Effects of Thermal Modification on Selected Physical Properties of Sapwood and Heartwood of Black Poplar (*Populus nigra* L.)

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Black poplar (*Populus nigra* L.) was subjected to thermal modification in superheated steam. The modification was performed at 160 °C, 190 °C, and 220 °C for 2 h. The equilibrium moisture content of the black poplar wood was examined when it was exposed to 76% ± 2% relative humidity at a temperature of 20 °C ± 2 °C. The thermal modification of the poplar wood changed its moisture-exchange-related physical properties to a large extent. The effects of temperature on individual properties (density, mass loss, hygroscopicity, swelling, and water absorption) were diverse, and the intensity of these effects increased with increasing temperature of the thermal treatment process. In most cases, no significant differences were observed between the changes in properties of the sapwood and the heartwood.

Keywords: Equilibrium moisture content; Heartwood; Poplar; Sapwood; Swelling; Thermal modification

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INTRODUCTION

The *Populus* genus, belonging to the willow family (Salicaceae), includes 35 species of trees grouped in several sections (Russell *et al*. 2004; Pirc 2006). The EN 13556 (2003) standard concerning the terminology of wood traded in the European market mentions 10 poplar species or hybrids, including the black poplar (*Populus nigra* L.). It is one of the three poplars (other aspen, white poplar) that occur naturally in Poland. The advantages of this species include a fast growth of biomass and plantation potential (Zajączkowski 2013; Niemczyk *et al*. 2016). Growing stock of standing poplar wood in Poland is estimated at 20 million m^3 (including black and white poplars at 2 million m^3). Forest area of poplar in Poland is about 80 thousand of ha (including black and white poplars at 6 thousand of ha). It accounts for 0.9% of the forest area in Poland (for black and white poplars 0.1%). Average age of tree poplar stands is 47 years (Statistics Poland 2018).

The basic characteristic of wood is density, which determines the remaining properties and potential applications. Wood with low density is usually produced by fast-growing species, including black poplar. Unfortunately, low-density wood usually also has poor natural durability. According to the EN 350 (2016) standard, poplar wood has the lowest natural resistance to fungi activity: On a 1 to 5 scale, it is grade 5, non-resistant wood. It also has low resistance parameters (Wagenführ 2007). It lacks a clearly formed heartwood (strongly saturated with non-structural compounds), which results in a highly hydrophilic and hygroscopic character of this wood (openness for water exchange).

Moreover, it has a drab greyish colour and monotonous pattern. Due to the abovementioned drawbacks, the wood of black poplar is rarely used for the production of solid wood elements. Usually, it serves as a source of cellulose for the paper industry, for the production of derivative wood materials, or as a firewood fuel. Thermal modification of black poplar enhances its properties and thus can extend its range of applications.

Thermal modification of wood is influenced by material factors (*e.g*., species, chemical composition, wood anatomical structure, and wood density) and technological factors. The most frequently analysed correlations include the influence of technological factors (such as temperature of the modification process) on wood properties. Among quality indicators of thermally processed wood, the most frequently used are mass loss (Sandak *et al*. 2015; Candelier *et al*. 2017; Marcon *et al*. 2018), equilibrium moisture content (Majano-Majano *et al*. 2012; Sandak *et al*. 2015; Marcon *et al*. 2018), static bending strength, modulus of elasticity (Todorović *et al*. 2015; Marcon *et al*. 2018), colour change (Bekhta and Niemz 2003; Johansson and Morén 2006; González-Peña *et al*. 2009), and bonding properties (Zigon *et al*. 2015; Hubbe *et al*. 2018). The mass loss of thermally modified wood depends, most of all, on the content and composition of extractive compounds (Todorović *et al*. 2015), as well as hemicellulose content.

Thermal modification can improve many properties of wood. For example, it can reduce its hygroscopicity, which in turn improves dimensional stability (Hill 2006; Korkut *et al*. 2012; Pétrissans *et al.* 2013). An increase in durability is also noticed, that is, a better resistance to fungal activity (Hillis 1984; Tjeerdsma *et al*. 2000). When choosing the parameters of thermal modification (primarily, temperature and time of the thermal process), it is important to consider the initial values of different properties of the modified wood.

This is the starting point for determining which of those properties need to be improved (modified) and to what extent. When using the wood of poplar or other species for building facades, for example, the reduction of hygroscopicity will be the most important objective (hydrophobisation), which will result in less dimensional variations (shrinking). Another positive effect of thermal modification is the change of wood colour to a darker hue (Mitsui *et al*. 2001; Bekhta and Niemz 2003; Johansson and Morén 2006; Esteves *et al*. 2008; Chen *et al*. 2012; Marcon *et al*. 2018), which leads to a wider sales market for such wood.

Thermally modified poplar is available on the market (Grześkowiak and Bartkowiak 2015), but there is not enough data on the black poplar (*Populus nigra* L.) (Sandak *et al*. 2017). Reference literature in this field lacks data on the physical properties of thermally treated black poplar wood, especially with division into sapwood and heartwood.

The differences between heat treated sapwood and heartwood from softwood species are usually analyzed: *Pinus pinaster* Ait. (Esteves *et al*. 2014), *Cedrus libani* A. Richard (Bal 2013), *Pinus sylvestris* L. and *Picea abies* (L.) H. Karst (Metsä-Kortelainen *et al*. (2006). Although the process of heartwood creation in poplars is not very intense, it is still important to be aware of the differences between those two areas of wood, in terms of refining processes and usage of products made of thermally processed black poplar wood.

EXPERIMENTAL

Materials

The study was performed on black poplar (*Populus nigra* L.) obtained from a plantation forest in Poland (eastern part of the Mazovian province, State Forest District Sokołów Podlaski). The tested wood was solid wood of 40-year-old poplars. The trees had a diameter at breast height (DBH) up to 0.5 m and a mean growth ring width greater than 5 mm. The round wood was sawn into boards in a sawmill located in this region. Wood was supplied in the form of selected air-dried timber that was 21 mm thick (radial), 140 mm wide (tangential), and 2500 mm long. It was high quality timber without material defects such as knots, tangled fibres, cracks, insect trails, or rot. The timber was cut, creating sets of samples with sapwood and heartwood. The wood samples were exposed to 76% ± 2% relative humidity at a temperature of 20 °C ± 2 °C. Then sets of samples with similar average density and share of sapwood and heartwood were created (Table 1). The dimensions of the samples used for thermal modification were as follows: 300 mm (longitudinal), 20 mm (tangential), and 20 mm (radial). The surface of the wood samples was finished by planing. Thirty samples were used in each set. One set of samples was kept as a control group and was not subjected to thermal modification. The control samples as well as samples after different variants of thermal modification were divided into smaller samples for testing individual properties (Table 1).

Table 1. Main Properties of Black Poplar Samples

Variable	Property				
	Density	Mass loss	Equilibrium Moisture Content	Water absorption	Linear swelling
Dimensions (mm, L×R×T)	60x20x20	300x20x20	300x20x20	40x20x20	
No. of samples	12	30	12	20	

	Density of samples before heat modification (standard deviation in parentheses)							
Modification temperature (°C)	non-modified		160		190		220	
Area	S	H	S	H	S	H	S	H
	384 (21)	392 (16)	376 (28)	391 (26)	388 (30)	388 (28)	374 (29)	396 (33)

Methods

Thermal modification

The black poplar was modified in superheated steam in laboratory conditions in an oven (model 800, Memmert GmbH + Co. KG, Schwabach, Germany). The working oven temperature range was from 5 °C above ambient temperature up to 250 °C, with a chamber volume 749 cubic decimetres. The peak modification temperatures were 160 °C, 190 °C, and 220 °C, over a period of 2 h. Thermal modification was performed for sapwood and heartwood of the black poplar. The course of the thermal modification is presented in Fig. 1.

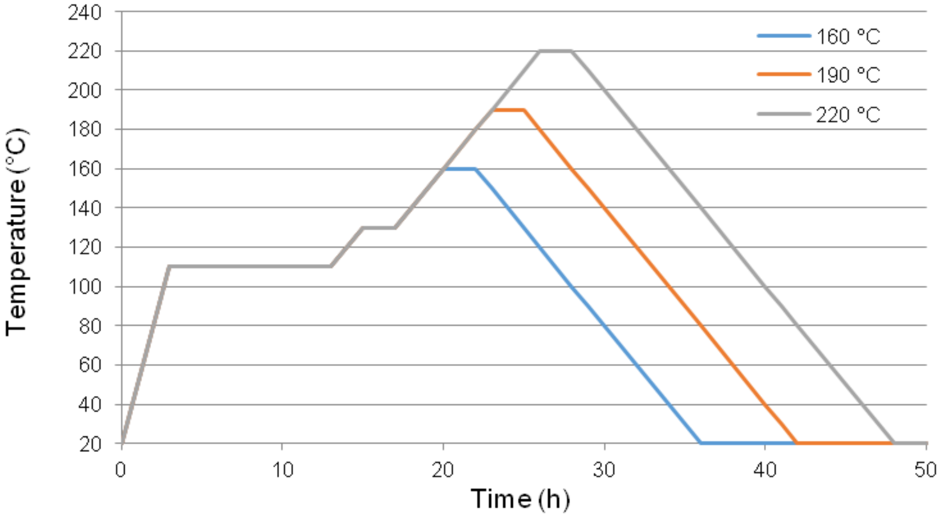

Fig. 1. The course of the thermal modification of the black poplar

Density and mass loss

The wood density was determined according to the ISO 13061-2 (2014) standard, and moisture content was measured according to ISO 13061-1 (2014). The mass loss was determined by comparing the mass of the samples before and after the thermal process. As a point of reference, an oven dry state of wood samples was used. The loss of mass was expressed as a percentage of the initial mass of the totally dry wood. The loss of wood mass was calculated according to Eq. 1, where m_o is the mass of the oven-dried wood (g), and m_{otm} is the mass of the oven-dried wood after thermal modification (g):

$$M_L = \frac{m_o - m_{otm}}{m_{otm}} \cdot 100\% \quad (1)$$

Determination of the equilibrium moisture content of wood

The tests of the equilibrium moisture content (EMC) were performed separately for the sapwood and heartwood of the black poplar. The wood samples, in an oven dry state, were placed in containers in which the relative humidity was 76% ± 2% at a temperature of 20 °C ± 2 °C. In these conditions, native wood reaches a moisture content of approximately 12% (the typical annual average wood moisture in Europe), as required for the tests of mechanical properties (Kozakiewicz and Matejak 2013). Wood conditioning under the test conditions was achieved using a saturated solution of sodium chloride. The chemical substance used was of proanalysis (p.a.) grade and was obtained from Chempur (Piekary Śląskie, Poland). The samples were weighed after 1 h, 3 h, 6 h, 8 h, 24 h, and 48 h of humidification. The wood EMC was measured when the mass of the wood samples remained unchanged over three weighings at 48 h intervals. The mass of samples was determined with an accuracy of ± 0.001 g. The relative humidity was measured using an AZ 9871 anemometer (AZ Instrument Corp., Taichung City, Taiwan).

Water absorption measurement

The wood samples were dried to constant mass at a temperature of 103 °C ± 2 °C. The samples were dried until the mass between two successive measurements did not differ by more than 0.2%.

The samples were cooled to room temperature (20 °C ± 2 °C) in a desiccator. Then, the samples were soaked in distilled water at a temperature of 20 °C ± 2 °C. The samples were weighed after 0.25 h, 0.50 h, 1 h, 2 h, 3 h, 5 h, 8 h, and 24 h of soaking in water. Water absorption (*WA*) was calculated according to Eq. 2, where m_o is the mass of the oven-dried wood (g), and $m_{\mathrm{s(t)}}$ is the mass of the wood after soaking in water (g) for a considered time:

$$WA(t) = \frac{m_{\mathrm{s(t)}} - m_\mathrm{o}}{m_\mathrm{o}} \cdot 100\% \tag{2}$$

Determination of linear swelling

Linear swelling was determined in the radial and tangential directions. The dimensions of the samples were determined with an accuracy of ± 0.001 mm. Linear swelling for the radial (S_r) and tangential (S_t) directions was calculated using Eqs. 3 and 4,

$$S_\mathrm{r} = \frac{r_\mathrm{c} - r_\mathrm{o}}{r_\mathrm{o}} \cdot 100\% \tag{3}$$

$$S_\mathrm{t} = \frac{t_\mathrm{c} - t_\mathrm{o}}{t_\mathrm{o}} \cdot 100\% \tag{4}$$

where r_o and t_o are the dimensions (mm) of the wood samples in the oven-dried condition, measured in the radial and tangential directions, respectively, and r_c and t_c are the dimensions (mm) of the wood samples conditioned at 76% ± 2% relative humidity at 20 °C ± 2 °C, measured in the radial and tangential directions, respectively.

Statistical analysis

Statistical analyses were performed using STATISTICA (version 12) software from StatSoft, Inc. (Tulsa, OK, USA). The statistical analyses of the results were performed using a significance level (p) of 0.050. Statistical analyses are presented in Tables 2 to 4, where the individual symbols describe variants of the investigated black poplar wood (*e.g.*, S – sapwood, H – heartwood, S_NM – non-modified sapwood, H_NM – non-modified heartwood).

RESULTS AND DISCUSSION

The density of the non-modified black poplar wood (exposed to 76% ± 2% relative humidity at 20 °C ± 2 °C) was 384 kg/m^3 ± 21 kg/m^3 and 392 kg/m^3 ± 16 kg/m^3 for sapwood and heartwood, respectively (Fig. 2a). The differences between sapwood and heartwood density were not statistically significant in the black poplar wood (Table 2).

According to data from reference tables, the density of air-dried black poplar wood averages 450 kg/m^3 and usually falls between 410 kg/m^3 and 560 kg/m^3 (Galewski and Korzeniowski 1958; Wagenführ 2007).

The density of the tested wood was lower than the typical range given in the literature. This shift probably resulted from the young age of these trees and the predominance of juvenile wood in the research material. Notably, a young age of logged wood is typical for plantations of fast-growing species (West 2014; Niemczyk *et al*. 2016).

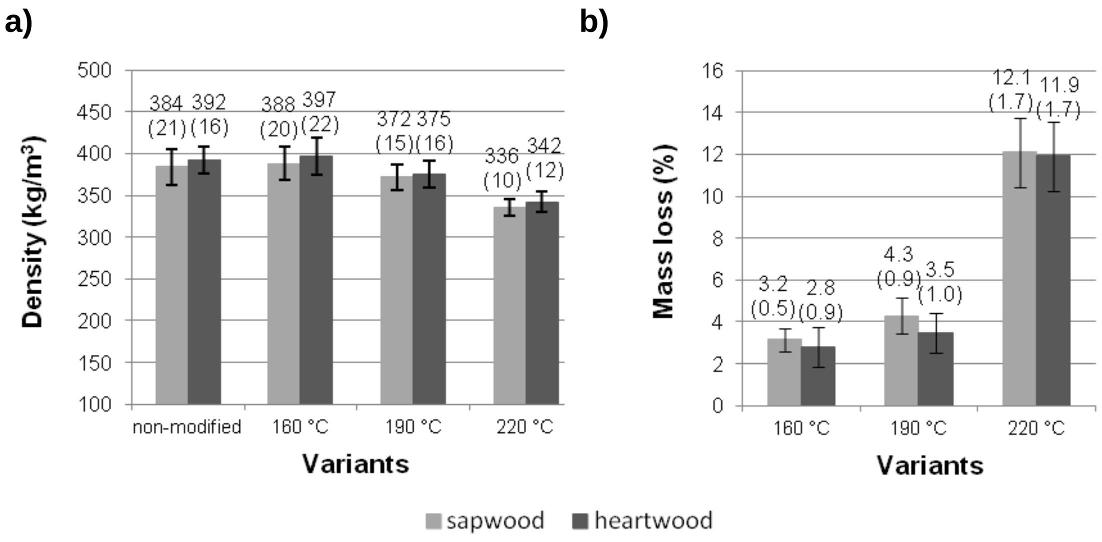

Fig. 2. (a) Density of thermally modified black poplar exposed to 76% ± 2% relative humidity at 20 °C ± 2 °C and (b) mass loss (error bars - standard deviation)

Table 2. Statistical Analysis of Wood Density Results (t-test)

Variant	S_NM	H_NM	S_160 °C	S_190 °C	S_220 °C	H_160 °C	H_190 °C	H_220 °C
S_NM	-	ns	ns	ns	s	ns	ns	s
H_NM	ns	-	ns	s	s	ns	ns	s
S_160 °C	ns	ns	-	ns	s	ns	ns	s
S_190 °C	ns	s	ns	-	s	ns	ns	s
S_220 °C	s	s	s	s	-	s	s	ns
H_160 °C	ns	ns	ns	ns	s	-	ns	s
H_190 °C	ns	ns	ns	ns	s	ns	-	s
H_220 °C	s	s	s	s	ns	s	s	-

ns – no significant dependence; s – significant dependence, $p < 0.050$

As a result of thermal modification, wood mass decreased (Fig. 2b), which in turn caused a reduction of the black poplar wood density. Decreasing wood density as a result of thermal modification could also be influenced by the lowering of the equilibrium moisture content of wood (EMC). Lower EMC affects the lower wood mass but also lower shrinkage (*i.e.* lower wood volume), resulting in a slight change in density. Significant decreases in the properties under analysis were observed, most of all, in the set of black poplar wood that was modified at 220 °C (Table 2). The mass loss for sapwood and heartwood was approximately 12%, while density decreased by approximately 13%. These changes resulted from a degradation of wood components, mostly hemicelluloses, under the influence of high temperature. İmirzi *et al*. (2014) reported that hemicelluloses tend to degrade at temperatures greater than 120 °C, and the level of degradation depends on the time of the thermal process. No significant differences in density were found between the sapwood and heartwood of the thermally modified black poplar. Nonetheless, a slightly greater mass loss was observed for sapwood, as compared to heartwood, which did not depend so much on the temperature of the thermal process. While Bal (2013) stated that as the treatment temperature was increased, the mass of the *Cedrus libani* heartwood decreased more than that of the sapwood, which may be due to the fact that the heartwood had greater extractives content.

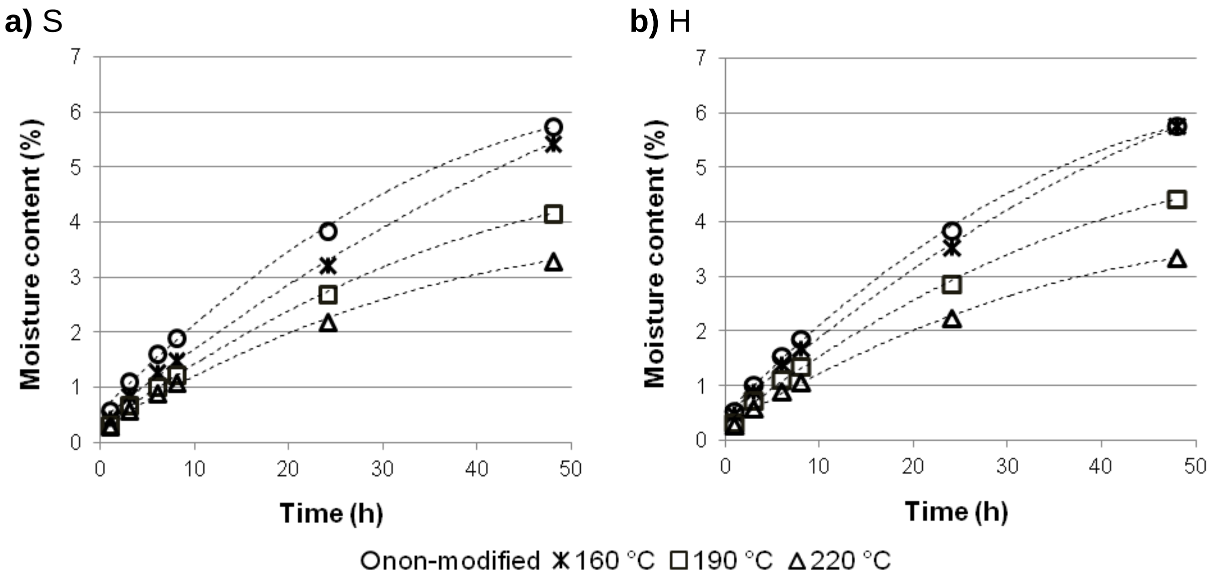

Fig. 3. Changes in wood moisture content of the thermally modified black poplar (a) sapwood and (b) heartwood during the first 48 h of humidification

Figure 3 presents the changes in moisture content of the thermally modified black poplar sapwood and heartwood during the first 48 h of humidification (76% ± 2% relative humidity at 20 °C ± 2 °C). The process itself was noticeably dynamic: In the first 48 h of vapour absorption, the change in moisture content was very fast, after which the absorption becomes slow and approaches the EMC asymptotically (Kozakiewicz and Matejak 2013). The dynamics of the changes in sapwood and heartwood moisture content were very similar (Fig. 3a and 3b). No highly hydrophobic substances are produced in the formation of heartwood in poplars (Galewski and Korzeniowski 1958; Wagenführ 2007), which is why the hygroscopic properties of sapwood and heartwood are similar in this species. In general, it should be noted that increasing the temperature of the thermal modification decreased the EMC of the black poplar wood (Fig. 4). Similar correlations were observed by Akyildiz and Ateş (2008), Nguyen *et al*. (2012), and Brito *et al*. (2018).

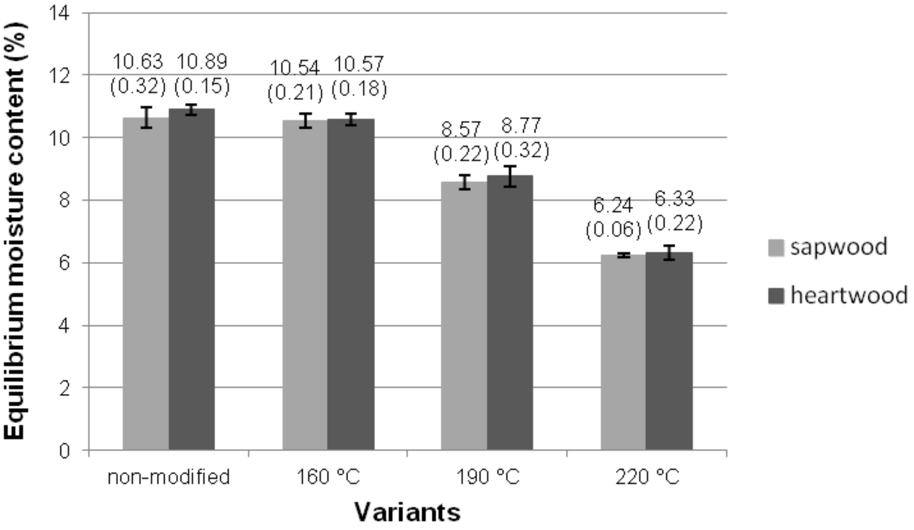

Fig. 4. Equilibrium moisture content of the thermally modified black poplar exposed to 76% ± 2% relative humidity at 20 °C ± 2 °C (error bars - standard deviation)

Table 3. Statistical Analysis of the EMC Test Results (t-test)

Variant	S_NM	H_NM	S_160 °C	S_190 °C	S_220 °C	H_160 °C	H_190 °C	H_220 °C
S_NM	-	ns	ns	s	s	ns	s	s
H_NM	ns	-	ns	s	s	s	s	s
S_160 °C	ns	ns	-	s	s	ns	s	s
S_190 °C	s	s	s	-	s	s	ns	s
S_220 °C	s	s	s	s	-	s	s	ns
H_160 °C	ns	s	ns	s	s	-	s	s
H_190 °C	s	s	s	ns	s	s	-	s
H_220 °C	s	s	s	s	ns	s	s	-

ns – no significant dependence; s – significant dependence, $p < 0.050$

Statistically significant differences were found in the EMC of the non-modified black poplar wood, as compared to wood modified at 190 °C and 220 °C. No statistically significant differences were observed between the EMC of sapwood and heartwood (Table 3). The EMCs of the black poplar wood modified at 190 °C and 220 °C were less than the EMC of the non-modified black poplar wood by 19% and 42%, respectively. Brito *et al*. (2018) observed similar correlations when examining the EMC of yellow poplar (*Liriodendron tulipifera* L.). Yellow poplar, similarly to black poplar, is a fast-growing species, and its anatomical structure is similar as well. The authors stated that the EMCs of yellow poplar thermally modified at temperatures of 180 °C and 220 °C (for a duration of 2.5 h) and exposed at 21 °C and 65% relative humidity were approximately 16% and 51% less, respectively, than the EMC of non-modified yellow poplar. Analogous EMC change correlations were observed by Akyildiz and Ateş (2008) in selected Turkish wood species. The changes in EMC result from the degradation of hemicelluloses and amorphous cellulose areas, caused by the high temperature of the treatment process (Esteves and Pereira 2009). Thermal modification with increasing temperature causes changes in the properties of modified wood, usually they have a logarithmic character (Marcon 2018). Nevertheless, in the studied range of temperatures between 160 °C and 220 °C, it can be approximated that the reduction of black poplar EMC is directly proportional to the increase in temperature of the thermal modification process, independently of whether it is sapwood or heartwood.

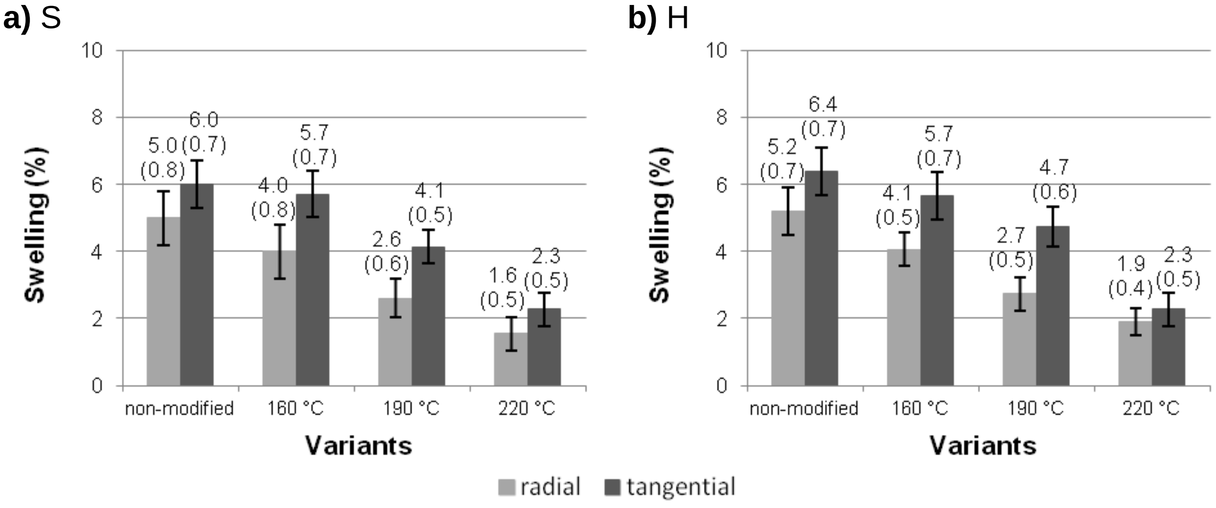

Fig. 5. Swelling in the radial and tangential directions of the thermally modified black poplar (a) sapwood and (b) heartwood (error bars - standard deviation)

The swelling test results in the radial and tangential directions for the black poplar wood, divided into sapwood and heartwood, are presented in Fig. 5. The non-modified black poplar wood under analysis had low swelling anisotropy of 1.2, resulting probably from the dominant presence of juvenile wood and a notable curvature of annual growth rings in the tested samples (it was difficult to indicate exact tangential anatomical direction). In mature black poplar wood, the swelling anisotropy (the ratio of swelling in tangential *vs*. radial sections) is approximately 1.8 (Galewski and Korzeniowski 1958; Wagenführ 2007).

As a result of the thermal modification of the black poplar wood, significant reductions of swelling in the radial and tangential directions were observed (Fig. 5). As the modification temperature increased, the reduction in swelling was enhanced. No significant differences were found between the swelling of sapwood and heartwood (Fig. 5a and 5b). This refers both to the non-modified and the thermally modified wood. Notably, thermal modification allowed for reducing the swelling of the black poplar threefold (for a modification temperature of 220 °C). This refers to swelling in both the tangential and radial directions, while swelling anisotropy remained at a rather similar level, with a slight trend to reduce with the modification temperature. Schneid *et al*. (2014) stated that thermal modification of wood greatly increases its dimensional stability and reduces its hygroscopicity due to the degradation of hemicelluloses, polymer reticulation, and breaking of hydroxyl groups from amorphous zones of cellulose. This is confirmed by research performed by Čermák *et al*. (2015).

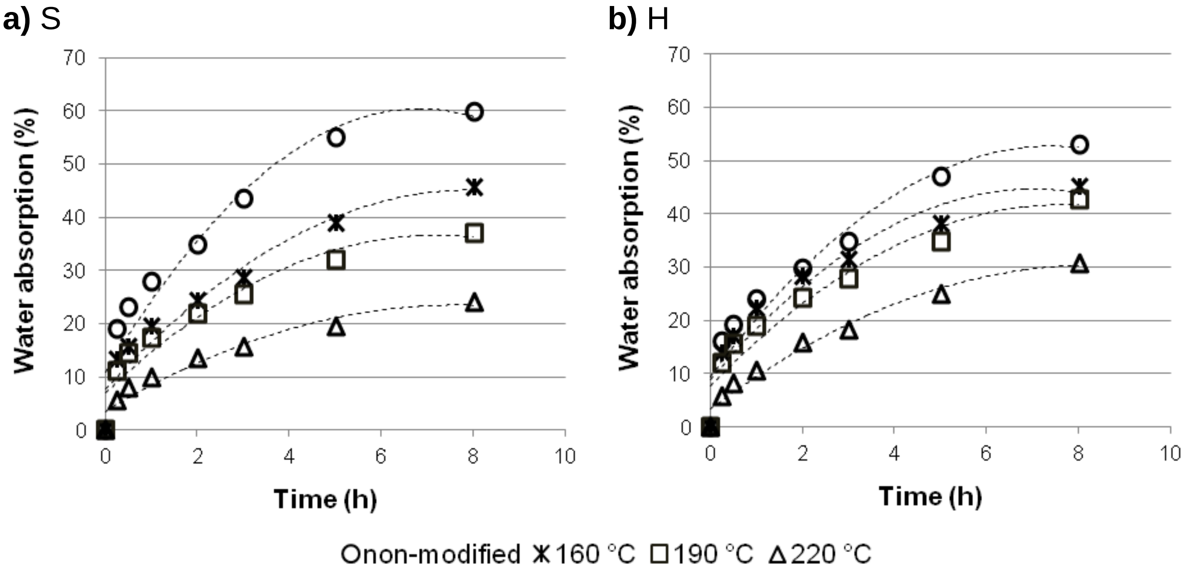

Fig. 6. Water absorption of the thermally modified black poplar (a) sapwood and (b) heartwood during the first 8 h of soaking

The non-modified black poplar sapwood had a more dynamic water absorption curve during the first 8 h of soaking than heartwood (Fig. 6a and 6b). The structural elements of heartwood have closed pits, which significantly hinders the flow of water and thus slows down the water absorption process. Independently of the degree of thermal modification of the poplar wood, water absorption happened in the same way. At the beginning, in the first few hours, the process was dynamic (Fig. 6), and then it slows down considerably and asymptotically approaches the final moisture content level. Thermal processing resulted in a more balanced water absorption process for the sapwood and heartwood.

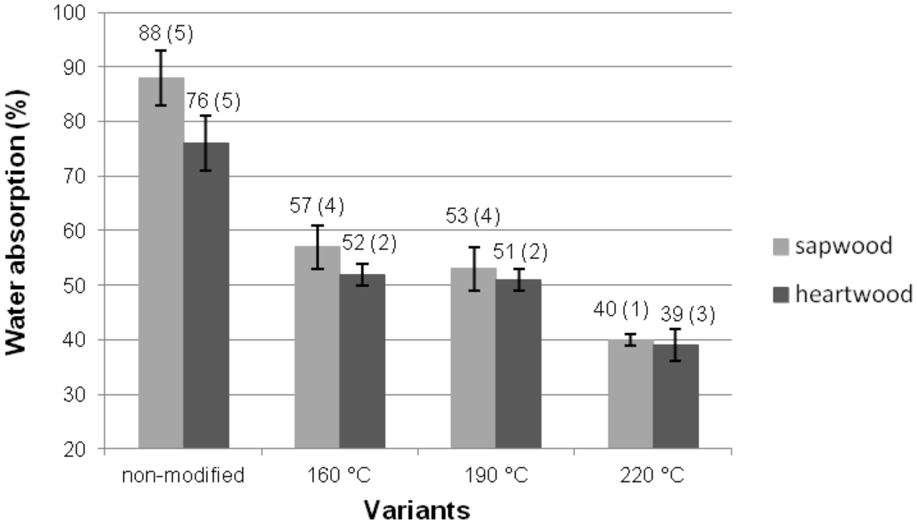

Fig. 7. Water absorption of the thermally modified black poplar after 24 h of soaking in water (error bars - standard deviation)

Table 4. Statistical Analysis of the Water Absorption Test Results (t-test)

Variant	S_NM	H_NM	S_160 °C	S_190 °C	S_220 °C	H_160 °C	H_190 °C	H_220 °C
S_NM	-	ns	s	s	s	s	s	s
H_NM	ns	-	ns	s	s	s	s	s
S_160 °C	s	ns	-	ns	s	ns	ns	s
S_190 °C	s	s	ns	-	s	ns	ns	s
S_220 °C	s	s	s	s	-	s	s	ns
H_160 °C	s	s	ns	ns	s	-	ns	s
H_190 °C	s	s	ns	ns	s	ns	-	s
H_220 °C	s	s	s	s	ns	s	s	-

ns – no significant dependence; s – significant dependence, $p < 0.050$

There were no significant differences between the water absorption of black poplar sapwood and heartwood (Table 4). In contrast, Metsä-Kortelainen *et al.* (2006) found that the thermally modified heartwood of Scots pine and Norway spruce absorbed less water than sapwood. Probably the difference in findings results from differences in the anatomical structure of hardwood and softwood species.

The water absorption of the thermally modified black poplar largely depended on the temperature of the modification process (Fig. 7). The lowest temperature of the thermal process in this study (160 °C) lowered water absorption by as much as 25 percentage points compared to the non-modified wood. The next significant drop by another 25 percentage points happened after thermal modification at 220 °C. The thermal process yielded strong hydrophobisation of the wood, resulting in the significant and favourable reduction of humidity-induced dimensional variations, as described above. This result is confirmed by other research. Kartal *et al.* (2007) stated that heat treatment evidently decreased the water absorption, and the heat-modified specimens absorbed less water than unheated specimens.

As treatment temperature and duration increased, the amount of absorbed water decreased. Meanwhile, thermally modified wood has a different propensity for adhesives and surface refining (Esteves and Pereira 2009).

CONCLUSIONS

1. The density of the black poplar wood decreased significantly after thermal modification at 190 °C, and even more so after modification at 220 °C. This decrease co-occurred with a significant mass loss exceeding 10%. Independently of the temperature of the thermal process, a slightly greater mass loss was observed for sapwood, compared to heartwood.
2. Significant differences were observed in the hygroscopic properties of the unmodified and thermally modified black poplar. As the applied treatment temperature increased, the EMC of the black poplar decreased. There were no significant differences between the equilibrium moistures of sapwood and heartwood.
3. As a result of thermal modification, there was an important reduction in the swelling degree of the thermally modified black poplar in the radial and tangential directions. The swelling reduction was greater after modification in higher temperatures (even threefold at 220 °C). There were no significant differences in swelling between sapwood and heartwood.
4. The thermal modification reduced the differences in kinetic to water absorption of sapwood and heartwood. Higher temperatures of the thermal modification process caused greater reduction of kinetic to water absorption of the modified black poplar wood.
5. Thermal modification in superheated steam significantly changed the physical properties of the black poplar wood related to moisture exchange. From a practical point of view, the division into sapwood and heartwood seems to be irrelevant in this case.

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