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THE SELECTED PROPERTIES OF FOSSIL OAK WOOD FROM MEDIEVAL BURGH IN PŁOŃSK

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ABSTRACT

Oak wood is characterized by a high resistance to effects of biotic factors and thereby it is often found in archaeological excavations. While lying over many years in the wet environment, the wood turns black as a result of reaction with iron compounds. Archaeological oak is a valuable raw material. In this paper selected mechanical and chemical properties of a thousand years archaeological oak were investigated. Then the archaeological oak properties were examined and compared with the contemporary oak wood. Archaeological oak wood as well as contemporary oak wood has a directly proportional relationship between MOE (modulus of elasticity) and wood density, and similar relationships between wood density and compressive strength, bending strength, content of mineral substances. Contemporary wood of the same density as the archaeological oak wood would show significantly higher mechanical properties.

KEYWORDS: Archaeological oak wood, physical, mechanical and chemical properties of wood.

INTRODUCTION

European oak wood (*Quercus robur* L.) belongs to group of species characterized by high natural durability of wood. According to EN 350-2: 1994 the heartwood of European oak belongs to second class (the wood is durable and it exhibits high resistance to biotic factors). This class does not allow for usage of mentioned wood directly outdoors in contact with the ground. However, this species can buried for many years in water or moist soil layers. Because of that, it is often found in archaeological excavations not only in Poland but also in Europe (Giachi et al. 2003; Haneca et al. 2009, Krutul and Kozakiewicz 1999, Krutul et al. 2010, Babiński et al. 2011, Kolář et al. 2012). Besides, it is nearly as resistant to biotic factors as charcoals (Jankowska and Kozakiewicz 2013). Main factors which affect the speed of change in physical properties and chemical composition of wood are biotic: fungi, bacteria and abiotic: pH, temperature, exposure to oxygen and water (Sandström et al. 2004). Their impact on change of wood properties was described by Jensen and Gregory (2006). There are only three types of bacteria which are capable to degrade wood in such conditions - erosion, tunnelling and cavity bacteria (Daniel et al. 1998, Kim and Singh 2000, Singh 2012). In wet environment the oak wood is a subject of progressive changes: gray degradation caused by bacteria anaerobic, hydrolytic degradation, leaching of unstructured substances and mineralization. The first three lead to reduction of wood density, but mineralization has opposite effect (Dzbeński 1970, Kozakiewicz and Matejak 2013). Mechanism of formation of archaeological oak is not fully understood. The black colour of wood is mainly as a result of increased content of iron (Krutul and Kozakiewicz 1999, Krutul et al. 2010, Mańkowski et al. 2013, Rüther and Jelle 2013). The intensity of this process depends on the concentration of minerals in the ground and the possibility of contact with the structure of the wood (Sandström et al. 2007, Zborowska et al. 2007). This process does not occur uniformly (Reinprecht 1992, Krutul and Kozakiewicz 1999). The consequence of chemical changes in the old wood is a change of its physical and mechanical properties, which depends on the environment where the timber remained (Dzbeński 1970, Borgin et al. 1979, Babiński et al. 2011 Kolář et al. 2014). The archeological oak wood acquired from wet environment may have similar properties as a wood of contemporary oak. In dry-air conditions density and strength of the archeological oak wood allows to perceive it as a valuable material.

MATERIAL AND METHODS

The archaeological oak wood was excavated from fluvial terrace of Płonka river during archaeological explorations in the area of medieval burgh in Płońsk in the nearby of so-called Kaban Hill. Dating the ornaments and pieces of ceramic found in excavation allowed to date the other findings as 12th century objects (Bednarczyk 2009). The wood was acquired (in wet state) from full-scale constructional elements of the settlement. They had been used as the part of the stockade of significant borough object - probably some kind of a temple or a place of worship. Among the archaeological wood there were - inter alia - 2-meter-long carved and halved oak stems with sharpened ends. Four elements were chosen for further investigation. The pales had been made from full-scale stems which nearly full sections contained tens to hundred and more growth rings. The dominant heartwood permeated with iron compounds had intense dark colour (gray-black) and the brighter tiny sapwood was decayed to high extent by microorganisms.

Physical properties

Average width of annual rings and percentage of late wood were assessed according to PN-D-04110: 1955. The study included only full annual rings, which were visible in transverse sections of samples prepared for compressive strength tests.

On the basis of the measured data the average width of annual rings at the measuring section was calculated according to Eq. 1:

$$b_{AV} = \frac{b_w + b_p}{n} \tag{1}$$

where: b_{AV} - average width of annual rings at the measuring section (mm),
b_w - the total width of the early wood at the measuring section (mm),
b_p - the total width of the late wood at the measuring section (mm),
n - the number of annual rings at the measuring section,

The percentage of late wood was calculated according the Eq. 2:

$$U_p = \frac{b_p}{b_w + b_p} \tag{2}$$

where: Up- percentage of late wood (%)

For all samples the density of wood was determined (ISO 3131: 1975) and after the strength tests also the moisture content was specified (ISO 3130: 1975).

The volumetric swelling (ISO 4860: 1982) was investigated and the technical properties were evaluated using the shortened method by the five-point scale developed by Dzbeński (1970). For this purpose the coefficient S_1 was calculated according to Eq. 3 (Dzbeński 1970):

$$S_1 = (K_{FosV} - K_{ConV})/K_{ConV} \tag{3}$$

where: K_{FosV} - volumetric swelling of archaeological oak wood,
K_{ConV} - volumetric swelling wood of contemporary calculated using the equation,
$K_{ConV} = 0.025 \bullet \rho Con$ (ρCon - density of contemporary oak wood in an absolutely dry state).

Mechanical properties

Mechanical properties examination was performed on the Instron 3382, using the computer program "INSTRON SERIES IX/S". Archaeological oak had been mild dried and aged (over a year) to obtain a moisture content of approx. of 12 % (ISO 3130: 1975). From such prepared material there were obtained samples for each test: MOE and static bending strength (ISO 3133: 1975) – sample size 20x20x300 mm.

For testing the compressive strength along fibres (ISO 3787: 1976) there were used the ends of samples previously tested for static bending strength – their size was 20x20x60 mm.

For comparison, the strength quality factor was calculated (for compression) using the following Eq.4:

$$W_j = R/\rho_{12\%}, \tag{4}$$

where: R - comprehensive strength of wood,
$\rho_{12\ \%}$ - density wood of a moisture content MC=12 %.

Archaeological oak results were compared with the results of contemporary oak wood. In investigation there was used the heartwood without visible defects acquired from 150-years-old sessile oak (*Quercus robur* L.). This wood was collect from forest near Płońsk city. The trees grew in fresh forest on brown soil.

Chemical analysis

After compressive strength measurement samples were fragmented to sawdust and fractionated by sieves. Two fractions of oak wood were used for chemical analysis. First of them was isolated and conducted from sawdust fraction passing the 1.02 mm sieve and remaining on 0.43 mm sieve. This material was pre-treated by chloroform-ethanol mixture according to method (Antczak et al. 2006). After that sawdust without extractive compounds was treated by 1 % NaOH solutions. Three analysis were made for each sample of oak wood. The samples were dried before experiment. Wood material was boiled in 95°C in 100 cm^3 of 1 % NaOH solution for one hour (Kačik and Solar 1999).

Second fraction which was collected on the mesh sieves was smaller than 0.43 mm and was used to determine mineral substances in wood. 5 g of dried dust samples were placed in porcelain dish (formerly combused in 600˚C). Then they were placed in muffle oven. Heating started from 100˚C degree. After one hour the temperature was raised up to 200˚C. In this temperature samples were outstayed for one hour. Next steps were similar. When the temperature in oven reached 600˚C the samples were combused as long as they have constant weight (PN-92 P-50092: 1992).

RESULTS AND DISCUSSION

Test results are shown in Figs. 1, 2, 3, 4, 5 and Tab. 1.

Investigated wood of archaeological oak is characterized by higher density (approx. 850 kg•m^{-3}) than the contemporary oak wood used for comparisons (670 kg•m^{-3}) It is also higher than the average oak wood density reported in the literature (690 kg•m^{-3}) (Wagenführ 2007). The tree-ring width of archaeological wood was significantly diversified and its value varied from 0.6 to 4.0 mm depending on the element (with dominance of narrow annual rings – the average value was 1.4 mm). There is known relationship between the width of annual rings and the density of wood, determined by variable percentage of late wood in ring porous species. The density of contemporary oak wood with 1.2 mm tree-rings width in air-dry condition is approximately 600 kg•m^{-3} (Kollmann and Côte 1984). The distribution of density of samples was very close to normal distribution.

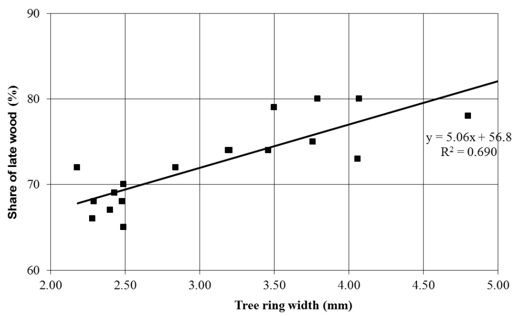

Fig. 1: Relationship between share of late wood and tree ring width.

In the Fig. 1 the relationship between share of late wood and tree ring width is presented. There is also visible a significant correlation between measured entities (R^2=0.69). This function is described by the formula Y=5.06x+56.77. The result is similar with the effects of former study (Dzbeński 1970).

Density of the investigated archaeological oak wood was 250 kg•m^{-3} higher than it is implied by the average width of annual rings, whereas in the contemporary reference sample the proper relationship was preserved. The increase of archaeological oak wood density over the average of typical contemporary wood is a result of its strong supersaturation of mineral compounds including iron compounds (Mańkowski et al. 2013). The less significant reason could be external organic and inorganic inclusions (Dzbeński 1970, Waliszewska 2009).

Tab. 1: Comparison of selected properties of archaeological and contemporary oak wood.

Property	Unit	Research material	Number of samples	Value			Standard deviation
				Min	Average	Max	
Width of annual rings	(mm)	Contemporary oak	26	1.3	2.1	2.9	0.50
		Archaeological oak	57	0.6	1.4	4.0	1.01
Density	(kg•m^{-3})	Contemporary oak	135	618	662	711	146
		Archaeological oak	274	596	846	1 049	108
Volumetric swelling	(%)	Contemporary oak	26	12.1	13.2	13.8	0.61
		Archaeological oak	57	17.3	20.5	26.3	2.14
MOE	(MPa)	Contemporary oak	30	8 500	10 050	11 750	750
		Archaeological oak	67	4 300	9 600	13 300	2 300
Static bending strength	(MPa)	Contemporary oak	50	101	121	145	10
		Archaeological oak	67	58	125	175	31
Compressive strength	(MPa)	Contemporary oak	105	60	72	84	6.0
		Archaeological oak	237	52	80	105	11
Content of mineral substances	(%)	Contemporary oak	-	-	0.38*)	-	-
		Aarchaeological oak	15	1.42	1.86	2.92	0.38
Content of substances soluble in 1% NaOH	(%)	Contemporary oak	-	-	23.1*)	-	-
		Archaeological oak	9	18.8	20.0	21.9	0.01

* Krutul 1996

Fossil oak wood has considerably higher tendency to swell and shrink. Average volumetric swelling of archaeological oak wood was 20.5 % (in the radial direction - 6.8 %, in the tangential direction - 13.1 %), whereas in contemporary oak wood with a larger tree-ring width (so theoretically it should be more susceptible to dimensional changes) volumetric swelling was only 13.2 %. It is confirmed by the results of previous papers, which indicate greater sensitivity of archaeological wood to dimensional changes (Dzbeński 1970, Borgin et al. 1979, Babiński et al. 2011, Kolář et al. 2014). Significant deformations of the excavated oak are associated with progressive hydrolysis and depolymerization of cellulose in the cell wall, which are a result of staying in wet environment, (Grattan and Mathias 1986, Waliszewska 2009). These changes increase the sorption potential of wood substance. Higher concentrations of secondary products of cell walls decomposition leads to increase in vapour and free water absorbency of the wood tissue, which – as a result of this process - abnormally increases its size. It is facilitated by the plasticizing of wood structure (Dzbeński 1970). Increased values of archaeological oak wood contraction reduced the average widths of tree rings width measured in wood of 12 % moisture, hence - assuming that original average of tree rings width was proportionally larger - increase of density as the result of mineralisation probably was smaller - it means about 150 kg•m^{-3}, not 250 kg•m^{-3} as it had been formerly assumed.

The analysis of the values presented in Tab. 1 indicates the high variability of mechanical properties of archaeological oak wood as compared to contemporary wood. This variability arises not only from the increased volatility of tree-rings width but also the mineral compounds supersaturation (Mańkowski et al. 2013). The average value of the modulus of elasticity of archaeological oak wood is lower than the contemporary one, and the range of variability of this feature is significantly greater and understated in comparison to literature data. According to Wagenführ (2007) the MOE of oak wood (including pedunculate and sessile oak) in air-dry condition typically ranges from 9 200 MPa to 13 500 MPa.

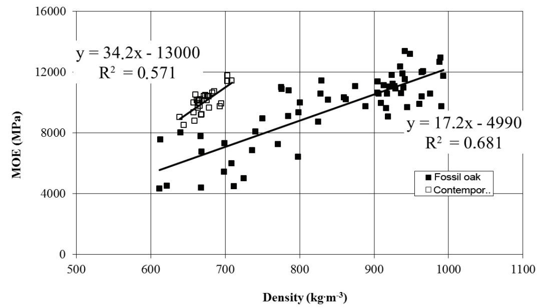

Fig. 2: Relationship between bending MOE and the density of wood in air-dry condition (moisture content of wood =12 %).

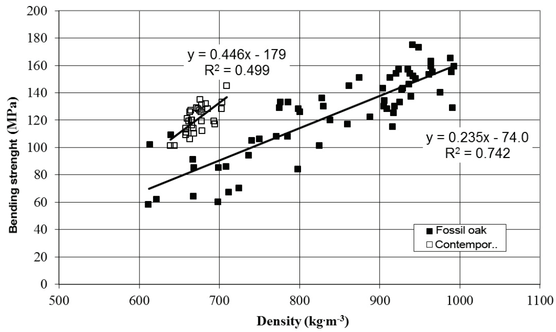

Fig. 3: Relationship between static bending strength and density of wood in air-dry condition.

Calculated values of average bending strength and compressive strength along fibres of archaeological oak is higher in comparison to contemporary wood (Tab. 1). However, considering the level of density these results look differently (Figs. 1, 2, 3). Compared to modern oak wood of the same density, archaeological oak has over 40 % reduced modulus of elasticity and the static bending strength (Figs. 2 and 3). Similar reduction in the archaeological oak MOE (36 %) had been discovered in previous studies (Kolář et al. 2014).

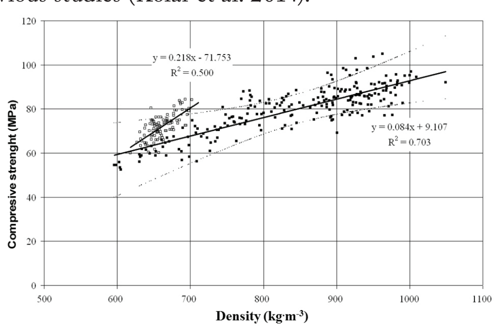

Fig. 4: Relationship between compressive strength along the fibres and the density of the timber in air-dry.

The relationships describing compressive strength along the fibre (Fig. 4) behave similarly. The differences of the coefficients of the linear function approximating the relationship between compressive strength along the fibres and the density are significant.

Equation of linear function for contemporary oak is a Rc = 0.217* g-71.8, and for archaeological oak: Rc = 0.084* g + 9.1. First equation matches simple correlation equation (Rc = 0.158* g-62.0) obtained by Vorreiter (1949) and the equation (Rc = 0.1204* g-24.387), obtained by Kolář et al. (2014) for the oak wood and the other results of the same authors (Kolář et al. 2014) for archaeological oak Deposited in Holocene sediments (Rc = 0.0639* g-4.7828). The differences in compressive strength between contemporary and archaeological oak wood are statistically significant (t Stat = 8.171).

Archaeological oak wood is characterised by several times higher percentage of mineral substances then contemporary oak wood (Tab. 1). This proportion is consistent with result obtained earlier by several different authors (Kúdela and Reinprecht 1990, Krutul et al. 2010) and data which are result of research on content of mineral substances in archaeological oak wood deposited in holocene near old village Biskupin situated in Central Poland (Babiński 2005) – there it was estimated between 1.93 and 2.12 (Kolář et al. 2014), the result of our research is 1.9. The percentage of mineral substances in heartwood of contemporary oak is less than 0.5 % (0.2-0.4) (Krutul 1996, 1997). Different authors as Wagenführ (2007) during their research obtained quite higher concentration of mineral substances (ash) (between 0.4 % and 0.6 %). Probably these results were obtained for samples acquired only from heartwood but also sapwood:

Research indicates directly proportional relationship (r=0.720) between concentration of ash and compressive strength along fibres (Rc = 33.3•UA + 9.33 (MPa), where UA – percentage of mineral substances). This is a consequence of strong correlation between content of ash in wood and its density: UA = 0.004•g -0.886 (r=0.801). Content of compounds which are soluble in 1 % NaOH in archaeological oak wood from Płońsk averaged 20.0 %. This result is little lower compared to contemporary oak wood (23.1 %). Much higher result was obtained (more then 27 %) in contemporary oak heartwood which contained knots. In this case the different procedure of investigation can had influence on the difference between results (PN-92 P-50092). Content of compounds which are soluble in 1 % NaOH solution can be higher if the sapwood is considered (Wagenführ 2007). Comparative research on archaeological oak and contemporary oak heartwood wood (Krutul et al. 2010) indicated similar amount of compounds soluble in 1 % NaOH (21.5 - 23.1 %). Probably two processes took place in excavated oak wood from Płońsk as a result of centuries-long stay in moist soil. Water could dissolve some compounds of low molecular mass which had been formerly degraded. One of the fastest degrading substances in oak wood are xylans which has lower resistance to degradation processes then other hemicelluloses included in hard wood (Witomski and Babiński 2009). These factors also contributed to change in structure of cellulose – its depolymerization. Occurrence of iron ions in ground caused slow hydrolysis of cellulose chain (Florian and McCawley 1977). As a result it is possible to dissolve hemicelluloses and short chain of cellulose during the treatment wood in 1 % NaOH solution. In such conditions (long time in wet ground) hydrolysis processe is very slow. In this case ratio of soluble substances in 1 % NaOH to the rest of compounds in wood is very similar to the content in contemporary oak wood. Lack of dependency between density and content of mineral substances is a result of small changed in wood chemical compounds (very low decline of substances soluble in 1 % NaOH and minimal growth mineral substances weight). These changes were too small to have any influence on statistic correlation between density of wood and content of mineral substances.

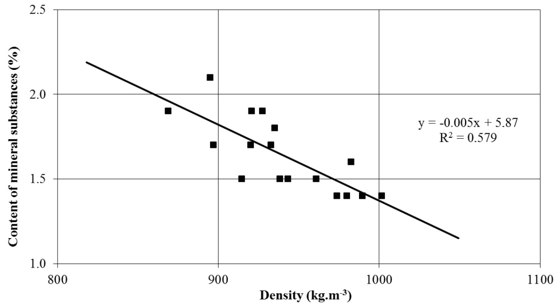

Fig. 5: Relationship between content of mineral substances and density.

The density of archeological oak is inversely proportional to the content of mineral substances (Fig. 5). The reason for that is growth of wood density accompanied by the decline of its porosity, which leads to lower amount of water (and mineral substances dissolved in it) which can penetrate the wood. The water evaporates in drying process but the mineral substances remain in wood.

Quality indicator for compressive strength of archaeological oak wood averaged 9.5 km and for contemporary wood 10.8 km. This parameter indicates the relatively small decrease in the quality of archaeological wood. Contemporary wood with a width of annual increments of 1.2 mm and a density of approx. 600 kg•m^{-3} would have a volumetric swelling of approx. 15 %.

Using the method of shortened assessment of the technical characteristics of archaeological wood elaborated by Dzbeński (1970), which is based on comparison of the of swelling size of archaeological wood with contemporary wood of the same annual rings width, the parameter S was calculated. Value of the parameter S for archaeological oak wood from Płońsk is 0.36 (Class I according to Dzbeński (1970). It means that the wood excavations preserved very good condition (in terms of technical applications practically does not differ from contemporary oak).

CONCLUSIONS

Based on extensive investigations and analysis the following conclusions were formulated:

1. Modulus of elasticity, static bending strength, compressive strength along the fibres are significantly dependent and directly proportional to the density of contemporary and archaeological oak wood. Correlation coefficients of simple equations related to wood contemporary oak, and the archaeological one are significantly different.
2. Archaeological oak has lower modulus of elasticity, bending strength and compressive strength along the fibres in comparison to contemporary oak wood having the same density.
3. There exists an important and directly proportional relationship between the content of ash in the archaeological oak wood and the density and compressive strength along fibres.
4. The content of soluble compounds in 1 % NaOH from archaeological oak wood is almost a constant value, independent from the density of the wood and it does not correlate with the ash content and strength characteristics.
5. Archaeological oak wood from Płońsk which stayed for centuries in the moist soil environment, according to the definition of volumetric swelling in relation to the density, preserved very good technical condition.

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40. ISO 3787: 1976: Wood - Test methods - Determination of ultimate stress in compression parallel to grain.
41. ISO 4860: 1982: Wood – Determination of volumetric swelling.
42. PN-D-04110: 1955: Physical and mechanical properties of wood. Examination of percentage share early and late wood. (Fizyczne i mechaniczne własności drewna. Badanie procentowego udziału drewna wczesnego i późnego) (in Polish).
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