The Effects of Tree Age, Thickness, and Depth of Timber on Density and Mechanical Properties of Heat-treated Black Poplar Wood (*Populus nigra*)

Saeed Hassani,[a] Mohammad Talaeipour,[b,*] Behzad Bazyar,[b] Amir Hooman Hemmasi,[b] and Saeed Mahdavi [c]

ThermoWood is a wood modification method that is used to improve the wood application and dimensional stability of wood. In this study, poplar wood (*Populus nigra*) was used in two age groups of 18- and 38-year-old trees to investigate the effect of tree age, thickness, and depth of heat-treated timbers on mechanical properties and density. In each age group, the timbers were prepared according to thickness of 40, 50, and 60 mm. The experimental samples were prepared from the surface (S) and middle (M) depth of heat-treated timbers based on age and thickness. Some properties of heat-treated and control wood such as density (oven-dry density, air-dry density, and basic density) and mechanical properties (modulus of rupture (MOR), modulus of elasticity (MOE), and impact strength) were measured. In general, density and mechanical properties of heat-treated wood were decreased compared with the control samples. Density and mechanical properties of heat-treated and control wood samples were increased from 18- to 38-year-old trees. There were no differences in density and mechanical properties after changing thickness. Surface and middle depth specimens of heat-treated timbers showed a positive effect on the impact strength but had no considerable effect on densities and other strengths of heat-treated wood timbers.

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Keywords: Thermowood; Tree age; Timber Thickness; Timber Depth; Density; Mechanical properties

*Contact information: a: Ph.D. student, Department of Wood and Paper Science, Science and Research Branch, Islamic Azad University, Tehran, Iran; b: Department of Wood and Paper Science, Science and Research Branch, Islamic Azad University, Tehran, Iran; c: Wood and Forest Products Division, Research Institute of Forests and Rangelands, Agricultural Research Education and Extension Organization (AREEO). Tehran, Iran; *Corresponding author: m.talaeipoor@srbiau.ac.ir*

INTRODUCTION

Wood is a natural material with beautiful appearance, suitable ductility, reasonable cost, low density, and easy accessibility. While wooden material has features that are appreciated by its users, it has numerous disadvantages such as wettability, swelling, shrinkage, and low resistance to biological degradation. Many studies have attempted to modify these undesirable qualities of wooden material (Percin *et al*. 2016; Aytin *et al*. 2018).

Density is an important property of wood. Fast-growing species such as black poplar wood have low resistance parameters (Kozakiewicz *et al*. 2019). In general, there is a high correlation between the density and mechanical strength of wood. Density and strength of wood are decreased after heat treatment (ThermoWood Handbook 2003).

Heat treatment improves some wood properties by decreasing moisture adsorption, improving dimensional stability, and increasing biological durability of wood (Akgul and Korkut 2012; Jebrane *et al*. 2018; Kozakiewicz *et al.* 2020). The main disadvantage of this process is the reduction of mechanical resistance depending on the type, temperature, time, rate, and atmosphere of treatment (Kubojima *et al*. 2000; Bekhta and Niemz 2003; Hill 2006). Density is decreased during heat treatment, and this change is associated with a decrease in equilibrium moisture content (Kozakiewicz *et al*. 2019). Weight loss occurs after heat treatment, and this reduction depends on temperature, time, and medium of heat treatment (Esteves *et al*. 2007; Esteves and Pereira 2009).

There are numerous commercial heat treatment methods including ThermoWood in Finland, PlatoWood in Holland, OHT-Oil Heat Treatment in Germany, and Bois Perdure and Retrification in France (Esteves and Pereira 2009). ThermoWood is an industrial-scale wood heat treatment process that was developed at VTT, together with the Finnish industry (Viitaniemi 1997). This process is carried out at 180 to 250 °C using water vapor (Jamsa *et al*. 2000). In 2000, ThermoWood process was officially launched by the ThermoWood Association, with the introduction of the treatment classes named Thermo-S and Thermo-D (Torniainen *et al*. 2021). The letter (S) in Thermo-S stands for "stability" and the letter (D) in Thermo-D stands for "durability". The process is divided into three stages including temperature increasing and high-temperature drying, heat treatment, and finally cooling and moisture conditioning (ThermoWood Handbook 2003).

Heat treatment conditions are not the same in all research. No research has investigated the effect of tree age, thickness and depth of heat-treated timber, and most research has examined the effect of temperature and duration of heat treatment on different species. Nonetheless, the influence of tree age and timber thickness on the properties of heat-treated wood remain unclear.

This study investigated the effect of tree age, thickness, and depth of heat-treated timber on density and mechanical properties of black poplar wood (*Populus nigra*) with ThermoWood process.

EXPERIMENTAL

Materials

Black poplar (*Populus nigra*) trees were taken from a planted forest in Zanjan state, Iran. Five 18- and 38-year-old black poplar trees were used. The trees had a diameter at breast height (DBH) of 250 and 500 mm, respectively. Logs were coated by silicone based alkyd resin as a weather protective to inhibit cracking. They were cut into timbers with 40, 50, and 60 mm thickness (radial), 150 mm width (tangential) and 2000 mm length (longitudinal) after decreasing of moisture to 25%. All wood samples were selected according to their high quality and absence of defects such as cracks, knots, or rot. The classified timbers were transferred to a ThermoWood factory for heat treatment, and the control timbers were stored to achieve equilibrium humidity with the air (Fig. 1).

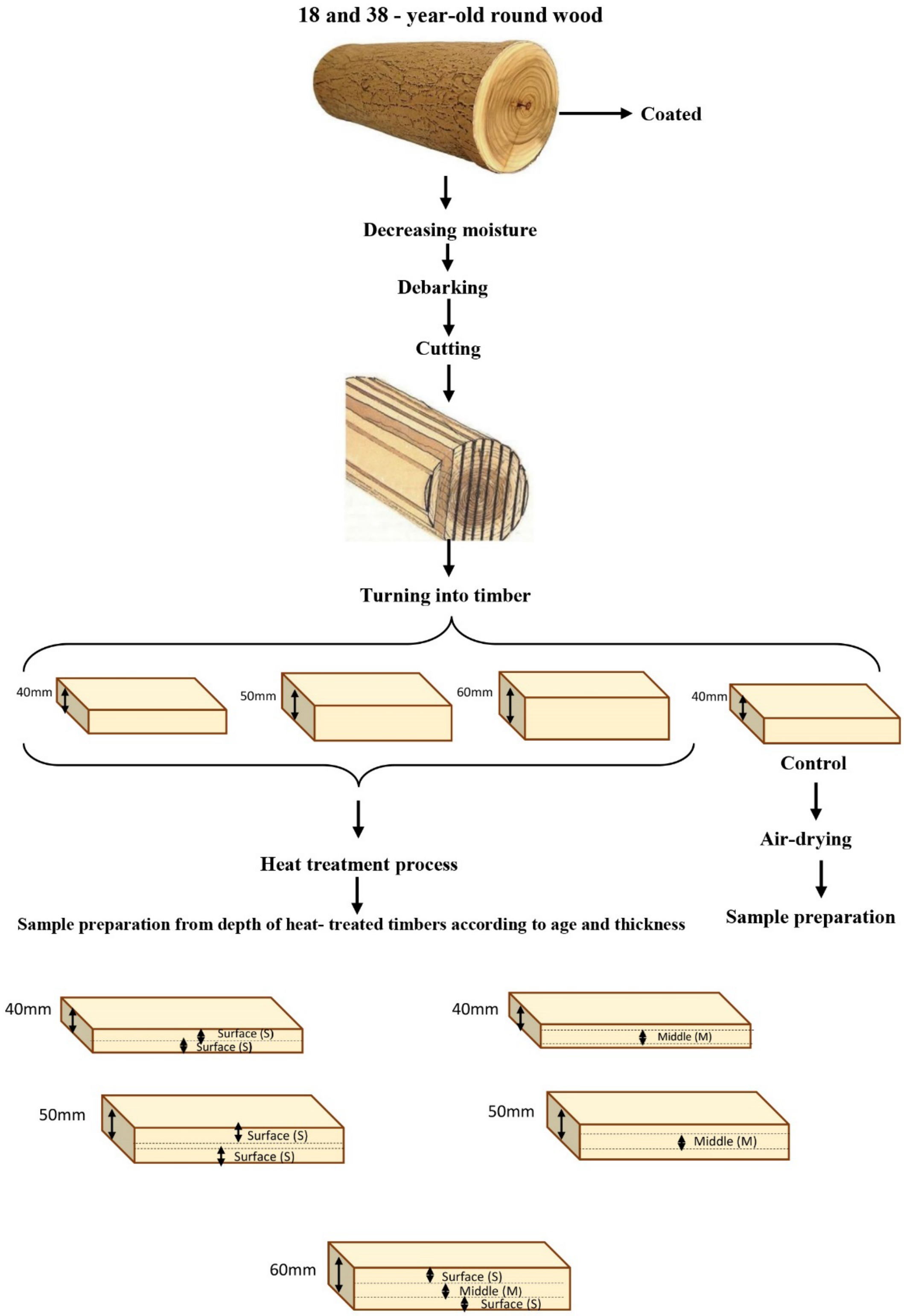

Fig. 1. Steps of converting round wood to timber and sampling

Heat Treatment

The average moisture content of timbers measured before heat treatment was 25%. The timbers were loaded into the drying kiln. The temperature and humidity sensors were placed inside these timbers, and the ThermoWood process was started. The process was divided into three stages. The first stage was temperature increasing and high-temperature drying. The drying kiln temperature was raised rapidly to 100 °C using heat and water steam. Then, the temperature was increased gradually to 130 °C, during which time the high-temperature drying occurred and the moisture content in the wood was decreased to nearly zero. This stage lasted for 33 h. The second stage was heat treatment. During 6 h the temperature inside the drying kiln was increased from 130 to 212 °C. The maximum pressure and relative humidity inside the drying kiln for heat treated wood were 8.3 bar and 100%, respectively. In this study, the wood timbers were heat-treated at a constant temperature of 212 °C for 2 h. The final stage was cooling and moisture conditioning. The temperature was lowered using water spray systems. This process was performed until the moisture content of timbers was reached 4%. The cooling and conditioning stage lasted for 24 to 30 h, depending on thickness and width of timbers. The ThermoWood process was carried out in Mazand wood Aria factory (MCA), located in the suburb of Tehran city, Iran.

Sample Preparation

After the heat treatment, wood samples were prepared from surface (S) and middle (M) depth of heat-treated timbers according to the tree age and timber thickness (Table 1).

Table 1. Introduction of Treatments

	Tree Age (year)	Timber Thickness (mm)	Timber Depth
Heat-treated	18 38	40 50 60	Surface (S) Middle (M)
Control	18 38	-	-

Density Determination and Mechanical Properties

Heat-treated and control samples of black poplar wood (*Populus nigra*) were kept at standard environmental conditions (RH 65%, 20 °C) for laboratory tests. The density of heat-treated and control wood samples was determined according to ISO13061-2 (2014). The samples were weighed with an accuracy of ± 0.001 g, and the dimensions of the samples were measured with an accuracy of ± 0.01 mm.

The mechanical properties included modulus of rupture (MOR), modulus of elasticity (MOE), and impact strength of heat-treated and control samples. Experimental samples were prepared from the surface (S) and middle (M) depth of heat-treated timber based on tree age and timber thickness.

The MOR and MOE of heat-treated and control samples with a dimension of 25×25×410 mm^3 were determined in accordance with ASTM D143-94 (2007) and using an Instron device (model 1186, Waltham, MA, USA). The loading speed of the device was 2.5 mm/min, and the span length was 350 mm. The notched_impact strength test of heat-treated and control samples was carried out according to EN 10045-1 (1993) standard using Instron Wolpert device (model Pw5) with the Charpy method.

RESULTS AND DISCUSSION

Mechanical Properties

Figure 2 shows that the MOR of heat-treated samples was decreased compared with the control samples. There were considerable differences between the 18- and 38-year-old heat-treated and control wood samples. With increasing tree age from 18 to 38 years, MOR values were increased. The highest MOR in the 38-year-old control sample was 72.81 MPa. In 18- and 38-year-old heat-treated samples, much difference was not observed between thicknesses of 40, 50, and 60 mm and surface (S) and middle (M) depth of heat-treated samples. Although heat treatment reduced the MOR, increasing tree age in the heat treatment process improved the MOR. This difference was also evident in the control samples.

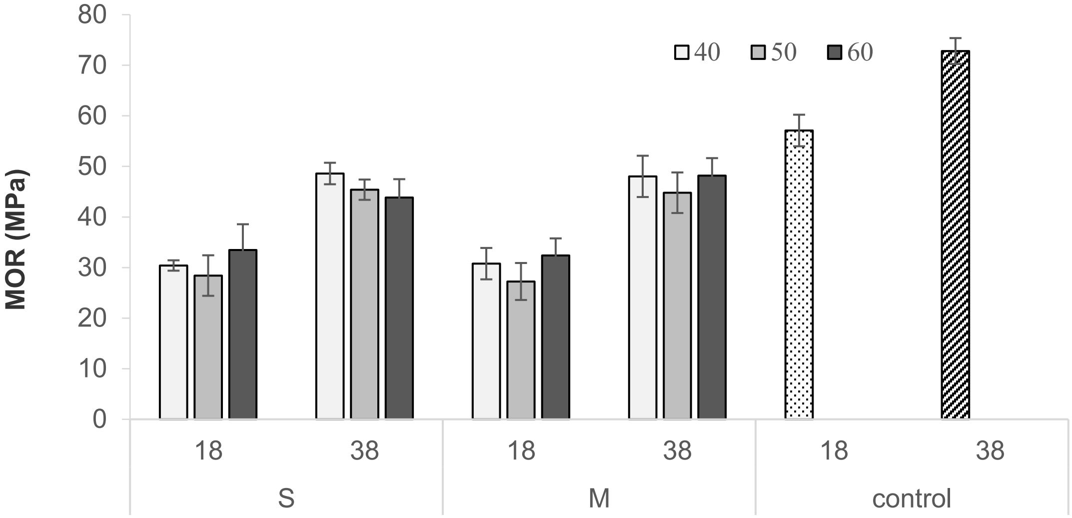

Fig. 2. Effect of heat treatment on modulus of rapture (tree age: 18, 38; timber thickness: 40, 50 and 60 mm; and timber depth: S and M)

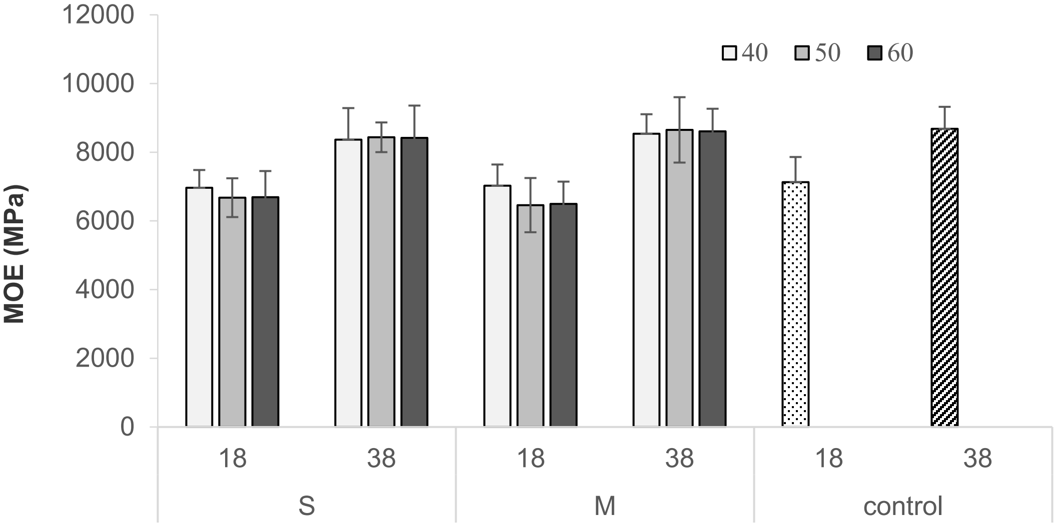

Fig. 3. Effect of heat treatment on modulus of elasticity (tree age: 18, 38; timber thickness: 40, 50 and 60 mm; and timber depth: S and M)

The modulus of elasticity of the heat-treated and control samples is shown in Fig. 3. Although heat treatment did not have a considerable effect on the modulus of elasticity, by increasing tree age the MOE values were increased in heat-treated and control samples. In general, there were few differences between thicknesses of 40, 50, and 60 mm and surface (S) and middle (M) depth of timber in 18- and 38-year-old heat-treated samples. The highest MOE values for 18- and 38-year-old wood control samples were 7,129 and 8,682 MPa, respectively. The lowest MOE was 6,459.5 MPa for 18-year-old heat-treated samples prepared from middle (M) depth by 50 mm thickness.

The results of the impact resistance test are shown in Fig. 4. During heat treatment the impact resistance of heat-treated samples was decreased compared with the control samples. There was a significant difference between the control samples of 18- and 38-year-old wood, such that the impact resistance of the control sample of 38-year-old compared to 18-year-old wood was increased by 52.5%. It would be said that by increasing tree age, the impact resistance of the control samples was increased, which is also evident in heat-treated samples.

The highest impact resistance in control samples of 18- and 38- year-old wood was 24.75 and 37.75 kJ/m^2, respectively. The impact resistance of the samples prepared from the middle (M) depth of the heat-treated timber was decreased compared with the samples prepared from the surface (S) depth. There was not much difference between thicknesses of 40, 50, and 60 mm.

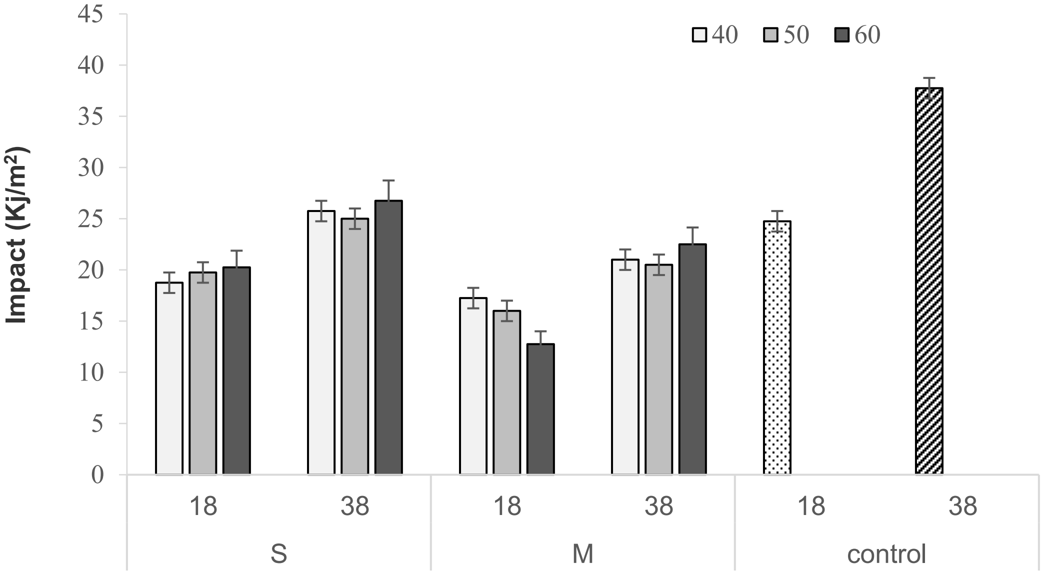

Fig. 4. Effect of heat treatment on impact strength (tree age: 18, 38; timber thickness: 40, 50 and 60 mm; and timber depth: S and M)

Density

Figure 5 shows the air-dry density of the heat-treated and control samples. There was a difference between 18- and 38-year-old control samples. The highest air-dry density value in 18- and 38-year-old control samples was 0.39 and 0.43 g/cm^3, respectively. By increasing tree age from 18 to 38 years, the air-dry density in the heat-treated and control samples were increased. Heat treatment reduced the air-dry density. No considerable difference was observed between thicknesses of 40, 50, and 60 mm and surface (S) and middle (M) depth of 18 and 38-year-old heat-treated samples.

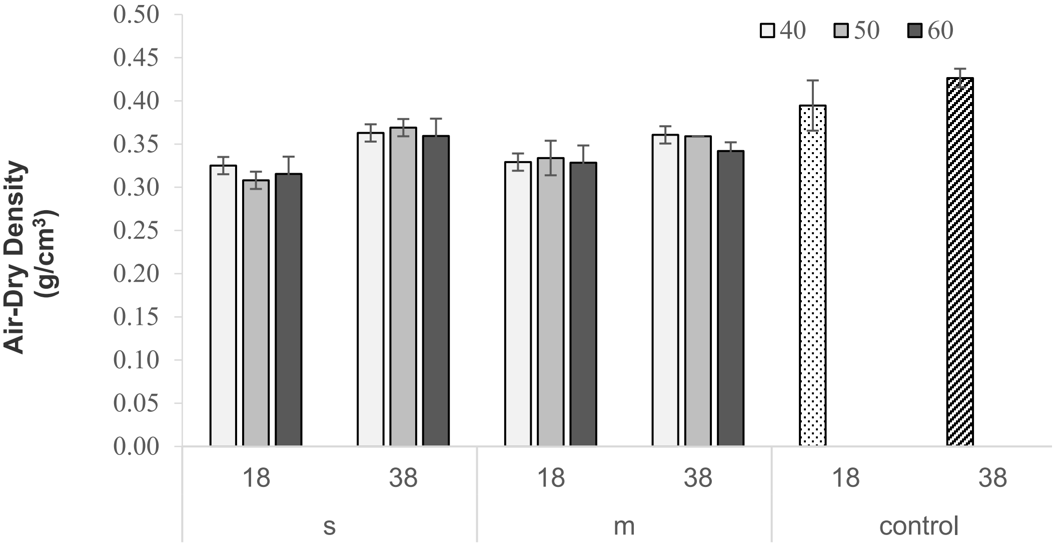

Fig. 5. Effect of heat treatment on air-dry density (tree age: 18, 38; timber thickness: 40, 50 and 60 mm; and timber depth: S and M)

Figure 6 shows the basic density of the heat-treated and control samples. Heat treatment did not have any considerable effect on the basic density, but increasing tree age improved the basic density in heat-treated and control samples. The highest basic density in 38-year-old control samples was 0.36 g/cm^3, and the lowest basic density in 18-year-old samples prepared from the surface (S) depth of heat-treated timber with a thickness of 50 mm was 0.29 g/cm^3. In general, much difference was not observed between different thicknesses and surface (S) and middle (M) depth of heat-treated timbers.

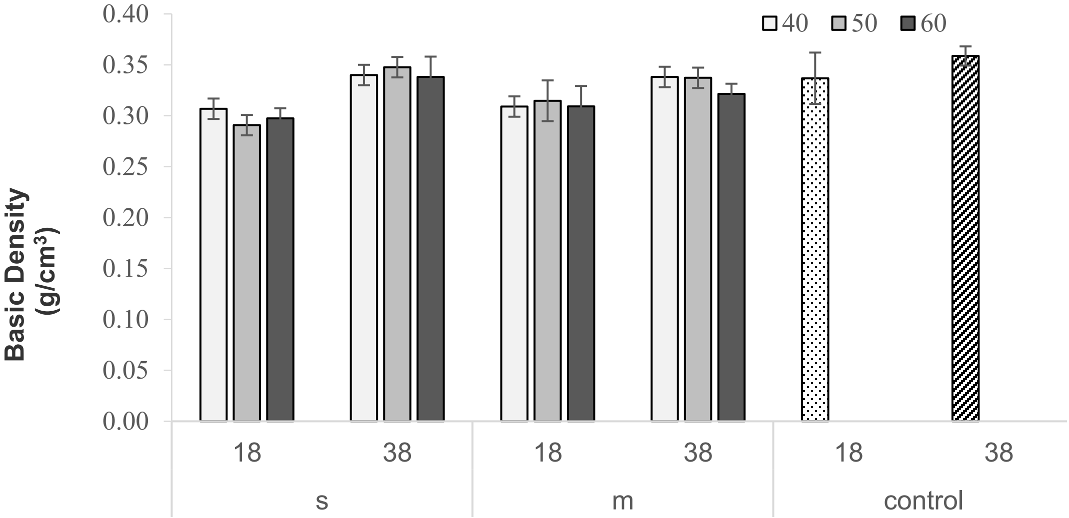

Fig. 6. Effect of heat treatment on basic density (tree age: 18, 38; timber thickness: 40, 50 and 60 mm; and timber depth: S and M)

Figure 7 shows the oven-dry density of heat-treated and control samples. The figure shows that heat treatment reduced the oven-dry density. There was a considerable difference between 18- and 38-year-old control samples. The highest dry density value in

18- and 38-year-old control samples was 0.37 and 0.40 g/cm^3, respectively. In the control and heat-treated samples, the oven-dry density was increased with increasing tree age from 18 to 38 years. There was little difference between thicknesses of 40, 50, and 60 mm and surface (S) and middle (M) depth of the 18 and 38-year-old heat-treated timbers.

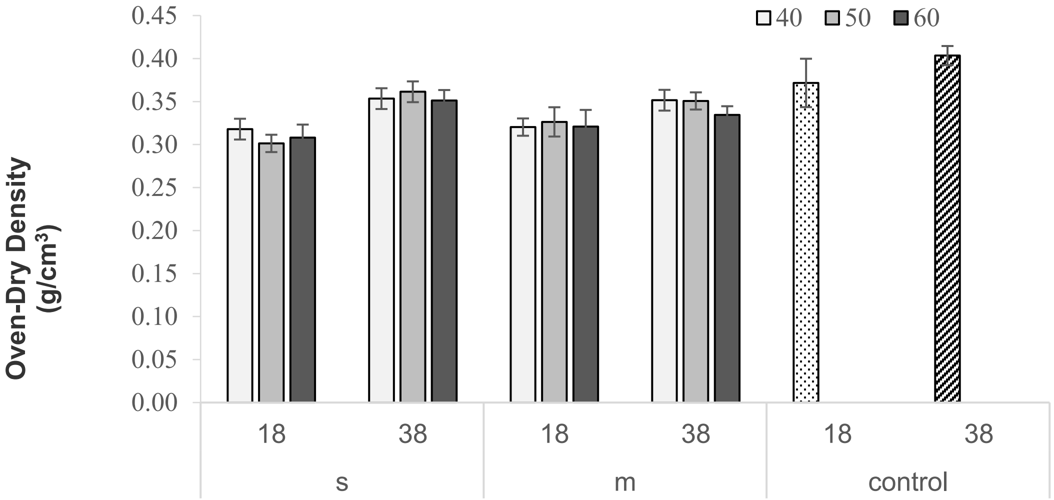

Fig. 7. Effect of heat treatment on oven-dry density (tree age: 18, 38; timber thickness: 40, 50 and 60 mm; and timber depth: S and M)

In this study, the effect of tree age, thickness, and depth of heat-treated timber at a constant temperature of 212 °C on density and mechanical properties were investigated. The results indicated that mechanical properties of heat-treated wood samples such as MOR and impact strength were decreased compared to the control samples but MOE did not decrease considerably. These findings are consistent with the results of previous studies (Hill 2006; Yildiz *et al*. 2006; Zhao *et al*. 2019; Gennari *et al*. 2021). Reduction of mechanical properties is mainly attributed to the thermal degradation of wood chemical components (Taghiyari *et al*. 2020). Hemicellulose in the cell wall is degraded by high temperatures, so wood strength is decreased (Bayani *et al*. 2019). High temperature accelerated the degradation of polysaccharides which results in the production of acetic acid, formic acid, and furfural (Boonstra and Tjeerdsma 2006). The presence of acid in the cell wall breaks down the glycosidic bonds of the cellulose polymer (Tjeerdsma and Militz 2005; Lionetto *et al*. 2012) and shortens the cellulose chains (Rowell 2005).

The obtained results showed that an increase in tree age from 18 to 38 years resulted in an increase in MOR, MOE, and impact strength in heat-treated and control samples. The fiber length and cell wall thickness is directly related to the age of the tree (Debell *et al*. 2002). It can therefore be said that as tree age increased from 18 to 38 years, fiber length and cell wall thickness increased, and thus, density and mechanical properties of heat-treated and control samples were increased. Bijak and Lachowicz (2021) stated that wood density and mechanical strength is increased significantly by increasing tree age. Increasing the thickness of timber from 40 to 60 mm and depth of the timber (surface and middle depth) did not significantly affect density and mechanical properties.

Heat treatment reduces the mass and the density of wood (Kozakiewicz *et al*. 2019). The main reason for the decrease in density in heat-treated wood is related to the degradation and reduction of wood carbohydrates, especially hemicelluloses and their constituent monosaccharides like xylose, mannose, arabinose, and galactose (Brito *et al*. 2008; Guller 2012). One important reason for the decrease in density during heat treatment is the destruction of hemicelluloses and evaporation of the extractive materials (Esteves and Pereira 2009; Aytin *et al.* 2015; Kozakiewicz *et al*. 2019). Decreasing the equilibrium moisture content (EMC) of the wood during heat treatment is another reason for reduced density (Kozakiewicz *et al*. 2019). The main reason for the decrease in moisture content of heat-treated wood at high temperatures is the modification of the wood polymer structure arrangement and the possible softening of lignin (Marcon *et al*. 2021), decrease of wood carbohydrates content as a result of decomposition of a large part of wood cell wall hemicelluloses (Gennari *et al*. 2021), and increase in lignin's proportional content (Abreu *et al*. 2008) that ultimately results in reduction of the wood ability to absorb water and decrease of the equilibrium moisture content of wood (Brito *et al*. 2008).

According to the reduction of mechanical strength of poplar wood due to heat treatment at high temperatures, it is recommended not to use this wood as a pillar in the structures that need to withstand loads. Due to the improvement of some properties of wood during the heat treatment process such as reducing wettability, increasing dimensional stability, and increasing the durability of wood (Jamsa and Viitaniemi 2001; Aytin *et al*. 2015), heat-treated wood can be used for cladding, garden furniture, outdoors, flooring, sauna, and bathroom furnishing (ThermoWood Handbook 2003).

CONCLUSIONS

1. Due to the Thermowood heat treatment, mechanical strengths were decreased. The modulus of rupture (MOR) and impact strength showed the highest decreases, but the modulus of elasticity (MOE) did not show a considerable decrease.
2. Increasing the age of the black poplar tree had positive effects on the strengths and densities of heat-treated wood and control.
3. Densities (oven-dry, air-dry, and basic) of heat-treated samples were decreased compared with control samples.
4. Increasing the thickness of black poplar heat-treated timbers had no considerable effect on the strengths and densities of heat-treated wood samples.
5. Surface and middle depth of heat-treated timbers had a positive effect on the impact strength but had no considerable effect on densities and other strengths.

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