2005). One should also not underestimate changes that appear in wood with certain natural defects (e.g. knots, reaction wood). At the same time, in the case of small archaeological objects it is difficult to evaluate the source from which the fragment of tested wood tissue was drawn.

Comparing the physical properties of archaeological wood to average values assumed for non-degraded fresh material may be unreliable from the point of view of wood decay evaluation. It is related to the lack of information on the properties of archaeological wood before its degradation. Therefore, such an evaluation, including the loss of wood substance calculated on the basis of changes in its basic density (Bilz and Macchioni, 2005; Grattan and Mathias, 1986), should always be treated as an approximation.

Most often a determination of the physical properties of archaeological wood under discussion is performed with the use of small, irregular samples. The mass and volume of the tested sample is determined by weighing the waterlogged wood in air and water, and weighing the oven-dried sample. The accuracy of such measurements is influenced by small amounts of air that still may remain in the wet wood, an excess of water on the sample surface, as well as by over-drying of the sample. Additionally, regarding the different properties of early and late wood – material that varies in macroscopic characteristics (e.g. width of annual increments) and non-homogeneity of the process of wood decay – the size of a sample will also influence the value obtained during the testing. In the case of small archaeological objects an evaluation of the degree of wood degradation is possible only with the use of non-destructive methods or by using very small samples. Due to the relatively high probability of a mistake resulting from weighing a wet sample and the increased density of sorbed water (Jensen and Gregory, 2006), an evaluation of wood decomposition on the basis of wood substance density determined on dry material with precise measurements of volume made with the use of a gas pycnometer seems worth considering.

The gas pycnometry method is based on Archimedes' principle of gas displacement. The sample volume is defined as the volume not occupied by gas in a calibrated chamber and determined by gas pressure measurements. Helium is the preferred displacement medium. It is a noble gas, it does not react with other substances and it is not adsorbed on the surface of solids at ambient temperature. Such properties of helium eliminate the problems connected with the formation of blow holes and dissolution of samples in liquids. In addition, helium molecules are small (0.255 nm), so they fill the tiniest pores of the sample and make it possible to precisely determine the sample volume. However, it should be noted that the closed pores that are inaccessible to helium are also included in the measured volume. This technique provides a fully automatic, high-precision and non-destructive measurements even on very small samples.

Wood substance density can be determined in water, some organic solvents or in gas (helium preferably). For fresh wood the values obtained in water are distinctly higher than the values obtained in helium or organic solvents (e.g. Christensen and Hergt, 1968). In spite of the differences in density of the main chemical components of cell walls (Beall, 1972) and differences in chemical composition of various wood species (Fengel and Grosser, 1975), in handbooks on wood science it is assumed that the density of fresh wood substance changes only within a narrow range and is independent of the type of tested wood. The research performed by Kellogg and Wangaard (1969) confirms this thesis to a great extent. Additionally, a strict dependence between wood substance density and the content of alpha-cellulose and crystallinity of cellulose has been found (Kellogg et al., 1975).

Changes in wood substance density predominantly result from the changes in percentage relationships between the main chemical components of the decomposing material. In the case of degraded archaeological wood in which the ratio of the content of polysaccharides to the content of lignin is decreased, wood substance density is lower than in non-degraded wood. This thesis was confirmed by Taniguchi et al. (1986a, 1986b). In turn, the influence of the loss of wood substance on the basic density of archaeological wood determined through various methods was tested by Passialis (1998).

In fresh wood, apart from the main chemical compounds and extractives (soluble in water or organic solvents), there is also a small content of mineral compounds. In wood of European species, the content of mineral compounds does not exceed 1% (Fengel and Grosser, 1975; Fengel and Wegener, 1989; Prosiński, 1984; Wagenführ and Schreiber, 1989). Due to the small amounts involved, their influence on wood substance density in sound wood was ignored. By contrast, in archaeological wood the ash content can increase by many times. It is particularly noticeable in material with a great degree of degradation in which the quantity of wood substance has decreased considerably. The higher participation of mineral compounds, which may even exceed 10% of the oven-dry mass of wood (Macchioni et al., 2012), results from the easy penetration of mineralized water into the wood. The density of salts in wood is considerably higher than the wood substance density. Therefore, following the increase in the degree of decay of carbohydrates and the increase in the content of mineral compounds, it is to be expected that the influence of mineral content on the density of wood substance and other physical properties measured for an evaluation of the degree of wood decomposition will be higher.

This research was aimed at making a comparison of the degree of decomposition of waterlogged archaeological wood evaluated on the basis of its basic density (and maximum moisture content) with an evaluation of wood degradation on the basis of wood substance density determined in water and helium. The research also included the influence of mineral compounds on selected physical properties and the estimated state of wood decomposition.

2. Material and methods

2.1. Wood

The research was done on 28 samples of waterlogged archaeological wood representing various hardwoods (oak, alder, ash, elm,

beech) and softwoods (Scots pine, Norway spruce) coming from different archaeological sites (located in Poland, Italy and Germany), taken from objects ranging from 350 to 12,000 years old. The material researched was supplemented with two samples of modern wood (oak, Scots pine) buried for eight years in the wet conditions of the archaeological site of Biskupin, Poland. Table 1 shows sample symbols, species of wood, origin and age of objects from which the research material was drawn.

The individual samples were characterised by homogenous wood degradation, initially evaluated with the use of a preparation needle. The samples had shapes similar to cuboids, the volume of which ranged from ca. 8 up to 40 cm^3 depending on availability and the degree of decomposition of the tested material. Oven-dry mass of the samples ranged from 1.7 to 15.3 g.

2.2. Determination of physical properties of wood

Prior to measurements, wood samples drawn from wet archaeological objects were stored in water. In order to obtain maximum saturation when immersed in distilled water, the samples were placed in a vacuum chamber. During each cycle of impregnation, the samples were kept in water for 4 h at a pressure of ca. 50 hPa and then for 20 h at atmospheric pressure. The treatment was repeated 5 to 15 times until a constant sample mass was achieved. After maximum moisture content was achieved, the samples were weighed in distilled water and then in air after removal of the excess water from the sample surface. Weighing of the samples in water and air was repeated 5 times and the average values were calculated. An analytical balance with an accuracy up to 0.0001 g was used for weighing. In the next stage of the experiment, the waterlogged samples were divided into smaller pieces and air-dried in the laboratory for two weeks, then oven-dried for 24 h at a temperature of 103 °C and weighed again in air. Further drying for 24 h did not change significantly the mass of the samples.

The degree of wood degradation was presented on the basis of Maximum Moisture Content (MMC), Basic Density (BD), Residual Basic Density (RBD), Loss of Wood Substance (LWS) and Wood Substance Density (WSD). MMC, BD and RBD were calculated according to the following formulae:

$$MMC = 100^{*}(m_w - m_d)/m_d$$

where

MMC – maximum moisture content (%),
m_w – mass of waterlogged sample (g),
m_d – mass of oven-dry sample (g),

$$BD = m_d/V_w$$

where

BD – basic density (g/cm^3),
m_d – mass of oven-dry sample (g),
V_w – volume of waterlogged sample (cm^3),

$$RBD = 100^{*}BD_a/BD_f$$

where

RBD – residual basic density (%),
BD_a – basic density of archaeological wood (g/cm^3),
BD_f – basic density of fresh wood (data given by Dietz, 1975) (g/cm^3).

Loss of wood substance (loss of mass) was calculated by the equation (after Grattan and Mathias, 1986):

Table 1
List of the examined wood samples.

Sample	Wood	Species	Object	Age (years)	Origin
Que-1	Oak, heartwood	*Quercus* sp.	Rampart	1000	Gdańsk, Poland
Que-2	Oak, heartwood	*Quercus* sp.	Pole	2750	Biskupin, Poland
Que-3	Oak, sapwood	*Quercus* sp.	Pole	2750	Biskupin, Poland
Que-4	Oak, heartwood	*Quercus* sp.	Pole	2750	Biskupin, Poland
Que-5	Oak, heartwood	*Quercus* sp.	Pole	2750	Biskupin, Poland
Que-6	Oak, heartwood	*Quercus* sp.	Beam	750	Szczecin, Poland
Que-7	Oak, heartwood	*Quercus* sp.	Pole	2750	Biskupin, Poland
Que-8	Oak, heartwood	*Quercus* sp.	Sample	Fresh (80)	Biskupin, Poland
Que-9	Oak, heartwood	*Quercus* sp.	Pole	2750	Biskupin, Poland
Fra-1	Ash	*Fraxinus excelsior* L.	Rampart	1000	Gdańsk, Poland
Fra-2	Ash	*Fraxinus excelsior* L.	Rampart	1000	Gdańsk, Poland
Aln-1	Alder	*Alnus glutinosa* Gearth.	Rampart	1000	Gdańsk, Poland
Aln-2	Alder	*Alnus glutinosa* Gearth.	Rampart	1000	Gdańsk, Poland
Ulm-1	Elm	*Ulmus* sp.	Rampart	1000	Gdańsk, Poland
Fag-1	Beech	*Fagus silvatica* L.	Board	1100	Pfettrach, Germany
Pin-1	Scots pine, sapwood	*Pinus sylvestris* L.	Pipeline	630	Krosno, Poland
Pin-2	Scots pine, heartwood	*Pinus sylvestris* L.	Beam	500	Żnin, Poland
Pin-3	Scots pine, heartwood	*Pinus sylvestris* L.	Pipeline	500	Żnin, Poland
Pin-4	Scots pine, sapwood	*Pinus sylvestris* L.	Pipeline	500	Żnin, Poland
Pin-5	Scots pine, heartwood	*Pinus sylvestris* L.	Beam	500	Żnin, Poland
Pin-6	Scots pine, sapwood	*Pinus sylvestris* L.	Tree trunk	12000	Koźmin, Poland
Pin-7	Scots pine, heartwood	*Pinus sylvestris* L.	Beam	350	Gdańsk, Poland
Pin-8	Scots pine, heartwood	*Pinus sylvestris* L.	Tree trunk	12000	Koźmin, Poland
Pin-9	Scots pine, sapwood	*Pinus sylvestris* L.	Sample	fresh (100)	Biskupin, Poland
Pin-10	Scots pine, heartwood	*Pinus sylvestris* L.	Tree trunk	12000	Koźmin, Poland
Pic-1	Norway spruce, sapwood	*Picea abies* (L.) Karst	Tree trunk	6000	Val di Non, Italy
Pic-2	Norway spruce, sapwood	*Picea abies* (L.) Karst	Tree trunk	6000	Val di Non, Italy
Pic-3	Norway spruce, heartwood	*Picea abies* (L.) Karst	Tree trunk	6000	Val di Non, Italy
Pic-4	Norway spruce, heartwood	*Picea abies* (L.) Karst	Tree trunk	6000	Val di Non, Italy
Pic-5	Norway spruce, heartwood	*Picea abies* (L.) Karst	Tree trunk	6000	Val di Non, Italy

$$LWS = 100^{*}\left(BD_f - BD_a\right)/BD_f$$

where

LWS – loss of wood substance (%),
BD_f – basic density of fresh wood (data given by Dietz, 1975) (g/cm^3),
BD_a – basic density of archaeological wood (g/cm^3).

Wood substance density determined in water was calculated with the formula stated by Jensen and Gregory (2006):

$$WSD_w = d_w/\left[1 + R_{sorb} + (M_{up} - m_w)/m_d\right]$$

where

WSD_w – wood substance density determined in water (g/cm^3),
d_w – density of water in pores (g/cm^3),
R_{sorb} – correction factor, due to sorbed water ($R_{sorb} = 0.028$),
M_{up} – mass of displaced volume of waterlogged sample (g),
m_w – mass of waterlogged sample (g),
m_d – mass of oven-dry sample (g).

After these tests, the same specimens were used for determination of wood substance density in helium (WSD_h). The measurements were performed in an AccuPyc 1330 automatic gas pycnometer (Micromeritics, USA). Determinations were made on the material coming from transverse and/or radial sections of wood, so both early and late wood from all available annual increments was tested. The samples were ground and oven-dried at 103 °C to constant mass. Prior to volume measurements, samples placed in the pycnometer chamber were rinsed with helium five times in order to remove particles of gases adsorbed on their surface. Sample weight ranged from ca. 0.5–2.1 g. Five to ten replications of density measurements were made for each sample.

2.3. Determination of the content of mineral compounds (content of ash)

The sawdust from each oven-dried sample was placed in a porcelain crucible and burnt in an electric furnace. The temperature was gradually increased from 100 up to 575 ± 25 °C and maintained for 3–4 h to the moment of complete combustion. All weight measurements were made with the accuracy up to 0.0001 g. The content of mineral compounds (ash) in the sample was expressed as a percentage of the oven-dry mass of wood.

2.4. Correction of the physical properties of wood

In the final stage of the study, physical properties of the tested wood were calculated assuming that the mineral compounds were replaced by water. Corrected physical properties of wood without minerals were calculated using the following formulae:

$$MMC = 100^{*}[m_w - m_d + m_d{}^{*}a^{*}d_w/(100^{*}d_a)]/[m_d{}^{*}(1 - a/100)]$$

$$BD = m_d{}^{*}(1 - a/100)/V_w$$

$$WSD_w = d_w/\left[1 + R_{sorb} + (M_{up} - cm_w)/cm_d\right]$$

$$cm_d = m_d{}^{*}(1 - a/100)$$

$$cm_w = m_w - (m_d - cm_d) + (m_d - cm_d)/d_a$$

$$WSD_h = m_d{}^{*}(1 - a/100)/[V - m_d{}^{*}a/(100^{*}d_a)]$$

where

a – content of ash (%),
d_a – density of ash (g/cm^3),
cm_w – corrected mass of waterlogged sample (g),
cm_d – corrected mass of oven-dry sample (g),
V – volume of sample (wood with mineral compounds) (cm^3).

For the other symbols see section 2.2.

Ash density was determined in a gas pycnometer on the material obtained after burning the wood at a temperature of 575 °C. The same procedure as for determination of wood substance density was applied (see section 2.2).

3. Results and discussion

Table 2 presents the properties of tested wood containing mineral compounds. Maximum moisture content in oak wood ranged between 97% and 990%. Thus, among the samples under research, all three classes of wood degradation can be distinguished, according to MMC limit values of 185% and 400% (Christensen, 1970; De Jong, 1977). The rest of the samples of hardwood, evaluated according to the same criterion, may be recognized as highly decayed (MMC from 573% to 1121%). In pine and spruce wood, the MMC was from 141% to 499%. About half of the softwood material fell into the MMC range characteristic of non-degraded wood of Scots pine or Norway spruce. Basic density (BD) of hardwood with the highest degree of decomposition was lower than 0.150 g/cm^3. For comparison, the BD of modern oak wood buried for eight years in the wet conditions of the archaeological site of Biskupin (sample Que-8) was 0.562 g/cm^3, which is similar to the average value accepted for sound wood (Dietz, 1975). By contrast, in the case of sample Que-9, the basic density of the archaeological wood was distinctly higher than the average value characteristic of fresh material. Among the softwood samples, a few (samples Pin-8, 9 and 10 as well as Pic-4 and 5) had higher BD values than the values accepted for sound wood of pine and spruce.

In order to compare the state of preservation of various species of wood, the degree of decomposition was presented as Residual Basic Density and Loss of Wood Substance, calculated in relation to the average basic density of fresh wood (BD_f). When the basic density of archaeological wood was higher than the value for non-degraded wood, the RBD stated in Table 2 acquired a value above 100%. This was observed for the slightly degraded material (Que-9, Pin-8, 9 and 10 as well as Pic-4 and 5). Then the LWS indices took on negative values. An RBD above 100% or a negative LWS resulted from the fact that instead of the (unknown) initial basic density of the examined archaeological wood (before its decomposition), the average basic density of fresh wood was used in the calculations. Therefore, the indices obtained will be treated as incorrect. Actually, RBD should be less than 100% and LWS should take on positive values. Using average basic densities for calculations is encumbered with errors resulting from differences in the properties (including density) of wood within each species (Wagenführ and Schreiber, 1989). Among other things, it is a result of the differences in macroscopic structure that appear mainly in softwood and ring-porous hardwood. The differences in wood density also seem to depend on which tree section the sample was taken from, as well as the conditions of tree growth. At the same time, it cannot be excluded that the RBD and LWS values stated here for material

Table 2
Physical properties of wood with mineral compounds.

Sample	MMC (%)	BD_a (g/cm^3)	BD_f (g/cm^3)	RBD (%)	LWS (%)	WSD_w (g/cm^3)	SD (WSD_w) (g/cm^3)	WSD_h (g/cm^3)	SD (WSD_h) (g/cm^3)
Que-1	990	0.095	0.577	16.5	83.5	1.465	0.000262	1.423	0.002540
Que-2	670	0.134	0.577	23.2	76.8	1.294	0.000334	1.314	0.002345
Que-3	617	0.145	0.577	25.1	74.9	1.345	0.000248	1.343	0.001975
Que-4	269	0.296	0.577	51.3	48.7	1.406	0.000086	1.369	0.001014
Que-5	179	0.411	0.577	71.2	28.8	1.496	0.000100	1.436	0.001948
Que-6	124	0.518	0.577	89.8	10.2	1.402	0.000089	1.387	0.002944
Que-7	118	0.549	0.577	95.1	4.9	1.501	0.000022	1.472	0.001231
Que-8	113	0.562	0.577	97.4	2.6	1.475	0.000344	1.447	0.001843
Que-9	97	0.625	0.577	108.3	−8.3	1.514	0.000019	1.448	0.001693
Fra-1	1121	0.084	0.568	14.8	85.2	1.453	0.000091	1.429	0.001887
Fra-2	1076	0.087	0.568	15.3	84.7	1.466	0.000345	1.443	0.001500
Aln-1	1062	0.089	0.447	19.9	80.1	1.468	0.000667	1.433	0.001097
Aln-2	921	0.101	0.447	22.6	77.4	1.460	0.000337	1.423	0.001421
Ulm-1	928	0.100	0.568	17.6	82.4	1.441	0.000025	1.417	0.002347
Fag-1	573	0.155	0.578	26.8	73.2	1.377	0.000091	1.363	0.001321
Pin-1	499	0.176	0.418	42.1	57.9	1.407	0.000088	1.382	0.001922
Pin-2	356	0.237	0.418	56.7	43.3	1.457	0.000122	1.435	0.003577
Pin-3	276	0.291	0.418	69.6	30.4	1.435	0.000049	1.423	0.003621
Pin-4	247	0.318	0.418	76.1	23.9	1.434	0.000044	1.413	0.001787
Pin-5	200	0.371	0.418	88.8	11.2	1.402	0.000031	1.416	0.005204
Pin-6	196	0.381	0.418	91.1	8.9	1.457	0.000134	1.410	0.002329
Pin-7	179	0.404	0.418	96.7	3.3	1.394	0.000142	1.389	0.000607
Pin-8	164	0.433	0.418	103.6	−3.6	1.429	0.000026	1.418	0.001458
Pin-9	151	0.459	0.418	109.8	−9.8	1.447	0.000208	1.443	0.001351
Pin-10	141	0.481	0.418	115.1	−15.1	1.438	0.000111	1.415	0.003273
Pic-1	327	0.253	0.403	62.8	37.2	1.440	0.000620	1.406	0.004740
Pic-2	235	0.331	0.403	82.1	17.9	1.428	0.000143	1.393	0.001745
Pic-3	208	0.366	0.403	90.8	9.2	1.468	0.000369	1.435	0.001001
Pic-4	164	0.434	0.403	107.7	−7.7	1.447	0.000221	1.425	0.001117
Pic-5	156	0.450	0.403	111.7	−11.7	1.446	0.000136	1.413	0.001728

Abbreviations in Tables 2 and 3: MMC – maximum moisture content, BD_a – basic density of archaeological wood, BD_f – basic density of fresh wood (Dietz, 1975), RBD – residual basic density, LWS – loss of wood substance, WSD_w – wood substance density determined in water, WSD_h – wood substance density determined in helium, SD – standard deviation.

characterised by a higher degree of degradation may also vary from the actual ones. Individual evaluation of the initial basic density of archaeological wood on the basis of selected features of its anatomical structure (e.g. width of annual increments and the percentage of late wood) could provide some solution to that problem. However, such an evaluation could raise some justifiable doubts and may not always be possible to implement.

Wood substance densities determined in water (WSD_w) ranged from 1.294 (Que-2) to 1.514 g/cm^3 (Que-9) (Table 2). The highest value was similar to that of fresh hardwood, determined by Kellogg and Wangaard (1969). In turn, the minimum value was lower than the average density of lignin. Wood substance densities determined in helium (WSD_h) were from 1.314 (Que-2) to 1.472 g/cm^3 (Que-7). In most of the samples, the values determined in helium were lower than the density determined in water. When the WSD determined in water was lower than in helium (e.g. Que-2 and Pin-5), it could be supposed that the values measured in water were lower than the actual ones. It could be a result of the presence of small amounts of air in some samples. One should also take into consideration the possibility of lower decay of the material that used for measurements in the gas pycnometer, was taken from the surface layer of the sample tested in water for WSD. Another reason could have been a higher content of mineral compounds in the sample tested in helium. At the same time, it was found that a decrease in the density of wood substance coming from hardwood does not correspond with an increase in the degree of its degradation (evaluated on the basis of a decrease of BD or an increase in MMC). One should also note the relatively high WSD observed in the case of the most intensively decomposed hardwood (Fra-1 and 2, Aln-1 and 2 as well as Ulm-1). Unexpectedly, a high value was also noted for the most degraded oak wood (Que-1). Increased WSD was observed also for sample Que-5 (in comparison to a considerably lower value determined for sample Que-6 showing a similar state of wood preservation). Lack of an explicit dependence between WSD and MMC or WSD and BD was also observed in the case of softwood. In spite of various values of basic density, the determined values of wood substance density were as a rule at a similar level, and in the case of a relatively well-preserved samples of pine wood (Pin-7) WSD was even distinctly lower than in more decayed samples.

Except for the samples Que-2 and Pin-5, the differences between WSD values determined in water and helium ranged from 0.002 to 0.066 g/cm^3. WSD_h was lower on average by about 0.025–0.030 g/cm^3. Lower wood substance densities determined in helium or a solvent which does not cause wood swelling have been noted and explained many times in the case of research on fresh wood (e.g. Christensen and Hergt, 1968; Kollmann and Côté, 1968; Wilfong, 1966). However, usually these differences are at least 0.050 g/cm^3. The smaller differences observed for some samples of tested archaeological wood could be a result of non-uniform degradation of wood tissue and of using different material for measurements in both environments (water, helium). The possibility of small amounts of air remaining in large samples used for WSD_w determinations cannot be excluded either.

Table 3 presents the content of ash in tested wood. Wood showing a higher degree of decomposition (evaluated on the basis of RBD or LWS) was characterized by a higher content of mineral compounds. As the ash density is about two times higher than the density of main chemical compounds of wood, it influenced significantly the determined wood properties. Corrected physical properties of the tested wood (without mineral compounds) and corrected RBD and LWS are listed in Table 3. After the correction

Table 3
Ash content and physical properties of wood without mineral compounds.

Sample	Ash	MMC	BD_a	RBD	LWS	WSD_w	WSD_h
	(%)	(%)	(g/cm^3)	(%)	(%)	(g/cm^3)	(g/cm^3)
Que-1	7.1	1068	0.088	15.3	84.7	1.411	1.366
Que-2	3.3	694	0.130	22.5	77.5	1.270	1.288
Que-3	6.1	659	0.136	23.6	76.4	1.299	1.294
Que-4	5.0	285	0.281	48.7	51.3	1.369	1.329
Que-5	3.3	186	0.397	68.8	31.2	1.471	1.410
Que-6	1.7	127	0.509	88.2	11.8	1.390	1.373
Que-7	2.4	122	0.536	92.9	7.1	1.483	1.452
Que-8	0.7	114	0.558	96.7	3.3	1.470	1.441
Que-9	4.6	103	0.596	103.3	−3.3	1.480	1.411
Fra-1	6.6	1202	0.078	13.7	86.3	1.403	1.376
Fra-2	8.0	1172	0.080	14.1	85.9	1.405	1.377
Aln-1	8.1	1158	0.082	18.3	81.7	1.406	1.366
Aln-2	7.3	996	0.094	21.0	79.0	1.404	1.363
Ulm-1	6.9	999	0.093	16.4	83.6	1.388	1.361
Fag-1	3.3	594	0.150	26.0	74.0	1.353	1.336
Pin-1	2.1	510	0.172	41.1	58.9	1.391	1.365
Pin-2	1.5	362	0.233	55.7	44.3	1.446	1.423
Pin-3	2.4	284	0.284	67.9	32.1	1.417	1.403
Pin-4	1.8	252	0.312	74.6	25.4	1.421	1.398
Pin-5	1.3	203	0.366	87.6	12.4	1.393	1.406
Pin-6	2.4	202	0.372	89.0	11.0	1.439	1.390
Pin-7	0.3	180	0.403	96.4	3.6	1.392	1.386
Pin-8	1.9	168	0.425	101.7	−1.7	1.415	1.403
Pin-9	0.9	153	0.455	108.9	−8.9	1.441	1.436
Pin-10	1.9	144	0.472	112.9	−12.9	1.424	1.400
Pic-1	1.0	331	0.250	62.0	38.0	1.432	1.398
Pic-2	0.9	237	0.328	81.4	18.6	1.421	1.386
Pic-3	1.2	211	0.362	89.8	10.2	1.459	1.426
Pic-4	0.7	165	0.431	106.9	−6.9	1.442	1.420
Pic-5	0.6	157	0.447	110.9	−10.9	1.442	1.409

MMC increased by 1–96%. By contrast, BD decreased by 0.001–0.029 g/cm^3. Correction of the basic density of archaeological wood resulted in lowered RBD and increased LWS values by a maximum of 5%. The density of wood substance without mineral compounds determined in water and helium decreased by 0.002–0.062 g/cm^3 and 0.003–0.067 g/cm^3, respectively. The greatest reduction in the density of wood substance without mineral compounds was observed in the case of the samples of highly decomposed hardwood with the highest ash content. In these cases the corrected WSD values lowered by not less than 0.050 g/cm^3. For the less decomposed wood of pine and spruce the decrease in the corrected WSD did not exceed 0.020 g/cm^3.

The corrected physical properties of wood were calculated on the basis of the content and density of the ash. These values may, however, differ slightly from the content and density of mineral compounds in the sample prior to the burning. Errors in the determination of the ash content may derive from the loss of ammonia and iron salts, alkali metal chlorides as well as from insufficient oxidation of carbonates of alkaline earth metals (Fengel and Wegener, 1989). In the fresh wood, the differences between mineral compounds before and after combustion of the sample are not taken into account. But due to the higher content of minerals, this difference might be more important in the case of archaeological wood. Nevertheless, identification of the error resulting from the use of ash parameters to calculate the corrected physical properties of wood was beyond the scope of this study. Science has yet to find an answer to the problem of the influence of mineral compounds before and after combustion of wood on its physical properties.

The relationship between basic density and maximum moisture content, determined on the basis of the properties of the tested samples, is presented in Fig. 1. Evaluation of the strength of correlation was based on the values of correlation coefficient (R). There is a close relationship between the analysed properties, both in the case of properties determined for wood with mineral compounds and for wood without mineral compounds. In both cases the correlation coefficient ranged between −0.99 and −1. Such a strict dependence is due to using the same results from the weighing of the wet and dry samples in order to calculate BD and MMC. Then, there is a widely acknowledged mathematical relationship MMC = 100/BD − 100/1.5, quoted in many publications on physical properties of an archaeological wood (e.g. Jensen and Gregory, 2006; Schniewind, 1990). A very good correlation ($-0.99 > R > -1$) between experimentally measured BD values and MMC values calculated from this equation was obtained both for wood with mineral compounds and for wood without mineral compounds. It is worth noticing that if we assume lower wood substance density the MMC values show only negligible changes (if WSD = 1.4 g/cm^3, MMC decreases by ca. 5%), and the correlation coefficient does not change significantly either. A similar high correlation coefficient ($R < -0.99$) has previously been found between basic density and porosity as well as between porosity and maximum moisture content (Babiński et al., 2012; unpublished report). As mentioned above, in fresh wood of a single species these properties can vary over a relatively wide range. This means that an evaluation of the degree of decomposition of archaeological wood on the basis of any of these properties might be affected by the error arising from the comparison of the present properties of archaeological wood with the average values characteristic for non-degraded wood. The scale of that error will depend on the difference between the average value for fresh wood and the actual properties (before decay) of the archaeological wood.

Dependences between basic density and wood substance density are presented in Figs. 2–4. In each of the diagrams there are four series of data corresponding to the dependence of BD of wood containing mineral compounds and substance density of wood with mineral compounds determined in water (WSD_w) and in helium (WSD_h) (data in Table 2), as well as BD of wood without mineral compounds and substance density of wood without mineral compounds, determined in water (WSD_w) and in helium (WSD_h) (data in Table 3). Correlation coefficients for the analysed series of data are presented in the legends for the individual diagrams.

Analysis of the data for all wood samples (Fig. 2) showed distinct differences in correlations between the investigated physical properties in the case of wood with mineral compounds and wood without this contamination. The correlation coefficients for the material which did not contain mineral contamination were nearly twice as big. A statistical linear dependence with a moderate degree of correlation was found in the case of wood substance density determined both in water ($R = 0.60$) and in helium ($R = 0.67$). In the next stage, the results obtained for hardwood (oak, beech, ash, alder, and elm) and softwood (pine and spruce) were separated. In the hardwood group, dependences between the tested properties remained at the level observed previously in the case of all tested samples combined. By contrast, in the case of softwood the R coefficients were distinctly lower, with the highest values being $R = 0.23$ and $R = 0.42$, as obtained for wood and wood substance without mineral compounds, determined in water and helium respectively. Due to the small quantity of samples tested, the collected data allowed for separate statistical analysis for only two species – oak (number of samples $n = 9$) and pine ($n = 10$). For oak wood, a strong correlation between the properties analysed was observed (Fig. 3). The highest value of R coefficient was obtained for material without mineral compounds when the WSD was determined in helium ($R = 0.82$). By contrast, data obtained for the pine wood was scattered (Fig. 4), which points to the lack of correlation between WSD and BD. It may be a result of the fact that among the

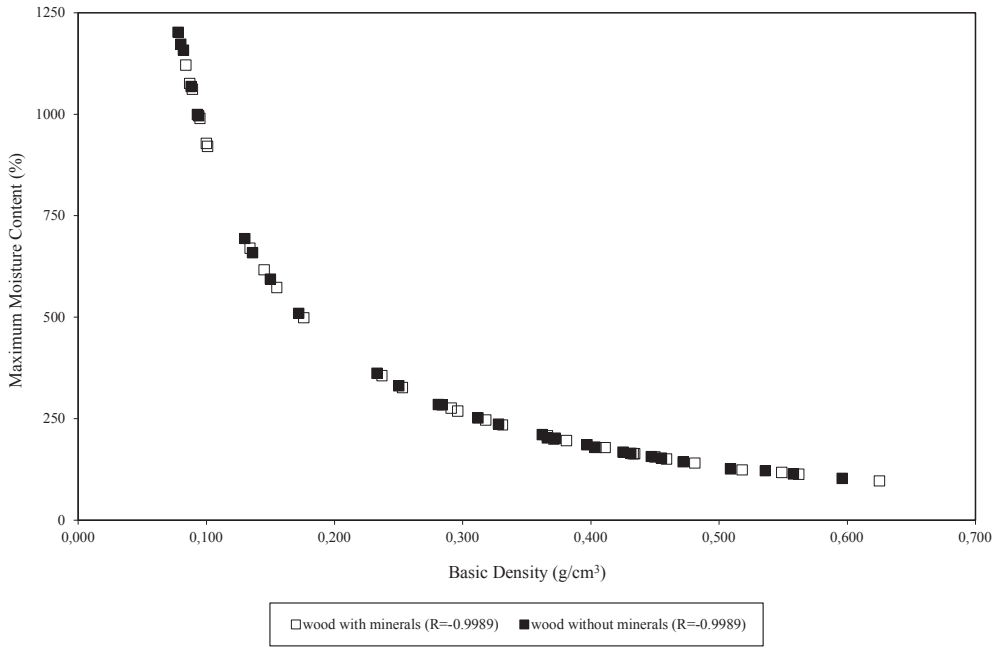

Fig. 1. Relationship between basic density and maximum moisture content for all examined wood samples with and without mineral compounds.

samples of pine wood there were more samples of wood in which the BD was higher than the average values for fresh wood (see LWS $< 0\%$ and RBD $> 100\%$; Table 3). It is possible that in the group of samples with higher degrees of degradation, the initial density of pine wood could also have been higher than the average value. A distinctly lower level of decomposition in comparison to oak wood could be also indirect proof for the lower correlation in the group of pine wood samples. However, a considerably stronger relationship between WSD and BD was observed in the case of spruce wood ($n = 5$), for which the R coefficient in the case of material without mineral compounds determined in helium was 0.54. It might point to the fact that the initial BD (before decay) of spruce wood was closer to the current average value than it was in pinewood. A higher variability of the initial BD of pine wood and related to that weaker correlation between WSD and BD might be accounted for by the greater proportion of late wood in individual samples. Another reason for the lower correlation coefficients for the pine wood could be a higher content of extractives soluble in organic solvents and/or greater variability of their content depending on the wood degradation and sampling place (sapwood and heartwood of softwood, and mostly heartwood of oak wood; Table 1).

Comparing correlation coefficients for the individual series presented in Figs. 2 and 3 one can see that the best dependences between BD and WSD were obtained in the case of the corrected

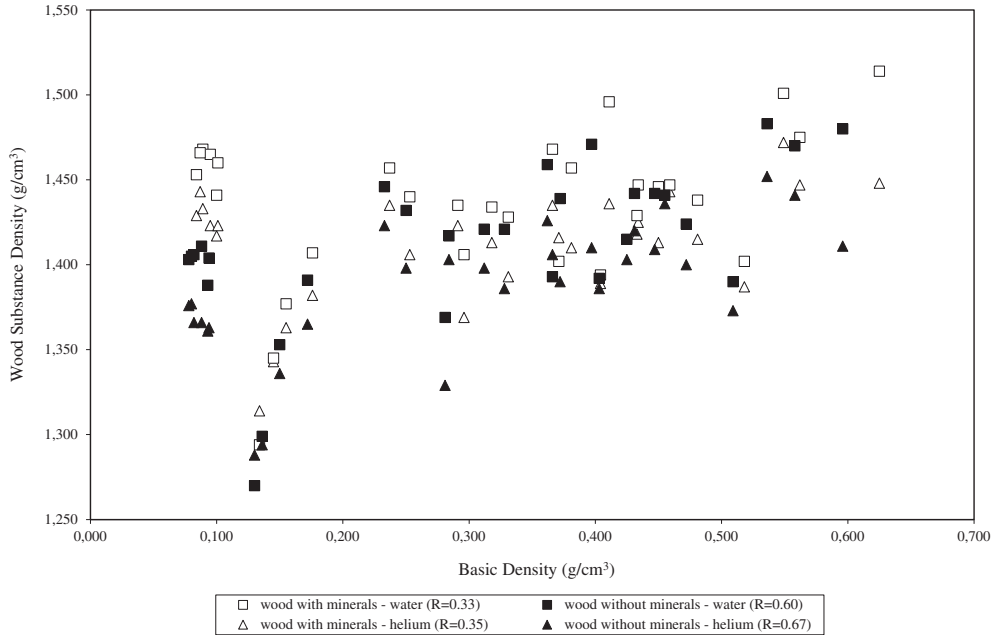

Fig. 2. Relationship between basic density and wood substance density for all examined wood samples.

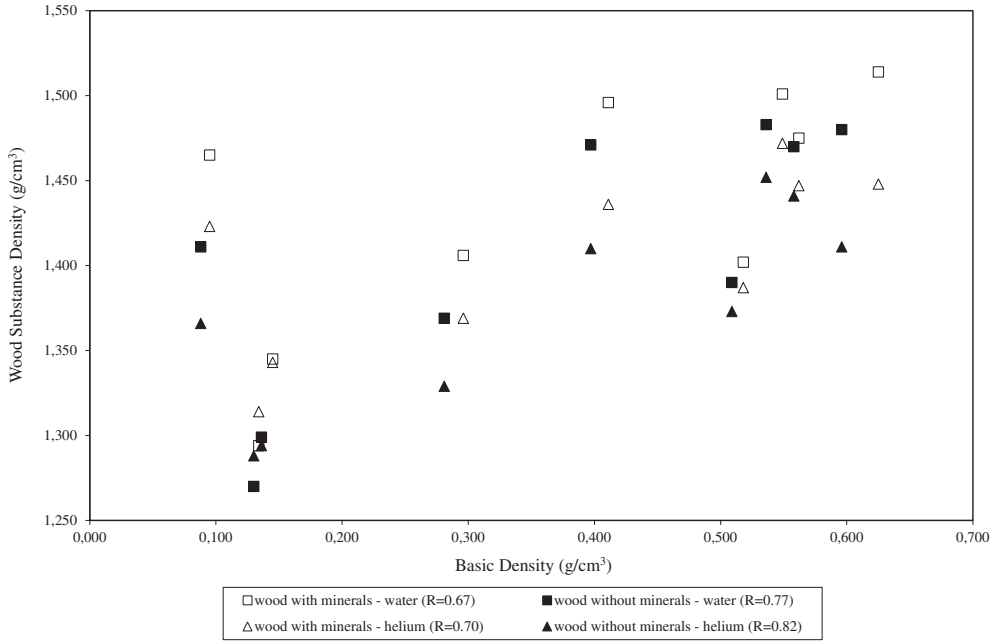

Fig. 3. Relationship between basic density and wood substance density for oak wood.

wood substance density (without mineral compounds) determined in helium. On the one hand this points to the necessity of making the correction of WSD after a determination of the mineral compounds, and on the other it confirms the advantage of the helium method over the traditional method of measuring density in water. Between the corrected wood substance density determined in water and helium one could, however, observe the following correlation: $WSD_h = 0.7598WSD_w + 0.3132$ with a very high correlation coefficient ($R = 0.92$) (Fig. 5).

The minor differences in wood substance densities observed in non-degraded (fresh) wood mean that a determination of the degree of decomposition of archaeological wood on the basis of the change in that property may be more reliable than an evaluation based on the basic density or maximum moisture content. However, when determining WSD, it is necessary to take mineral compounds into consideration as these can have a significant influence on the evaluation. The currently common evaluation of the state of wood preservation on the basis of BD and MMC can be erroneous due to the lack of consideration of the mineral compounds in archaeological wood.

An evaluation of the degree of decay of archaeological wood on the basis of the change in wood substance density can be carried out in a comparable manner to the loss of wood substance (loss of mass) suggested by Grattan and Mathias (1986). To do that, the loss

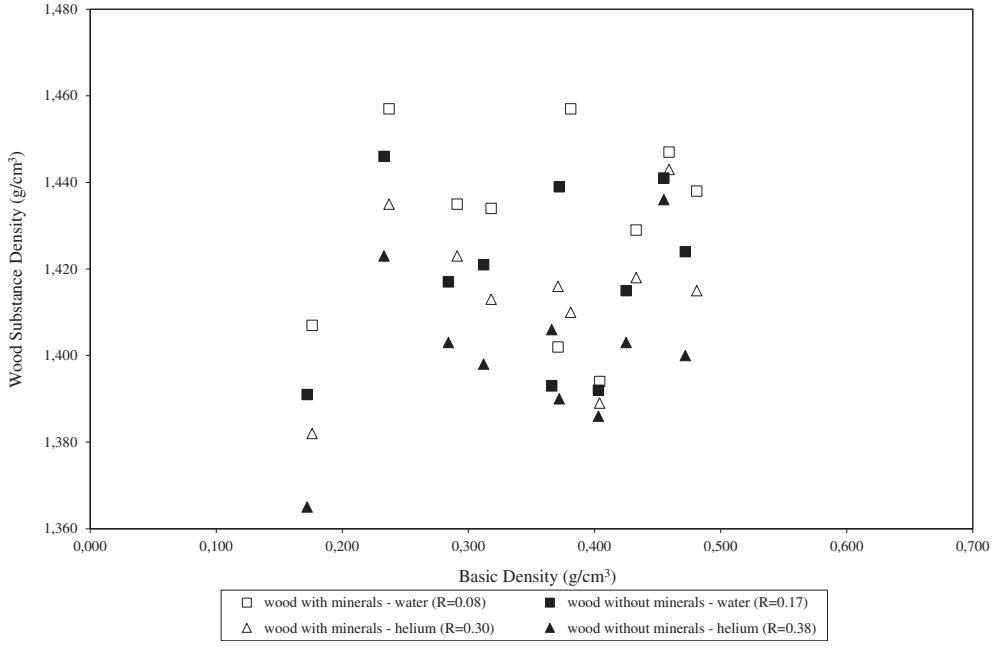

Fig. 4. Relationship between basic density and wood substance density for pine wood.

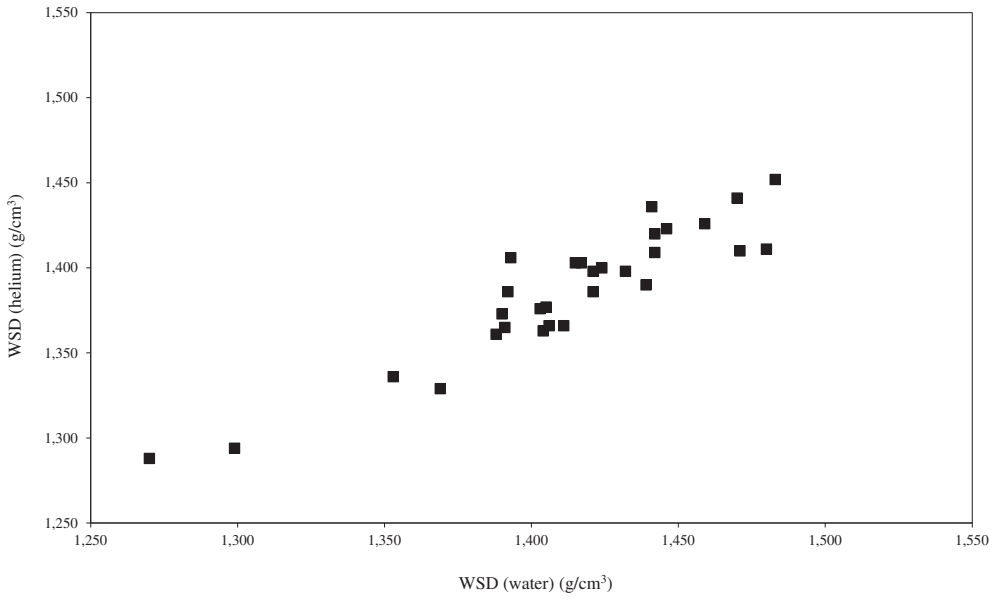

Fig. 5. Relationship between wood substance density determined in water and wood substance density determined in helium for all examined wood samples without mineral compounds.

of wood substance density was calculated according to the following formula:

$$\begin{aligned} LWSD &= 100^{*}\left(WSD_f - WSD_a\right)/WSD_f \\ &= 100^{*}(1.46 - WSD_a)/1.46 \end{aligned}$$

where

$LWSD$ – loss of wood substance density (%),
WSD_f – average wood substance density of fresh wood, determined in helium (1.46 g/cm^3),
WSD_a – wood substance density of archaeological wood without mineral compounds, determined in helium (g/cm^3).

The average wood substance density of fresh wood was calculated on the basis of density measurements of various wood species. The assumed WSD_f (1.46 g/cm^3) did not differ significantly from the published data (e.g. Christensen and Hergt, 1968; Wilfong, 1966). Nonetheless, it might have been slightly different from the unknown initial WSD of the tested archaeological wood. As such, LWSD calculated from the formula above should be regarded as an approximate value.

In the case of the most degraded archaeological wood, in which WSD measured in helium took values less than 1.300 g/cm^3 (Que-2 and Que-3), the suggested LWSD index takes on a value greater than 11%. A lower density of degraded wood substance than the average density of lignin could result either from a minor error in measurements or from the presence of the products of wood decomposition, which are characterized by low density.

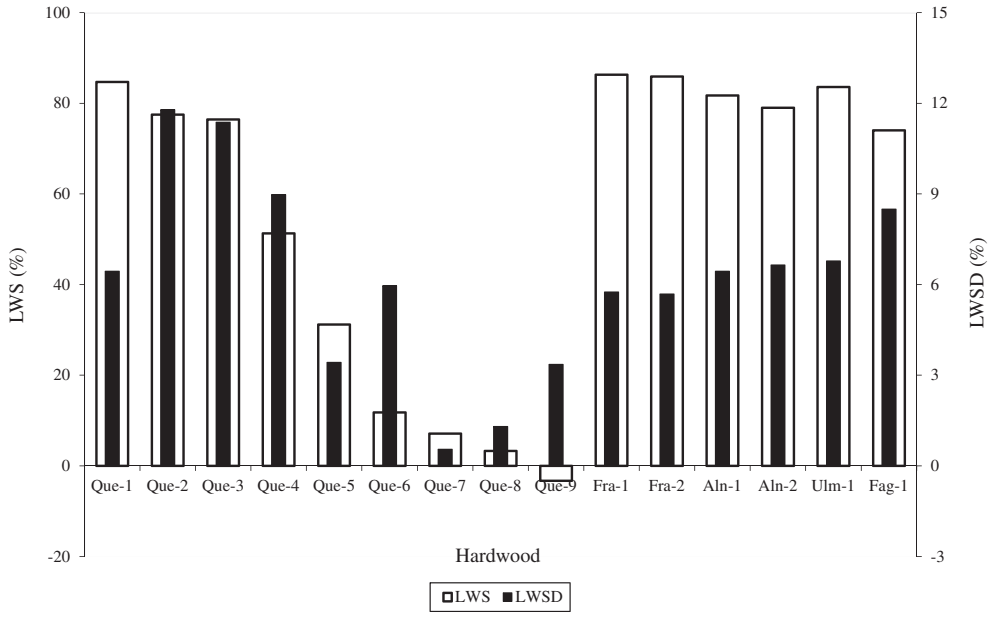

Fig. 6. Comparison of hardwood degradation assessed on the basis of loss of wood substance (LWS) and loss of wood substance density (LWSD).

Figs. 6 and 7 present the degree of wood decomposition assessed on the basis of the loss of wood substance (LWS) and the loss of wood substance density (LWSD). Differences were observed between the evaluated condition of wood preservation according to each index. In Table 3 a lower ordinal number for the samples of oak and pine wood indicates a higher LWS (as well as higher MMC and lower BD), pointing to a more advanced decay of wood. On the basis of LWSD, the most decomposed oak wood was represented by samples Que-2 (LWSD = 11.8%), Que-3 (11.4%) and Que-4 (9.0%), while the least decomposed ones were Que-7 (0.5%), Que-8 (1.3%), Que-9 (3.4%) and Que-5 (3.4%) (Fig. 6). In comparison with the evaluation performed on the basis of LWS a distinctly smaller degree of decomposition of oak wood (determined with the use of the LWSD) was observed for the sample Que-1 (LWS = 84.7%, LWSD = 6.4%), while considerably higher degree of decomposition was found for the samples Que-6 (LWS = 11.8%, LWSD = 6.0%) and Que-9 (LWS = −3.3%, LWSD = 3.4%). Nevertheless, the correlation coefficient between LWS and LWSD for oak wood was high ($R = 0.82$). The other samples of hardwood (alder, ash, beech, elm) were characterized by higher wood substance densities than one might have expected on the basis of the result of the evaluation performed with the use of LWS. The LWSD indices for hardwood samples from Gdańsk (Fra-1 and 2, Aln-1 and 2, and Ulm-1) took values comparable to the value obtained for the sample Que-1, drawn from the same site. For the Que-1 sample the WSD was also a little higher than in the case of the samples from Biskupin with a similar degree of decomposition. It could point to a different pattern of the process of wood decay in various environments. It can be supposed that the higher WSD for the extremely degraded objects from Gdańsk are a result of the decomposition (loss) of a relatively bigger quantity of lignin than in the case of wood from Biskupin. A larger degradation of lignin and the increase of the ratio H/L (holocellulose/lignin) could have an influence on wood substance density. Changes in molecular weight distribution of lignin (Colombini et al., 2009; Ferraz and Durán, 1995; Guerra et al., 2003) and greater loss of its lighter fractions should be also taken into account.

Lower WSD and higher LWSD point to the decrease in ratio of easily decomposing polysaccharides to more resistant lignin. However, it turns out that an evaluation of the degree of wood degradation on the basis of wood substance density can also be unreliable, especially if materials coming from various sites are compared. At the same loss of mass, wood substance density can assume various values depending on whether decomposition of carbohydrates was accompanied by decay of lignin. These differences, observed for wood coming from various environments, may prove that degradation of wood takes place according to various mechanisms. Tests on the chemical composition of wood can confirm it unequivocally. Nevertheless, even at the present stage, it should be considered that comparing the degree of decomposition of wood coming from various sites, based only on wood substance density, may turn out to be insufficient and testing should be complemented with other physical properties of the examined wood. Only completion of such an evaluation with the approximate loss of wood substance (loss of mass) determined on the basis of its basic density will provide more complete information on the scope, and even direction, of wood degradation.

The evaluation of the state of wood preservation based on both compared indices of degradation differs even more in the case of pine wood (Fig. 7, $R = 0.38$). The highest divergences were observed for samples Pin-2 (LWS = 44.3%, LWSD = 2.5%) and Pin-10 (LWS = −12.9%, LWSD = 4.1%). The low correlation coefficient between the analysed wood decomposition indices can be assumed to be the result of 1) comparing material which comes from various sites (different course of wood decay) and 2) of wood characterised by a large difference between its initial basic density and the average value assumed for that property. This can be shown by the negative values of LWS noted in the case of a few samples of softwood (Table 3).

4. Conclusions

Evaluation of the degree of degradation of waterlogged archaeological wood, performed in many conservation laboratories on the basis of its basic density and/or maximum moisture content, can be encumbered by certain errors. These are a result of comparing the properties of archaeological wood to the average

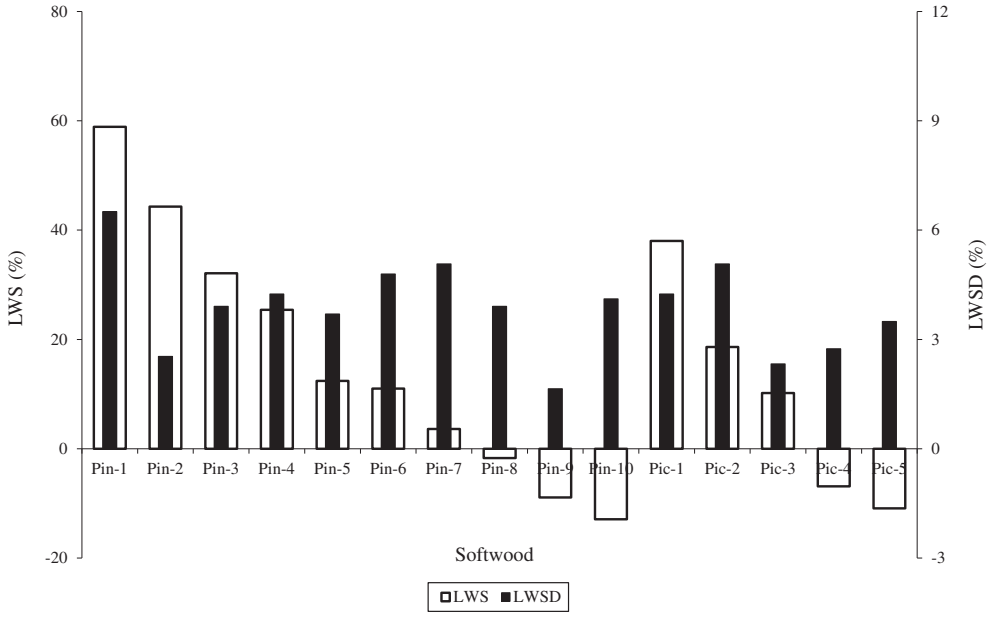

Fig. 7. Comparison of softwood degradation assessed on the basis of loss of wood substance (LWS) and loss of wood substance density (LWSD).

values characteristic of fresh material and not to the initial (unknown) properties of archaeological wood before its degradation. The state of wood preservation evaluated in this way can differ from the actual degree of decomposition of the wood tissue. This can be shown by the results of the tests presented in the article (LWS $< 0\%$ and RBD $> 100\%$). An incorrect evaluation of the degree of degradation can take place independently of the advancement of wood decay, and such an error can be easily recognised only in the case of material with a slight degree of degradation. Additionally, the physical properties under discussion are determined for material that includes wood and mineral compounds. The maximum moisture content is then lower and basic density is higher than the actual value (determined for wood without mineral compounds). So these measurements also influence the accuracy of the evaluation of the state of preservation of tested wood. However, determination of basic density enables the calculation of an approximate loss of wood substance, which is the simplest index of the degree of degradation of archaeological wood.

Mineral compounds in wood influence wood substance density even more strongly. Thus, to perform a reliable evaluation of wood degradation it is necessary to determine mass and volume of wood substance without mineral compounds. Decrease in wood substance density is a result of the decomposition of wood and the changing ratio of the content of carbohydrates (higher density and lower resistance to decay) to the content of lignin (lower density and higher resistance to decay). Unfortunately, the change in the ratio between the main chemical compounds fails to provide information about the loss of mass in the tested material. When the loss of carbohydrates and lignin stays at a similar level, even in highly decomposed wood the wood substance density might be similar to the substance density in fresh wood. However, confirmation of the existence of a strict relation between wood substance density and the chemical composition of archaeological wood (with regard not only to the main components, but also to the products of their decomposition) was not the aim of this study and it requires further detailed investigation.

In order to evaluate the state of preservation of waterlogged archaeological wood and the direction of the process of its decomposition, the index of the loss of wood substance density (LWSD) was introduced. Comparison of the evaluation of wood degradation on the basis of the loss of wood substance and on the loss of wood substance density shows differences that are particularly distinct for wood deposited at various sites (in various environments) and for the material with the highest degree of decomposition. In the case of the evaluation made on the basis of basic density, the lack of knowledge on the initial density of archaeological wood presents a serious obstacle, while in the case of using wood substance density, information on the loss of wood mass is missing. Still, a comparison of both indices (LWS and LWSD) provides some complementary information, which is useful in the complex evaluation of the state of preservation of wood and in the preparation of a procedure for its protection and conservation. Therefore, it would be advisable to evaluate the state of preservation of archaeological wood on the basis of both its basic density and wood substance density.

The study was conducted on a limited number of samples taken from the available archaeological objects. Wood of different species, degree of degradation (mainly in the case of oak and pine wood), age, origin and sampling place was tested. Nevertheless, it would be interesting to compare the results obtained with the results of measurements carried out on material taken from other archaeological sites, on more decomposed softwood as well as on wood with a higher content of mineral compounds. Depending on the availability of archaeological wood such a study will be undertaken in the future.

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