

## Correlations between Oak Wood Properties

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Correlations between the chemical composition and physical properties of oak wood were studied by correlation analysis. The specimens were produced from thermally treated oak wood at temperatures of 20 °C, 160 °C, 180 °C, 210 °C, and 240 °C. The physical properties were affected by the chemical composition of oak wood. The correlations of equilibrium moisture content (EMC) and oak density at EMC were similar, in accordance with the investigated properties. Oak end hardness was affected by treatment temperature. The depth of indentation significantly affected the hardness. Mass specific heat capacity and effusivity were positively correlated with EMC, sugars, holocellulose, cellulose traits, and ash and negatively affected by total extractives. Thermal diffusivity was slightly affected by treatment temperature.

*Keywords:* Oak wood; Heat treatment; Correlation; Chemical composition; Macromolecular traits of cellulose; Physical properties

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### INTRODUCTION

The logging and utilization of oak wood are traditional and have been known since ancient times (Sinković *et al.* 2009; Welling *et al.* 2018). Recently, Ruiz-Aguino *et al.* (2018) mentioned that approximately 400 species of *Quercus* are widely recognized. From a macroscopic point of view, surely Turkey oak wood can be recognized, relative to other oak woods, based on the color and morphology of the wood rays. Turkey oak is less utilized because it is prone to cracks and possible formation of false heartwood. Some research has been directed towards the processing of Turkey oak wood (Tolvaj and Molnár 2006; Todaro *et al.* 2012) with a goal to find the optimum procedure for Turkey oak wood valorization (Ferrari *et al.* 2013). The associated problems with the growth and utilization of Turkey oak are not as frequent as pedunculated or sessile oaks (Makovíny 2010). This probably was the reason for the decision to cultivate oaks in Slovakia. Currently, most oak wood production comes from pedunculated or sessile oak species in Slovakia (MARD 2017).

Oak wood properties are widely appreciated. The measurements of some wood properties are difficult; therefore, there is an effort to correlate the properties and determine their values indirectly. Moreover, the overall character of wood is characterized by a set of independent properties. The correlations clarify the behavior of wood after processing. The elevated surrounding temperature and the possible heat flow to wood volume is an example of such processing (Militz 2002; Gérardin 2016). The different procedures in the cited work showed different results for wood properties and the wood exhibited more diversity and utilization possibilities. The chemical composition influences wood properties. For example, the equilibrium moisture content of wood is proportional to free hydroxyl groups

in the amorphous part of cellulose (Babiak 1991). The hypothesis is that a positive correlation will be found between wood density at equilibrium moisture content (EMC) and EMC itself.

Wood is composed of molecules having different mass. Smaller molecules are able to move faster than the larger ones. Also, thermal diffusivity is proportional to the speed of the isothermal surface (Lykov 1968). It is hypothesized that a negative correlation can be found among the amount and thermal effusivity of wood low mass molecules, which is inversely proportional to thermal diffusivity.

The correlations elucidate the behavior of wood after processing. For example, the thermally modified wood must be machined carefully because after high-speed machining, evidence of deterioration will be apparent (Kaplan *et al.* 2018). Therefore, the hypothesis about the hardness of thermally modified wood as a property influenced by treatment temperature has been supported by some evidence.

The aim of this work is to verify the above mentioned introductory hypothesis about oak wood properties associations by correlation analysis. The larger aim is to gain increased understanding of the variability of oak wood properties values and thereby increases its possibility to be successfully utilized.

## EXPERIMENTAL

### Materials and Methods

Oak wood (*Quercus robur* L.) was obtained from Budča, Central Slovakia. The age of the logging wood was 96 years with a diameter of 40 cm. The final moisture content of 10% for flat sawn timber dimensions of 500 mm per 25 mm in thickness was achieved by the kiln drying method at Technical University in Zvolen. Then hydrothermal treatment was performed in the Volga State University of Technology in Joškar-Ola, Russia. Ten pieces of timber were thermally treated according to the following procedure:

The 1<sup>st</sup> period: the lumber was oven-dried in the environment of air at a temperature of 105 °C to constant mass for 24 h.

The 2<sup>nd</sup> period: the lumber was heated to treatment temperature (160 °C, 180 °C, 210 °C, and 240 °C) for 5 h.

The 3<sup>rd</sup> period: the lumber was cooled to an ambient temperature of 20 °C and target moisture content was reached up to the desired value by the water spraying method.

**Table 1.** The Schedule of Heat Treatment

Temperature (°C)	1 <sup>st</sup> Period (h)	2 <sup>nd</sup> Period (h)	3 <sup>rd</sup> Period (h)
160	4	5	2
180	5	5	2.5
210	6	5	3
240	7	5	3.5

The treated oak timbers were equilibrated with humid air in a climatic chamber Binder KBF 720 (Tuttlingen, Germany). The controlled parameters of humid air were at a relative humidity of 65% and a temperature of 20 °C. The equilibrium was detected according to the constant mass of timber using technical balances Kern KB 1000-2 (Balingen, Germany). The equilibrated oak was used for specimen production.

### Chemical composition

Untreated (20 °C) and thermally treated samples were disintegrated to sawdust. Particles from 0.5 to 1.0 mm were extracted in the Soxhlet apparatus by a mixture of ethanol-toluene according to the ASTM D1107-96 (2013). The extractives content soluble in cold water was determined according to ASTM D 1110 (2007). Lignin content was determined according to ASTM D1106-96 (2013). Holocellulose was determined using the method of Wise *et al.* (1946). All measurements were performed on two replicates per sample. Data were presented as percentages of oven-dry weight per unextracted wood.

The ash content was determined according to Sluiter *et al.* (2008) with two replicates per sample.

Wood samples were hydrolyzed with 72% (w/w) sulfuric acid. Quantitative analyses of the saccharides were carried out by high-performance liquid chromatography (HPLC) using an Agilent 1200 chromatograph (Agilent Technologies, Santa Clara, CA, USA), equipped with a refractive index detector and an Aminex HPX-87P column (Bio-Rad Laboratories, Hercules, CA, USA), at 80 °C. Ultrapure water was used as the mobile phase at a flow rate of 0.6 mL/min. Measurements were performed on four replicates per each treatment condition.

### Macromolecular changes of cellulose

Cellulose was isolated using the Seifert method (Seifert 1956). Size exclusion chromatography analysis of the cellulose isolated from the samples was performed after their conversion into cellulose tricarbonylates. Cellulose tricarbonylates were dissolved in tetrahydrofuran and filtered through a Puradisc 25 NYL filter (Whatman International, Maidstone, UK) with a pore size of 0.45 µm. Size-exclusion chromatography was performed at 35 °C with tetrahydrofuran at a flow rate of 1 mL/min<sup>-1</sup> on a PLgel, 10 µm, 7.5 × 300 mm, MIXED-B column (Agilent) preceded by a PLgel, 10 µm, 7.5 × 50 mm, Guard-column (Agilent) as described by Kačík *et al.* (2009). Data acquisitions were carried out with ChemStation software (Agilent), and calculations were performed with the Clarity GPC module (DataApex, Prague, Czech Republic). Measurements were performed on four replicates per each treatment condition.

### Density, hardness and moisture content

Density ( $\rho$ ) at equilibrium moisture content was measured in accordance with STN 490108 (1993) and calculated as follows,

$$\rho = \frac{m}{LRT} \quad (1)$$

where  $m$  is the mass of the specimen and  $L$ ,  $R$ ,  $T$  are parallelepiped specimen dimensions in the longitudinal, radial, and tangential directions, respectively. The EMC was measured in accordance with STN 490103 (1979).

The moisture content ( $w$ ) was measured as follows,

$$w = \frac{m - m_0}{m_0} \quad (2)$$

where  $m$  is the mass of the specimen and  $m_0$  is the specimen oven dry mass.

Janka's end hardness ( $H$ ) was measured on a testing machine with a steel ball constant speed of 5 mm·s<sup>-1</sup>. The specimens had the same dimensions as for density at EMC testing. The force ( $F$ ) was recorded every second until 2.82 mm depth was reached. Janka's hardness  $H$  was computed according to Eq. 3,

$$H = \frac{F}{2Rh - h^2} \quad (3)$$

where  $h$  is the indentation depth, and the steel ball radius  $R$  is equal to 5.64 mm. The computed values were not recalculated to 12% moisture content. Instead, the values were compared at EMC.

#### *Thermal properties*

Mass specific heat capacity, thermal conductivity, thermal diffusivity, and effusivity were measured by the quasi-stationary method (Regináč and Babiak 1977; Hřčka and Babiak 2012). The thickness of specimens was oriented in the radial direction and measured by digital calliper Mitutoyo CD-15DCX (Kawasaki, Japan). The density at EMC and EMC measurement rules are detailed in STN 490108 (1993) and STN 490103 (1979).

#### *Correlation analysis*

Correlations were used to detect a predictive relationship between selected physical properties and the chemical compositions of heat treated oak wood. The magnitude of the correlation coefficient indicated the strength of the association, and the sign of the correlation coefficient showed the direction of the association. Statistical testing based on the correlation was realized by Statistica 12.0 and MS Excel 2016.

## RESULTS AND DISCUSSION

The changes in chemical composition of oak wood before and after heat treatment are shown in Table 2. The relative content of the lignin and total extractives increased with the heat treatment. However, there was a decrease in the relative content of holocellulose, water extractives, and ash in comparison to the untreated wood. Esteves *et al.* (2008) studied the content of extractives and the composition of heat treated wood. They found that almost all of the original extractives disappeared, and new compounds were formed including monosaccharides and their dehydration products, as well as syringaldehyde, syringic acid, and sinapaldehyde as the most prominent compounds.

**Table 2.** The Main Wood Component Contents of the Oak Wood

Temperature (°C)	Water Extractives (%)	T-E Extractives (%)	Total Extractives (%)	Holocellulose (%)	Lignin (%)	Ash (%)
20	7.45 (0.02)	2.55 (0.08)	10.00 (0.06)	67.65 (0.23)	25.10 (0.05)	0.20 (0.04)
160	7.25 (0.06)	3.96 (0.09)	11.21 (0.15)	66.46 (0.11)	22.84 (0.05)	0.15 (0.01)
180	6.50 (0.01)	4.97 (0.16)	11.47 (0.15)	60.68 (0.11)	24.84 (0.09)	0.10 (0.02)
210	5.39 (0.05)	8.86 (0.06)	14.25 (0.01)	53.43 (0.07)	25.13 (0.18)	0.08 (4E-03)
240	1.89 (0.03)	12.23 (0.04)	14.12 (0.07)	44.85 (0.20)	31.80 (0.40)	0.09 (0.01)

Note: T-E Extractives are toluene-ethanol extractives. Data represent means; numbers in parentheses represent standard deviation.

The total polysaccharide fraction of wood (cellulose and hemicelluloses) is holocellulose. In the treated samples, the relative contents of holocellulose decreased by 1.76% (160 °C), 10.3% (180 °C), 21.0% (210 °C), and 33.7% (240 °C). The fastest degradation of holocellulose started at the temperature 180 °C. Its decrease was due to the degradation of hemicelluloses. Hemicelluloses are less stable than cellulose due their amorphous structure and are therefore more affected by heat treatment (Yildiz *et al.* 2006). The hemicellulose monomers, particularly xylose, arabinose, mannose, and galactose, noticeably decreased after the heat treatment, while glucose content increased slightly to the temperature 210 °C compared with untreated wood, then its content was reduced (Table 3). The saccharide content at the most severe conditions in comparison to the untreated decreased in the following order: arabinose = galactose > mannose > xylose > glucose. Glucose reduction was 10.6%. Windeisen *et al.* (2007) found that the amount of polysaccharide decreased as the treatment temperature increased. As reported by Jebrane *et al.* (2018), a key factor influencing the thermal degradation of polysaccharides is the presence of acetyl groups that are thermally labile and lead to the formation of acetic acid, thereby causing acid-catalyzed degradation of the polysaccharides.

**Table 3.** Relative Contents of Saccharides in the Oak Wood

Temperature (°C)	D-Glucose (%)	D-Xylose (%)	L-Arabinose (%)	D-Mannose (%)	D-Galactose (%)
20	43.44 (0.09)	17.10 (0.08)	2.14 (0.04)	3.95 (0.18)	1.02 (0.06)
160	45.32 (0.05)	15.98 (0.17)	1.51 (0.09)	3.02 (0.12)	0.63 (0.09)
180	45.95 (0.12)	12.34 (0.05)	0.83 (0.06)	1.27 (0.07)	0.29 (0.06)
210	44.76 (0.12)	8.21 (0.08)	0.07 (0.04)	0.32 (0.05)	0.07 (0.04)
240	38.82 (0.06)	5.99 (0.16)	ND	0.04 (0.02)	ND

Note: Data represent means, numbers in parentheses represent standard deviation. ND, not detected

Lignin is the most stable component of wood. The relative content of lignin was 25.1 % (SD = 0.05) in untreated oak wood. Its content in the treated oak wood decreased with the severity of the heat treatment to the temperature of 180 °C and was highest (31.80 ± 0.40%) at the temperature of 240 °C. The increase in lignin content in the thermally treated wood was due to the loss of polysaccharide content during heating (Esteves *et al.* 2011; Todaro *et al.* 2018), as well as changes in its structure resulting from the simultaneous de- and re-polymerization reactions of lignin (Li *et al.* 2007; Kačíková *et al.* 2013; Ko *et al.* 2015 a,b).

The results on the macromolecular traits of cellulose in oak wood are presented in Table 4. The molecular weight of cellulose in the wood is reduced after different methods of treatment (Kačíková *et al.* 2013; Kučerová *et al.* 2016; Funahashi *et al.* 2018). Its changes depended on its initial molecular weight, temperature, and time of the treatment. From our work follows that the degree of polymerization of cellulose decreases at all treatment temperatures (Table 4), the biggest change was recorded at the temperature 240 °C. The average molecular weight of the untreated wood was 348,000 g/mol<sup>-1</sup> (SD = 0.228). At the temperature 240 °C, this weight decreased by 32.3%. A constant DP<sub>z</sub>/DP<sub>w</sub> or M<sub>z</sub>/M<sub>w</sub> ratio indicates homogeneity of the depolymerization process (Zou *et al.* 1994). An increase of the M<sub>z</sub>/M<sub>w</sub> ratio indicates the presence of other reactions. At the temperatures 20 °C, 160 °C, and 180 °C, the ratio remained constant, indicating that the depolymerization proceeds randomly without a preferential breakdown of the longest cellulosic chains. With increasing temperature above 210 °C this ratio increased.

**Table 4.** Macromolecular Traits of Cellulose in the Oak Wood

Temperature (°C)	$M_n$ (g/mol <sup>-1</sup> )	$M_w$ (g/mol <sup>-1</sup> )	$M_z$ (g/mol <sup>-1</sup> )	$M_z/M_w$	PD	DP <sub>w</sub>
20	18,779 (44)	348,035 (795)	976,999 (2,100)	2.81	18.53 (0.05)	2,148 (5)
160	18,580 (105)	342,629 (1,243)	986,210 (3,418)	2.88	18.44 (0.06)	2,115 (8)
180	17,991 (224)	342,520 (1,229)	982,312 (2,063)	2.87	19.04 (0.17)	2,114 (8)
210	17,232 (232)	299,887 (1,380)	918,761 (2,731)	3.06	17.40 (0.17)	1,851 (9)
240	14,563 (267)	235,589 (371)	815,344 (1,316)	3.46	16.18 (0.32)	1,454 (2)

Note:  $M_n$  is number average molecular weight;  $M_w$  is weight average molecular weight; DP<sub>w</sub> is a degree of polymerization; PD is polydispersity index

Data represent means, numbers in parentheses represent standard deviation

The measured values for density at EMC, hardness, and EMC in an environment of moist air relative humidity of 65% and of 20 °C are embedded in Table 5. All specimens' oven dry density was lower than water density; therefore with the increasing moisture content the density at EMC of oak wood increased (Fig. 1). The correlations among oak wood EMC (w) and density at EMC ( $\rho$ ) and others properties are shown in Fig. 2.

The EMC decreased significantly with the increasing treatment temperature. Similar results of EMC measured on oak wood were obtained by Zhou *et al.* (2013), Jiang *et al.* (2014), and Barcák *et al.* (2015). Significantly higher values of EMC at 160 °C were measured by Čabalová *et al.* 2018. The EMC is related to free –OH groups at the amorphous part of cellulose and dehydration of the wood was present. It was supposed that the amount of sorption sites decreases. Such phenomenon follows from an analysis of Dent sorption isotherm as was shown by Babiak and Németh (1998) for black locus wood. The changes of thermally treated oak wood lignin amount were weak to influence the EMC more than the amount of holocellulose (Fig. 2).

**Table 5.** Density at EMC, EMC, and Hardness

Temperature (°C)	Density at EMC (g/cm <sup>3</sup> )	EMC (%)	Hardness at 2.82 mm (MPa)
20	0.6446 (0.0197)	12.02 (0.12)	59.6 (4.1)
160	0.5991 (0.0110)	8.73 (0.15)	57.0 (2.8)
180	0.5894 (0.0063)	7.38 (0.25)	55.9 (3.9)
210	0.5830 (0.0095)	7.28 (0.73)	53.9 (2.5)
240	0.5654 (0.0114)	5.47 (0.09)	53.5 (1.7)

Note: The number of observations was equal to 16. Data represent means; numbers in parentheses represent standard deviation.

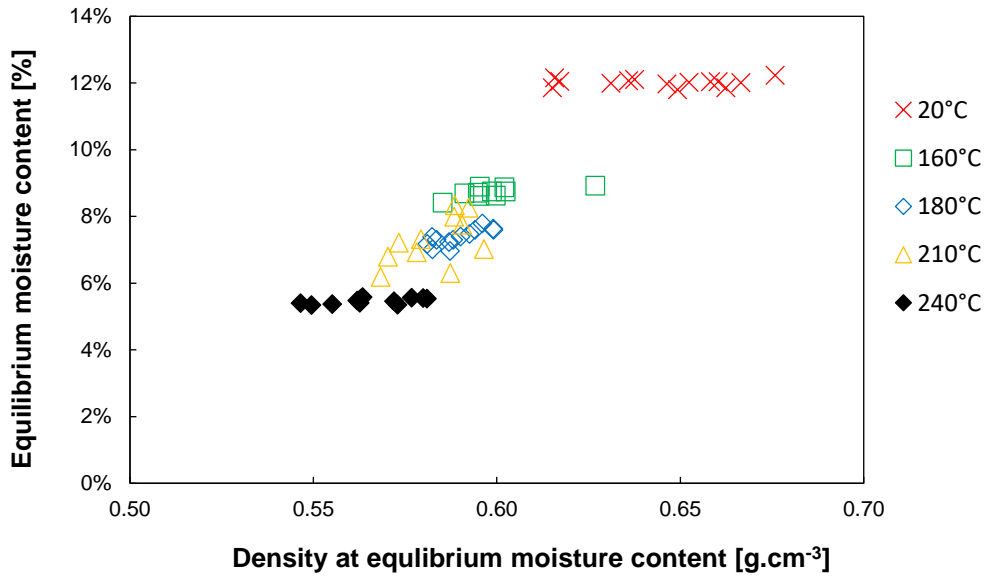


Fig. 1. The influence of EMC on density at EMC

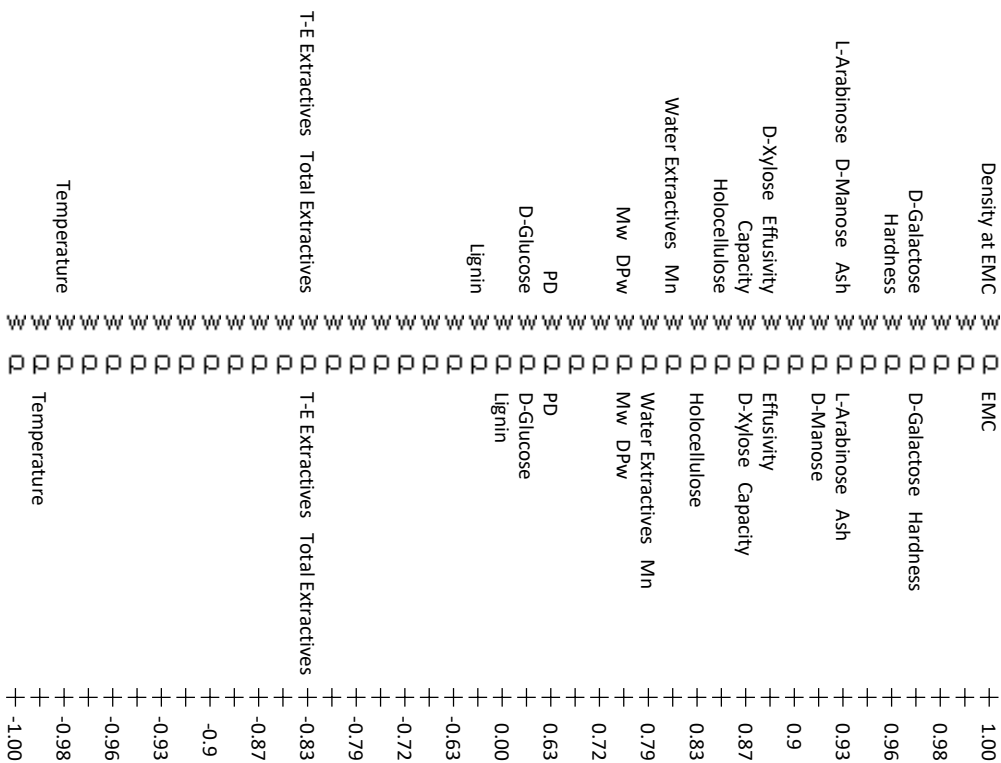
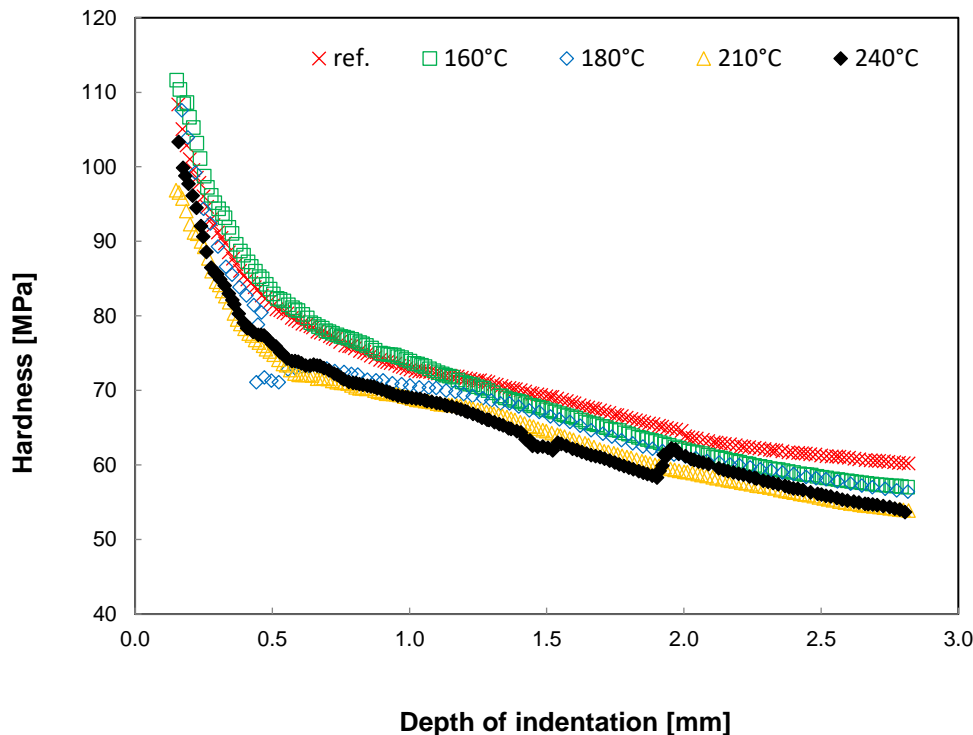


Fig. 2. Correlations among EMC, density at EMC, and selected oak wood properties

The analysis showed a strong positive association between values of both, EMC and density at EMC ( $r = 0.995$ ). In general, the analytical relationship is derived from definitions of density at EMC and EMC (Požgaj *et al.* 1993). The content of saccharides (*i.e.* D-galactose, D-mannose, and L-arabinose) is also positively associated with density at

EMC. Additionally, the changes in EMC and density at EMC were caused by the temperature changes and other parameters similarly (Fig. 3). From the point of moisture content measurement, the positive correlation was detected between water extractives and EMC. On the contrary, a negative correlation was observed between T-E extractives and EMC. The explanation is in oven dry mass chemical composition, in which some nonpolar substances from oven dry mass were dissolved in nonpolar solvents.

Janka's hardness is strongly influenced by the depth of the indentation (Fig. 3). The hardness at 20 °C was lower than the value of hardness at 160 °C at lower depths of the indentation (Fig. 3). The opposite conclusion is shown for depth of 2.82 mm (Table 5). The same result of comparison was also true for temperatures of 210 °C and 240 °C.



**Fig. 3.** The influence of depth of indentation on the hardness

The hardness was positively correlated with D-galactose, L-arabinose, and D-mannose contents but it had a negative correlation with the total extractives. It is also evident that hardness was affected by the treatment temperature (Fig. 4). The disadvantage of the hardness method of measurement is in its particular indentation depth definition of 5.64 mm, 2.82 mm, or force magnitude is prescribed. The detail force recording during the steady speed of indentation revealed a strong dependence of the force on the indentation depth. The bark-type surface is present on the specimens' surface, the reason for the occurrence of the harder surface at the beginning of hardness testing remains unclear. Merela and Čufar (2013) measured oak wood end hardness by Brinell's method. They published lower values of oak end hardness because of the different principal of the used method.

The measured thermal properties in the radial direction and at EMC are shown in Table 6.



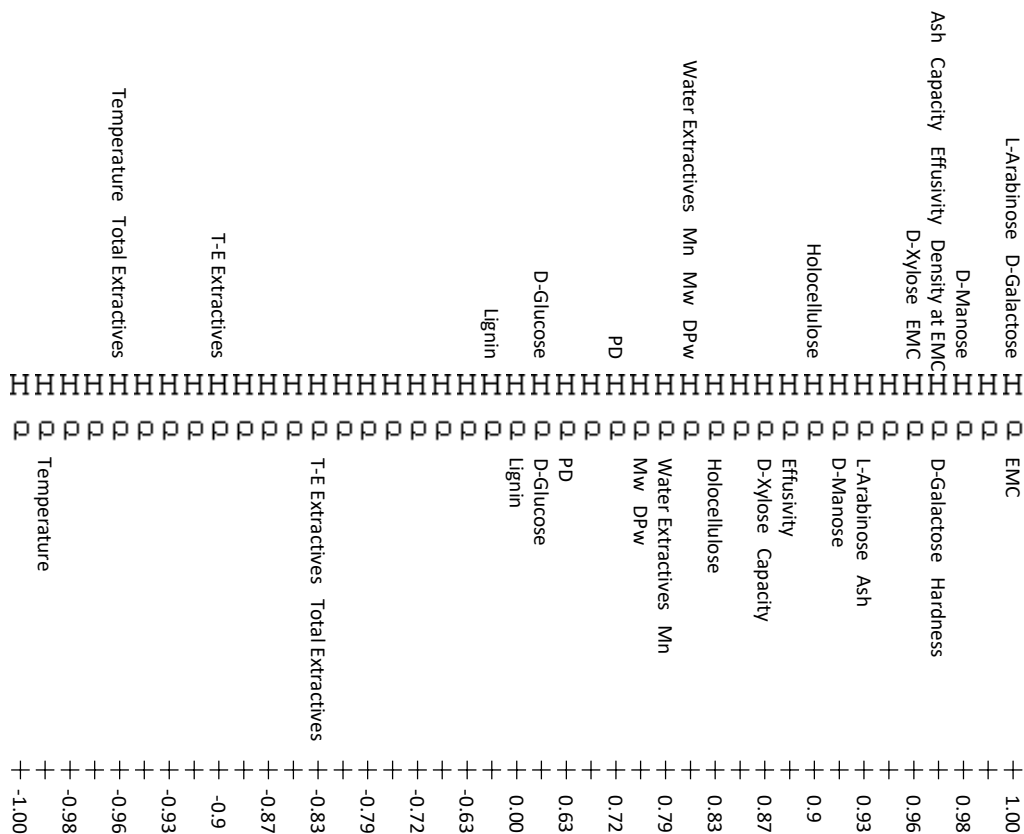


Fig. 4. Correlations among hardness, the density at EMC, and selected oak wood properties

Table 6. Thermal Properties

Temperature (°C)	Thickness (mm)	Mass Specific Heat Capacity (J·(kg·K) <sup>-1</sup> )	Thermal Conductivity (W·(m·K) <sup>-1</sup> )	Thermal Diffusivity (m <sup>2</sup> ·s <sup>-1</sup> )	Effusivity (W·m <sup>-2</sup> ·K <sup>-1</sup> ·s <sup>0.5</sup> )
20	3.95	1790 (140)	0.161 (0.028)	1.4·10 <sup>-7</sup> (1.4·10 <sup>-8</sup> )	430 (53)
160	4.01	1750 (61)	0.159 (0.016)	1.5·10 <sup>-7</sup> (1.3·10 <sup>-8</sup> )	410 (25)
180	4.01	1630 (170)	0.158 (0.031)	1.6·10 <sup>-7</sup> (1.3·10 <sup>-8</sup> )	400 (58)
210	3.79	1520 (81)	0.131 (0.008)	1.4·10 <sup>-7</sup> (1.3·10 <sup>-8</sup> )	350 (12)
240	3.95	1530 (90)	0.129 (0.013)	1.5·10 <sup>-7</sup> (8.2·10 <sup>-9</sup> )	330 (27)

Note: The number of observations was equal to 8. Data represent means; numbers in parentheses represent standard deviation.

The correlations among oak wood mass specific heat capacity, effusivity and other properties, and chemical composition are shown in Fig. 5. Thermal conductivity slightly decreased with the increasing treatment temperature, while thermal diffusivity almost remained unchanged (Table 6). The inverse relationship is between thermal diffusivity and effusivity. The effusivity is proportional to thermal conductivity. Correlation test confirmed a strong association between thermal properties and chemical composition. The effusivity is positively associated with the value of cellulose macromolecular traits ( $M_w$ ,

$M_n$ , PD, and  $DP_w$ ) (Fig. 5). The negative correlation is between total extractives and effusivity. While T-E extractives decreased the oven-dry mass, the water extractives decreased the oven dry mass and EMC. Moreover, the decrease in EMC caused the decrease of the mass specific heat capacity. The mass specific heat capacity was positively correlated with sugars, ash, and holocellulose and negatively correlated with total extractives. Table 6 shows the decrease of thermal conductivity with the increasing treatment temperature. Şahin Kol *et al.* (2010) measured the oak wood thermal conductivity. The results are comparable after computing their values to SI units. Vay *et al.* (2015) measured thermal conductivity by the steady-state method. The reasons for differences in the measured values are in the different densities at the moisture contents of the specimens used during measurement.

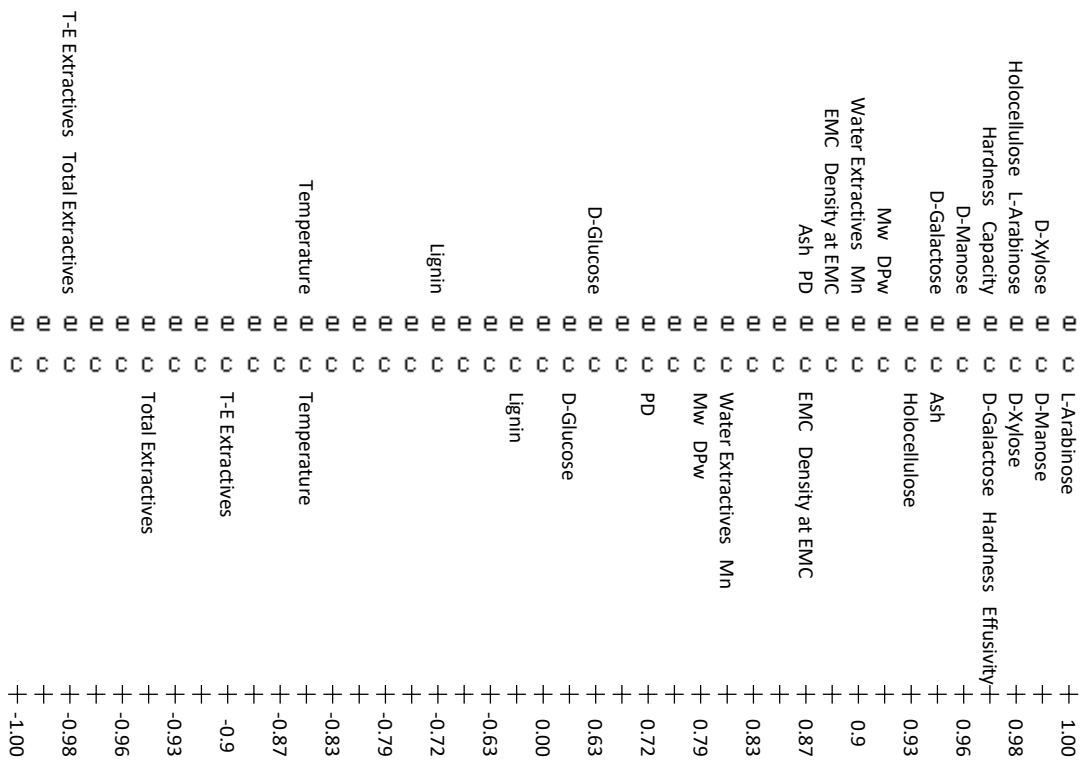


Fig. 5. Correlations among effusivity, mass specific heat capacity and selected oak wood properties

### CONCLUSIONS

1. The relative content of the lignin and total extractives increased with the heat treatment. However, there was a decrease in the relative content of holocellulose, water extractives, and ash in comparison to the untreated wood.
2. The EMC decreased significantly with the increasing treatment temperature. The changes of the thermally treated oak wood lignin amount were weak to influence the EMC more than the amount changes of holocellulose and extractives. The correlations of oak density at EMC and EMC were similar with investigated properties.

3. Oak end hardness is affected by treatment temperature. The depth of indentation significantly affects the hardness.
4. The decrease in EMC caused the decrease of the mass specific heat capacity. Moreover, mass specific heat was positively correlated with sugars, holocellulose, and ash and negatively correlated with total extractives. The thermal diffusivity is hardly affected by treatment temperature. The effusivity is positively associated with the value of cellulose macromolecular traits ( $M_w$ ,  $M_n$ , PD, and  $DP_w$ ), holocellulose, sugars, and EMC (Fig. 5). The negative correlation is between total extractives and effusivity.

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