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Influence of Densification on Mechanical Properties of Thermally Pretreated Spruce and Poplar Wood

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The effects of mechanical densification on density, Brinell hardness, bending strength (MOR), modulus of elasticity (MOE), and compression strength (CS) of thermally pretreated spruce (*Picea orientalis*) and poplar (*Populus nigra*) wood samples were investigated. Thermal treatment was applied on the wood samples at four different temperatures (140 °C, 160 °C, 180 °C, and 200 °C) and two different durations (7 h and 9 h) under atmospheric pressure. Wood samples were then densified by compression at a temperature of 150 °C to two degrees (20% and 40%) of compression. The results indicated that the density, hardness, and MOR values of both compressed and non-compressed thermally pretreated spruce and poplar samples decreased with increasing treatment temperature and duration. At temperatures below 200 ºC, the MOE was generally increased in thermally pretreated samples. However, the MOE was reduced in thermally pretreated samples at 200 ºC compared to the untreated samples. Additionally, all thermal pretreatments increased CS values in compressed and non-compressed wood samples. The CS tended to decrease in thermally pretreated samples (especially 40% compressed) at 200 ºC. After the densification, all of the strength properties tested in thermally pretreated samples increased significantly depending on the increase in compression ratio.

Keywords: Densification; Hardness; Mechanical strength; Thermal treatment; Wood material

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INTRODUCTION

Thermal modification, or heat treatment, of wood is a physical process based on the treatment of wet or dried wood in a kiln or vessel and at relatively high temperatures (150 °C to 260 °C) without using any chemicals. Although thermal treatments are physical processes, they cause chemical changes in the basic components of wood (cellulose, hemicelluloses, and lignin) that affect properties such as hygroscopicity, dimensional stability, permeability, and decay resistance in wood (Boonstra 2016). Thermal treatments have an important place in the woodworking industry. In many countries, different thermal treatment methods and processes are used, and these applications are being developed daily (Esteves and Pereira 2008; Şahin and Güler 2018). The thermal treatment of wood is increasingly recognized as an environmentally friendly technique to improve the properties of wood species that are not durable, especially in external part applications (Hill 2006).

As a result of the high temperatures applied to the wood during the thermal treatment, some permanent changes or degradation occur in the structure of the chemical compounds of wood (Kocaefe *et al.* 2008; Tümen *et al.* 2010; Sikora *et al.* 2018). Thermal

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degradation of the chemical compounds of wood occurs first in hemicelluloses and then in cellulose and lignin (Yang *et al.* 2007). The main effect gained after thermal treatment is the decrease in the hygroscopicity of wood (Boonstra 2016). The resistance against biological degradation (Lekounougou and Kocaefe 2014; Yalçın and Şahin 2015) of thermally treated wood that has gained new properties increases, and dimensional stability improves (Kaygın *et al.* 2009; Cai *et al.* 2013; Kocaefe *et al.* 2015). In addition, heat insulation (Şahin Kol and Sefil 2011) increases in thermally treated wood, and the color can be changed homogeneously to darker tones (Gündüz *et al.* 2010; Toker *et al.* 2016; Pelit 2017). In contrast, as a result of thermal degradation of wood components, the brittleness of thermally treated wood increases and most of the mechanical strength properties are reduced (Boonstra *et al.* 2007; Korkut *et al.* 2008; Cui *et al.* 2017). Where high mechanical strength is required for structural applications, the use of thermally treated wood is not suitable (Esteves and Pereira 2008).

High-density wood materials are preferred in structural applications and where strength is important because of their strong mechanical properties. However, due to the limited availability of high-density wood sources, these species of wood are difficult to obtain and generally have high costs. The density of relatively low-density wood species can be increased by densification processes. Thus, it is possible to obtain a higher specific strength than that of most structural metals and alloys. These properties make densified wood a cost-effective, lightweight, and high-performance alternative (Song *et al.* 2018; Fang *et al.* 2019). The main purpose of the densification is to increase the hardness, abrasion resistance, and mechanical strength properties of the wood (Báder *et al.* 2018). Generally, three different methods are used for the densification of wood material. The first is the mechanical compression of wood under high pressure under the influence of heat and/or steam. The second is to fill the cell cavities of wood after impregnation with synthetic or natural resins. The third is the combined use of mechanical compression and impregnation methods (Kutnar *et al.* 2008). Compared to mechanical compression processes, the resin impregnation processes affect the natural structure and sustainability of wood and are generally more costly (Navi and Heger 2004). Additionally, the brittleness of the wood increases due to the properties of the resin (Kollmann *et al.* 1975).

Most of the mechanical strength properties of wood are closely related to wood density (Sandberg *et al.* 2013). After mechanical densification, the density of wood can theoretically be increased to a value close to that of the cell wall, approximately 1500 kg/m^3, and thereby achieve considerable improvements in strength properties (Báder *et al.* 2018). Many previous studies have reported significant increases in the strength properties of mechanically densified wood (Dubey *et al.* 2016; Kariz *et al.* 2017; Pelit *et al.* 2018; Şenol 2018; Song *et al.* 2018; Gao *et al.* 2019). Additionally, the biological resistance of the mechanically densified wood increases depending on the compression ratio (Pelit and Yalçın 2017). The main issue associated with mechanically densified wood is the fixation of the compressed thickness. Mechanically densified wood tends to return to its original dimensions prior to compression when exposed to water or heat (Navi and Heger 2004; Laine *et al.* 2013).

The aim of this study is to determine the effect of mechanical final densification on improving the strength properties of wood that decrease as a result of thermal treatment. For this purpose, thermally pretreated spruce and poplar woods were mechanically densified with two different compression ratios. Tests on density, Brinell hardness, bending strength, modulus of elasticity, and compression strength parallel to the grain were

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performed to determine the effect of mechanical densification on the strength properties of thermally pretreated wood samples.

EXPERIMENTAL

Materials

In this study, Eastern spruce (*Picea orientalis*) and black poplar (*Populus nigra*) wood, which have relatively low densities, were used. Trees were supplied as round wood from a lumber yard in Istanbul, Turkey. Round wood was cut from the sapwood with a band sawing machine in accordance with the study methodology. Attention was paid to ensure that no rot, knot, crack, or density difference was present in the samples as per TS 2470 (1976). Sapwood planks were subjected to natural drying to approximately 12% moisture content, and then cut with a tolerance of 15% to 20% from the draft dimensions of the samples to be used for densification.

Thermal treatment

Thermal treatment was conducted in a laboratory-type oven (Elektro-mag M420P; Labomar Quality Control and Testing Devices Ind. Inc., Maltepe, Istanbul) and at atmospheric pressure. The samples placed in the oven were first stored at 103 ± 2 °C until they became fully dry (approximately 30 h to 36 h). The samples were then separately thermally treated at target temperatures (140 °C, 160 °C, 180 °C, and 200 °C) for 7 h and 9 h. The total duration of thermal treatment for each group was 40 h to 47 h. A certain number of wood samples were not thermally treated for use as control samples. All samples (thermally treated and untreated) remained in a conditioning cabin (relative humidity (RH) 65 ± 3% and 20 ± 2 °C) until they reached a stable weight, and then they were cut to the dimensions of 320 mm × 20 mm (longitudinal direction × tangential direction) and thicknesses 20 mm (for non-compressed samples), 25 mm, and 33.3 mm (radial direction). Wood thicknesses were prepared differently in order to achieve the targeted compression ratios (20% and 40%).

Densification

Wood samples were densified using special metal molds in a hydraulic test press (SSP-180 T; Cemilusta Wood Working Machinery Ind. Inc., Başakşehir, Istanbul). The densification was performed at 150 °C with compression ratios of 20% and 40%. Compression ratios have been determined by taking care not to cause deformation such as fracture and splitting especially in thermally treated samples at high temperature. Channels 10 mm in depth and 20 mm width were opened in the metal molds used for densification. The samples placed in the channels were preheated in a hot press for 20 min. Afterwards, the compression of the wood samples was performed in the radial direction with a loading speed of 60 mm/min. To achieve the targeted wood thickness (20 mm), the load was applied until the metal molds contacted each other (Fig. 1).

The compressed samples were kept under pressure for 10 min and then were removed from the press together with the molds and cooled to room temperature under an average pressure of 0.5 MPa to minimize the spring-back effect. After the densification process, samples remained in a conditioning cabin (RH 65 ± 3% and 20 ± 2 °C) until they reached a stable weight according to TS 2471 (1976). The test samples were prepared in a number sufficient to accommodate ten repetitions ($n = 10$) for each variable.
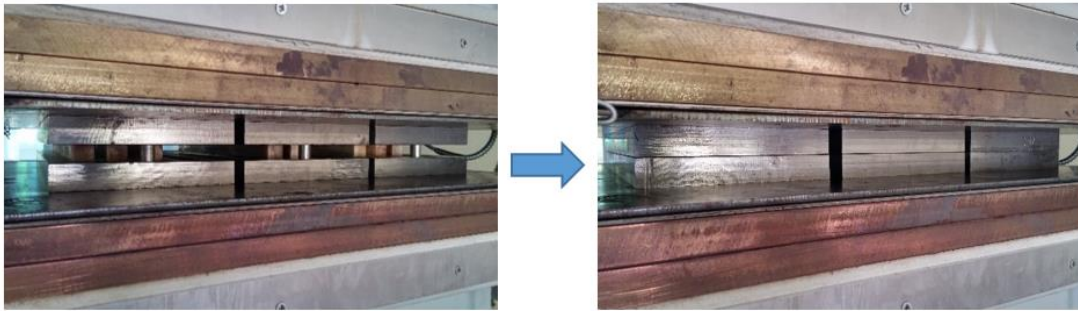
Fig. 1. Densification of wood samples using metal molds

Methods

Determination of density and Brinell hardness

Air-dry density of the wood samples was determined according to TS 2472 (1976). The mass of each sample (M) was measured on an analytical balance, with a sensitivity of ± 0.01 g. Dimensions (length, width, and thickness) were measured with a vernier caliper with ± 0.01-mm sensitivity, and volumes (V) were determined. The air-dry density (δ) was calculated using Eq. 1:

$$\delta \,(\text{g/cm}^3) = M / V \tag{1}$$

The radial and tangential Brinell hardness of the wood samples were determined according to TS 2479 (1976). A 10-mm diameter sphere (steel ball) at the end of the load application arm was set to the centre of the test material, and a load was applied for 30 s. The load was released in 15 s, and the diameter of the indentation made by the steel ball was measured using a digital caliper with ± 0.01-mm sensitivity and magnifier. The Brinell hardness (HB) was calculated using Eq. 2,

$$HB \,(N/mm^2) = 2.F/[\pi.D(D - \sqrt{D^2 - d^2})] \tag{2}$$

where F is the force applied (N), d is the diameter of the indentation made by the steel ball on the surface of the test material (mm), and D is the diameter of the steel ball (mm).

Determination of bending strength, modulus of elasticity, and compression strength

Bending strength (or modulus of rupture) (MOR) and modulus of elasticity (MOE) of the samples were determined according to TS 2474 (1976). The MOR and MOE values were calculated using Eqs. 3 and 4,

$$\text{MOR (N/mm}^2) = 3P_{max}L / 2bd^2 \tag{3}$$

$$\text{MOE (N/mm}^2) = PL^3 / 4bd^3\Delta \tag{4}$$

where P is the load difference in elasticity zone (N), L is the supporting span (mm), b is the width of the samples (mm), d is the thickness (depth) of the samples (mm), Δ is deflection at mid-length below the proportion deflection limit (mm), and P_{max} is the maximum load when the sample is broken (N).

Compression strength parallel to the grain (CS) of the samples was determined according to ISO 13061-17 (2017). The CS value was calculated using Eq. 5,

$$\text{CS (N/mm}^2) = P_{max} / bd \tag{5}$$

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where P_{max} is the maximum load applied to the samples (N), b is the width of the samples (mm), and d is the thickness of the samples (mm).

Statistical analyses

An MSTAT-C 2.1 statistical software program (Michigan State University, East Lansing, MI, USA) was used to evaluate the data obtained from the tests. Analysis of variance (ANOVA) tests were performed to determine the effect of thermal treatment and densification modifications on some technological and mechanical properties of spruce and poplar woods at the 0.05 significance level. Significant differences between the variables were compared using Duncan's test.

RESULTS AND DISCUSSION

Density and Brinell Hardness

The effect of thermal treatment and compression ratio factors on the air-dry density and Brinell hardness (radial and tangential direction) for spruce and poplar woods was statistically significant ($P \leq 0.05$). Duncan's one-way comparison results conducted for mean values of density and Brinell hardness measurements from wood samples thermally pretreated and compressed are shown in Table 1.

Regarding thermal treatment conditions, the highest density average was found in the untreated samples, and the lowest was determined in thermally treated samples for 7 h and 9 h at 200 ℃ (Table 1). In both control and compressed wood samples, the density value was generally reduced with increases in thermal pretreatment temperature and duration (Fig. 2a). The air-dry density value of spruce and poplar samples, which were thermally treated at 200 ℃ for 9 h, decreased 9% and 8%, respectively, compared to untreated samples. It could be said that the decrease in the equilibrium moisture content (EMC) of the wood samples and the mass losses in the samples were effective on the decreases in density after thermal treatment. The average EMC of untreated spruce and poplar samples was determined as 12.7% and 11.1%, respectively, whereas the average EMC of spruce and poplar samples thermally treated at 200 ℃ for 9 h was 8.1% and 5.9%, respectively. In the literature, it was reported that due to changes in the chemical structure of thermally treated wood, it was less hygroscopic and consequently reduced EMC (Boonstra 2008; Esteves and Pereira 2008). Additionally, the destruction of the main components of wood (especially hemicellulose) and the evaporation of the extractives caused mass losses in the thermally treated wood (Boonstra 2008; Esteves *et al.* 2008).

With respect to compression ratio, the highest density average for both wood species was found in the samples compressed with the ratio of 40%, while the lowest was obtained in the non-compressed samples (Table 1). The density values determined in both untreated and thermally treated samples were parallel with the compression ratios and higher density values that were obtained at high compression ratio (40%). Moreover, the increase in density after compression processes was similar or close in untreated and thermally treated samples (Fig. 2a). After densification, the density value of untreated spruce and poplar samples increased up to 45% and 46%, respectively, and the density value of spruce and poplar samples with thermal treatment at 200 °C for 9 h increased up to 41% and 43%, respectively. It has been stated in previous studies that increases in density can be explained by a decrease in the void volume of wood as a result of

compression and an increase in the cell wall amount per unit volume (Ülker *et al.* 2012; Pelit *et al.* 2018).

Table 1. Duncan's Test Results for Mean Values of Density and Brinell Hardness

Wood Species	Factor	Density (g/cm^3)		Hardness (Radial Direction) (N/mm^2)		Hardness (Tangential Direction) (N/mm^2)	
		Mean	SG	Mean	SG	Mean	SG
Spruce	Thermal Treatment						
	Untreated	0.457	a	18.04	abc	23.38	ab
	140 °C, 7 h	0.448	ab	18.63	ab	23.08	ab
	140 °C, 9 h	0.454	a	19.14	a	24.72	a
	160 °C, 7 h	0.445	ab	18.34	ab	23.10	ab
	160 °C, 9 h	0.438	abc	17.66	bc	22.08	bc
	180 °C, 7 h	0.436	abc	16.91	c	21.39	cd
	180 °C, 9 h	0.428	bc	14.95	d	19.87	de
	200 °C, 7 h	0.419	c	14.55	d	18.51	ef
	200 °C, 9 h	0.418	c	13.90	d	17.62	f
	Compression Ratio						
	Non-compressed	0.371	c	14.43	c	13.56	c
	20%	0.422	b	16.51	b	20.53	b
	40%	0.520	a	19.77	a	30.49	a
Poplar	Thermal Treatment						
	Untreated	0.488	a	20.37	bc	27.42	a
	140 °C, 7 h	0.480	ab	22.10	a	26.83	ab
	140 °C, 9 h	0.475	ab	22.33	a	25.70	bc
	160 °C, 7 h	0.478	ab	21.79	a	25.61	bc
	160 °C, 9 h	0.475	ab	21.44	ab	25.23	c
	180 °C, 7 h	0.471	b	20.22	c	22.85	d
	180 °C, 9 h	0.466	bc	19.08	d	21.63	de
	200 °C, 7 h	0.454	cd	18.12	d	20.28	ef
	200 °C, 9 h	0.448	d	18.22	d	19.64	f
	Compression Ratio						
	Non-compressed	0.395	c	16.71	c	14.61	c
	20%	0.455	b	19.47	b	22.41	b
	40%	0.562	a	25.05	a	34.71	a

SG: Statistical group (different letters denote a significant difference)

According to Table 1, the highest Brinell hardness values in the radial and tangential direction were determined in the samples thermally treated at 140 °C for 9 h. However, the hardness value in tangential direction of poplar wood was found to be higher in the untreated samples. The lowest radial and tangential hardness values were determined in the samples thermally treated at 200 °C for 7 h and 9 h. At lower temperatures (140 °C and 160 °C), the radial hardness values of thermally pretreated spruce and poplar samples tended to increase compared to untreated samples. However, in parallel with the increases in thermal treatment temperature and duration starting from the 180 °C limit, the radial hardness values of the wood samples decreased (Fig. 2b). Tangential hardness values of thermally pretreated wood samples decreased due to the increases in thermal treatment temperature and duration (Fig. 2c).
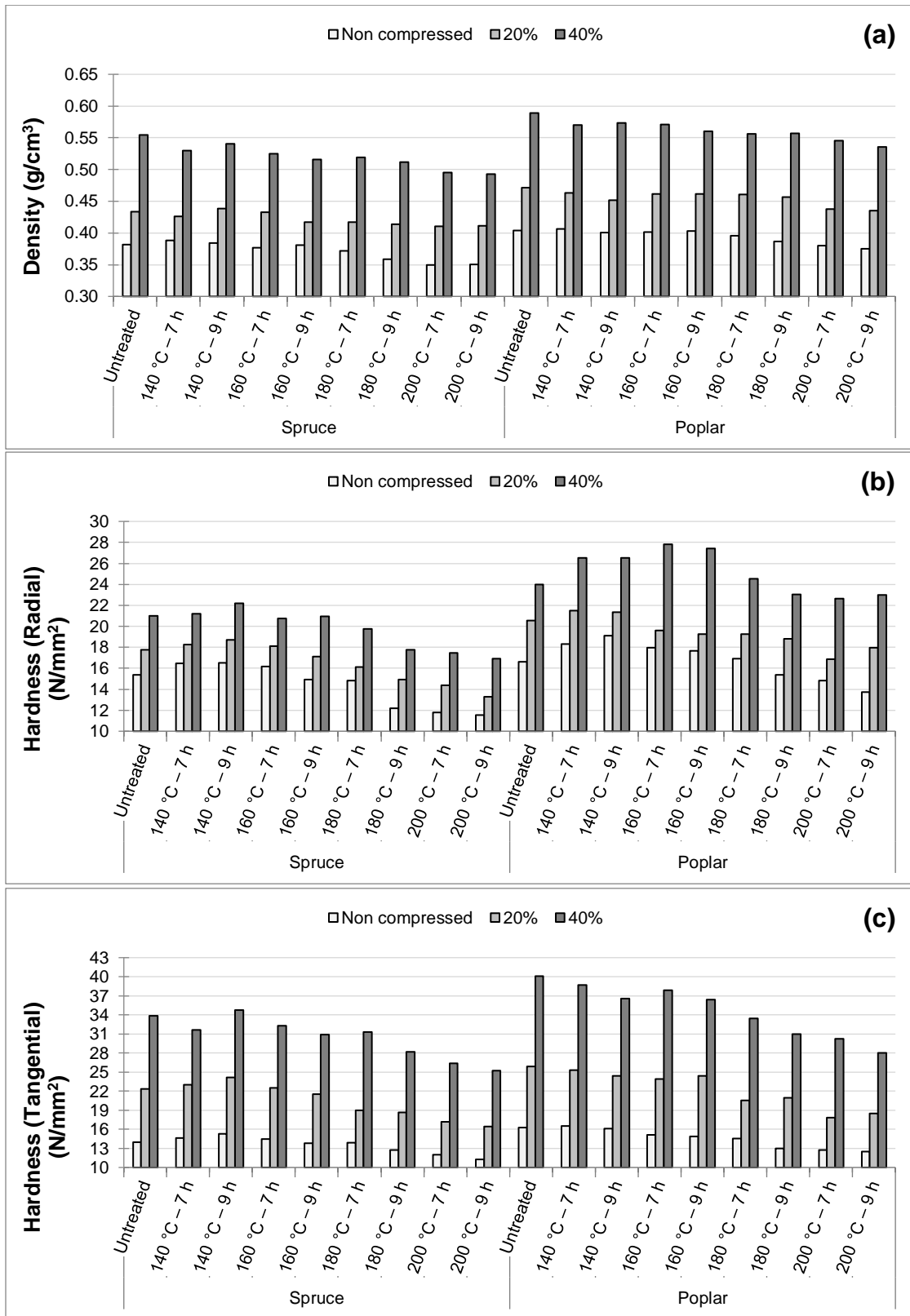
Fig. 2. Average density and hardness (radial and tangential direction) values of thermally pretreated and compressed wood samples

According to the untreated samples, radial hardness values of spruce and poplar samples that were thermally treated at 200 °C for 9 h decreased 23% and 11%, respectively, and tangential hardness values decreased 25% and 28%, respectively. It could be said that these decreases in hardness values were caused by density losses and thermal decomposition in the wood samples after thermal treatment (especially high temperature). In a previous study, it was reported that deformations, such as rupture and fragmentation in the cell wall of the wood, especially after the thermal treatment applied at high temperature, were effective in decreasing the hardness values of the wood samples (Budakçı *et al.* 2016). In contrast, Kariz *et al.* (2017) reported that the lower EMC in thermally treated wood compared to untreated wood positively affected the hardness values.

Regarding compression ratio, the highest Brinell hardness average in both radial and tangential directions for both wood species was found in the 40% compressed samples, and the lowest was obtained in non-compressed samples (Table 1). After the densification process, radial and tangential hardness values of untreated and thermally treated samples increased depending on the increase in compression ratio. These increases in hardness were more pronounced in tangential hardness values of wood samples (Figs. 2b and 2c). The results of this study were consistent with the results of the previous studies (Pelit *et al.* 2015; Budakçı *et al.* 2016). According to the non-compressed samples, the radial hardness of the spruce and poplar 40% compressed samples were increased by 37% and 50%, respectively, while the tangential hardness were increased by 125% and 138%, respectively. Brinell hardness values of wood samples were decreased as a result of thermal treatment increased with densification post-processes. In particular, significant increases were achieved in tangential direction hardness values. In previous studies, it was stated that the hardness of wood material increased after the densification processes in parallel with the increase in the compression ratio and/or wood density (Boonstra and Blomberg 2007; Ülker *et al.* 2012; Pelit *et al.* 2015).

Bending Strength, Modulus of Elasticity, and Compression Strength

According to ANOVA results, the effect of thermal treatment and compression ratio factors on bending strength (MOR), modulus of elasticity (MOE), and compression strength parallel to the grain (CS) for both wood species was statistically significant. Duncan's one-way comparison results conducted for mean values of MOR, MOE, and CS are shown in Table 2.

Regarding thermal treatment conditions, the highest MOR average was determined in untreated samples for both wood species, and the highest MOE average was obtained in the samples thermally treated at 140 °C and 160 °C for spruce wood and in the samples thermally treated at 160 °C and 180 °C for poplar wood. Furthermore, the lowest MOR and MOE averages of spruce and poplar wood was obtained in the samples thermally treated at 200 °C for 9 h (Table 2). The MOR values increased slightly in the non-compressed samples thermally treated at 140 °C and 160 °C. However, in all other wood samples with thermal pre-treatment (compressed and non-compressed), the MOR values were gradually decreased due to the increases in thermal treatment temperature and duration (Fig. 3a). According to untreated samples, the MOR values of spruce and poplar samples thermally treated at 200 °C for 9 h decreased 35% and 37%, respectively. It is thought that the possible degradation in the structure of the chemical components of the wood samples after the thermal treatment (especially at high temperature) has an effect on the results (Yang *et al.* 2007; Kocaefe *et al.* 2008; Tümen *et al.* 2010). In previous studies,

it was stated that after the thermal treatment at high temperatures, the brittleness of the wood increased and the mechanical strength properties of the wood decreased (Boonstra *et al.* 2007; Kocaefe *et al.* 2008; Korkut *et al.* 2008). Additionally, it was stated that thermal treatment caused some negative effects, such as lower bending strength in wood material and the decrease in strength depending on thermal treatment method, treatment temperature, duration, absence of oxygen, and wood species (Hill 2006).

Table 2. Duncan's Test Results for Mean Values of MOR, MOE, and CS in the Thermally Pretreated and Compressed Wood Samples

Wood Species	Factor	MOR (N/mm^2)		MOE (N/mm^2)		CS (N/mm^2)	
		Mean	SG	Mean	SG	Mean	SG
Spruce	Thermal Treatment						
	Untreated	68.99	a	8388	abc	36.48	c
	140 °C, 7 h	67.58	ab	8665	ab	39.73	bc
	140 °C, 9 h	68.34	ab	8912	a	39.93	bc
	160 °C, 7 h	64.36	bc	8830	a	41.04	ab
	160 °C, 9 h	60.45	cd	8845	a	41.72	ab
	180 °C, 7 h	57.26	de	8660	ab	44.35	a
	180 °C, 9 h	54.96	ef	8636	abc	43.82	a
	200 °C, 7 h	50.62	f	8062	bc	42.96	ab
	200 °C, 9 h	45.00	g	8010	c	43.04	ab
	Compression Ratio						
	Non-compressed	51.96	c	7327	c	36.54	c
	20%	57.88	b	8322	b	40.80	b
	40%	69.34	a	10020	a	47.02	a
Poplar	Thermal Treatment						
	Untreated	78.39	a	8583	bc	42.88	e
	140 °C, 7 h	77.55	ab	8578	bc	44.83	d
	140 °C, 9 h	77.31	ab	8816	ab	44.61	d
	160 °C, 7 h	75.80	ab	8907	a	45.01	cd
	160 °C, 9 h	74.63	b	8926	a	46.48	abc
	180 °C, 7 h	67.82	c	8796	ab	47.49	a
	180 °C, 9 h	63.17	d	8904	a	46.85	ab
	200 °C, 7 h	54.53	e	8301	cd	46.73	ab
	200 °C, 9 h	49.61	f	8090	d	45.54	bcd
	Compression Ratio						
	Non-compressed	57.60	c	7117	c	39.68	c
	20%	67.54	b	8557	b	45.26	b
	40%	81.12	a	10290	a	51.86	a

SG: statistical group (different letters denote a significant difference)

The MOE values in non-compressed and 20% compressed samples, which were thermally pretreated, tended to increase compared to untreated samples. However, in these samples thermally pretreated at 200 °C, the MOE decreased slightly. Additionally, the MOE generally tended to increase in the samples 40% compressed with thermal pretreatment at 140 °C and 160 °C. However, in these samples, MOE values decreased up to 11% with the increase of thermal pretreatment temperature starting from the 180 °C limit (Fig. 3b). Thermal pretreatments applied at temperatures below 200 °C generally had a positive effect on the MOE values of the samples. It could be said that the decrease of EMC

value in thermally pretreated samples had an effect on these results. Esteves and Pereira (2008) stated that the lower equilibrium moisture content might positively affect the strength properties of thermally treated wood. In contrast, softening of the cell wall due to the plasticization of the main wood components (especially lignin and the remaining hemicelluloses) during the thermal treatment might have had an effect on the results (Boonstra and Blomberg 2007).

Regarding compression ratio, the highest MOR and MOE average of wood samples was in the samples compressed with the ratio of 40%, while the lowest was found in the non-compressed samples (Table 2). Figures 3a and 3b showed that MOR and MOE increased in all wood samples (untreated and thermally treated) depending on the compression ratio after densification. The higher strength values were found at high compression ratio (40%). This situation could be explained by the amount of density increase in the samples depending on the compression ratio. It is well known that most of the mechanical strength properties of wood are closely related to the density of wood. Similar results have been reported in previous studies on wood densification (Kutnar *et al.* 2008; Pelit *et al.* 2018). Compared to non-compressed samples, the MOR increased 33% and 41%, respectively, and the MOE increased 37% and 45%, respectively, in 40% compressed spruce and poplar samples. The reductions in MOR values of wood samples due to thermal treatment at 200 °C were largely tolerated after densification. Additionally, significant gains were achieved in MOE strength.

According to Table 2, the highest CS average for both wood species regarding thermal treatment conditions was obtained in the samples thermally treated at 180 °C for 7 h and 9 h, and the lowest CS was found in the untreated samples. The CS values were increased in all thermally pretreated samples (compressed and non-compressed). The CS was generally increased due to the increases in temperature and duration of the thermal treatment up to 180 °C (including this temperature). However, the CS showed a tendency to decrease in the samples (especially 40% compressed) thermally treated at 200 °C (Fig. 3c). According to the untreated samples, the CS value increased 22% and 11%, respectively, in the spruce and poplar wood thermally treated at 180 °C for 7 h. According to Boonstra (2008), the increase of the CS parallel to the grain might be due to a lower amount of bound water in thermally treated wood. During thermal treatment, the amount of the highly ordered crystalline cellulose increased due to degradation and/or crystallization of amorphous cellulose. Because crystalline cellulose showed significant anisotropy, its stiff and rigid structure might be responsible for the observed increase of the CS parallel to the grain. An increased cross-linking of the lignin polymer network could be another reason for this improvement (Boonstra 2008). An increase in CS value of thermally treated wood was also reported in some previous study results (Boonstra *et al.* 2007; Özçifçi *et al.* 2009; Altınok *et al.* 2010). However, the results of some studies in the literature indicate that CS strength decreases after thermal treatment (especially above 200 °C) (Korkut and Aytin 2015; Perçin and Altınok 2017; Pelit *et al.* 2018). Yıldız *et al.* (2006) demonstrated that the main reason for the decrease in CS strength is the thermal degradation of hemicellulose determined by chemical analysis.

With respect to compression ratio, the highest CS average of spruce and poplar woods was found in the 40% compressed samples, while the lowest was determined in non-compressed samples (Table 2). The CS values of untreated and thermally treated wood samples increased due to the increase in compression ratio as in other strength properties. After densification, the increase in CS values of untreated wood samples was higher than in thermally treated samples (Fig. 3c).
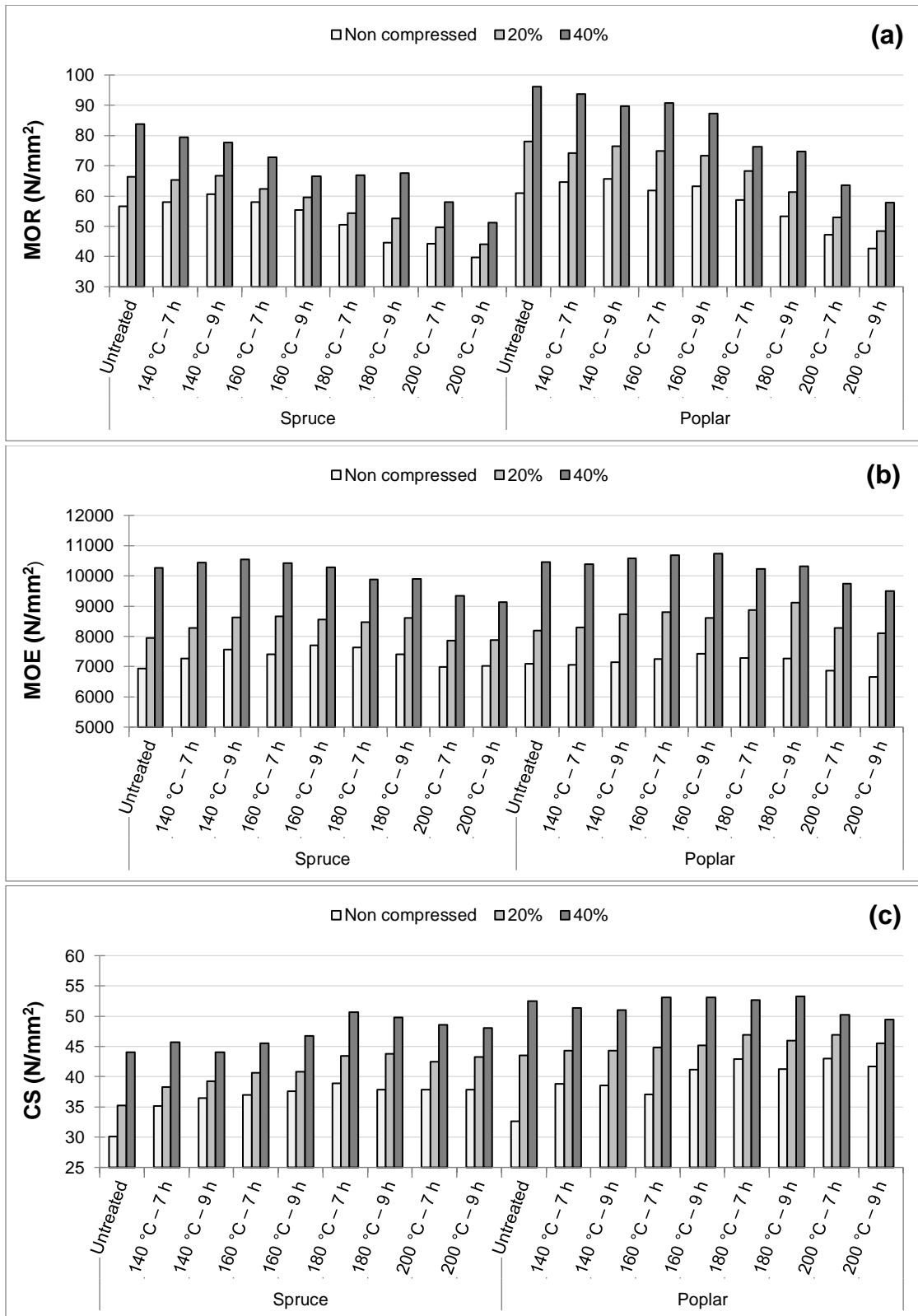
Fig. 3. Average MOR, MOE, and CS values of thermally pretreated and compressed wood samples

This could be explained by the more frequent cell deformations (collapses, breakage, cracking, *etc.*) that occurred after the compression in the thermally treated samples. In the literature, it was emphasized that cell deformations occur more especially in the wood materials densified with high compression rate (Doğu *et al.* 2010; Budakçı *et al.* 2016; Bekhta *et al.* 2017) and this causes a decrease in strength properties. Additionally, it was reported that the type and amount of cell deformation has an important effect on the physical and mechanical properties of the densified wood (Kutnar *et al.* 2009). After densification, CS values were increased by up to 46% and 61% in untreated spruce and poplar woods, respectively, and up to 27% and 28% in thermally pretreated spruce and poplar woods, respectively.

CONCLUSIONS

1. The density, Brinell hardness, and MOR decreased in thermally pretreated spruce and poplar samples (compressed and non-compressed) depending on increase in process temperature and duration. However, at low temperatures (140 °C and 160 °C), the hardness values in the radial direction of thermally pretreated wood samples tended to increase compared to the untreated samples. According to untreated samples, the density, hardness, and MOR values of wood samples thermally pretreated at 200 °C for 9 h decreased up to 9%, 28%, and 37%, respectively.

2. Moreover, thermal pretreatments applied at temperatures below 200 °C had a generally positive effect on the MOE values of spruce and poplar wood. However, the MOE was decreased by up to 11% depending on the treatment duration in the wood samples thermally pretreated at 200 °C. In contrast, CS values increased in all wood samples (compressed and non-compressed) with thermal pretreatment. However, the CS showed a tendency to decrease in the samples (especially 40% compressed) thermally pretreated at 200 °C.

3. The densification processes significantly affected all tested properties of thermally pretreated spruce and poplar wood. After the densification, the density of the wood samples increased due to the increase in the compression ratio and all the strength properties of the samples improved.

4. Compared with spruce wood, more successful results were obtained in poplar wood. Density, hardness, MOR, MOE, and CS were increased by up to 46%, 138%, 41%, 45%, and 61%, respectively, in the samples compressed at 40% ratio compared to non-compressed samples.

5. Significant increases were achieved in the density and strength properties of the wood samples, which had been decreased due to thermal treatment, by the densification post-treatment.

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REFERENCES CITED

Altınok, M., Özalp, M., and Korkut, S. (2010). "The effects of heat treatment on some mechanical properties of laminated beech (*Fagus orientalis* L.) wood," *Wood Res.* 55(3), 131-142.

Báder, M., Bak, M., Németh, R., Rademacher, P., Rousek, R., Horníček, S., Dömény, J., Klímek, P., Kudela. J., Sandberg, D., *et al.* (2018). "Wood densification processing for newly engineered materials," in: *5th International Conference on Processing Technologies for the Forest and Bio-based Products Industries (PTF BPI 2018)*, Freising/Münich, Germany, pp. 255-263.

Bekhta, P., Proszyk, S., Krystofiak, T., Sedliacik, J., Novak, I., and Mamonova, M. (2017). "Effects of short-term thermomechanical densification on the structure and properties of wood veneers," *Wood Material Science & Engineering* 12(1), 40-54. DOI: 10.1080/17480272.2015.1009488

Boonstra, M. J. (2008). *A Two-Stage Thermal Modification of Wood*, Ph.D. Dissertation, Ghent University, Ghent, Belgium, and Université Henry Poincaré, Nancy, France. DOI: 1854/10505

Boonstra, M. J. (2016). "Dimensional stabilization of wood and wood composites," in: *Lignocellulosic Fibers and Wood Handbook: Renewable Materials for Today's Environment*, N. Belgacem, and A. Pizzi (eds.), Wiley, Hoboken, NJ, USA, pp. 629-655. DOI: 10.1002/9781118773727.ch26

Boonstra, M. J., and Blomberg, J. (2007). "Semi-isostatic densification of heat-treated radiata pine," *Wood Sci. Technol.* 41, 607-617. DOI: 10.1007/s00226-007-0140-y

Boonstra, M. J., Van Acker, J., Tjeerdsma, B. F., and Kegel, E. V. (2007). "Strength properties of thermally modified softwoods and its relation to polymeric structural wood constituents," *Ann. For. Sci.* 64(7), 679-690. DOI: 10.1051/forest:2007048

Budakçı, M., Pelit, H., Sönmez, A., and Korkmaz, M. (2016). "The effects of densification and heat post-treatment on hardness and morphological properties of wood material," *BioResources* 11(3), 7822-7838. DOI: 10.15376/biores.11.3.7822-7838

Cai, J., Yang, X., Cai, L., and Shi, S. Q. (2013). "Impact of the combination of densification and thermal modification on dimensional stability and hardness of poplar lumber," *Dry. Technol.* 31(10), 1107-1113. DOI: 10.1080/07373937.2013.775147

Cui, W., Zhang, N., Xu, M., and Cai, L. (2017). "Combined effects of ZnO particle deposition and heat treatment on dimensional stability and mechanical properties of poplar wood," *Sci. Rep.* 7(1), article number 9961. DOI:10.1038/s41598-017-10606-5

Doğu, D., Tırak, K., Candan, Z., and Ünsal, Ö. (2010). "Anatomical investigation of thermally compressed wood panels," *BioResources* 5(4), 2640-2663. DOI: 10.15376/biores.5.4.2640-2663

Dubey, M. K., Pang, S., Chauhan, S., and Walker, J. (2016). "Dimensional stability, fungal resistance and mechanical properties of radiata pine after combined thermo-mechanical compression and oil heat-treatment," *Holzforschung* 70(8), 793-800. DOI: 10.1515/hf-2015-0174

Esteves, B. M., and Pereira, H. M. (2008). "Wood modification by heat treatment: A review," *BioResources* 4(1), 370-404. DOI: 10.15376/biores.4.1.370-404

Esteves, B., Graça, J., and Pereira, H. (2008). "Extractive composition and summative chemical analysis of thermally treated eucalypt wood," *Holzforschung* 62(3), 344-351. DOI: 10.1515/HF.2008.057

Fang, C.-H., Cloutier, A., Jiang, Z.-H., He, J.-Z., and Fei, B.-H. (2019). "Improvement of wood densification process *via* enhancing steam diffusion, distribution, and evaporation," *BioResources* 14(2), 3278-3288. DOI: 10.15376/biores.14.2.3278-3288

Gao, Z., Huang, R., Chang, J., Li, R., and Wu, Y. (2019). "Effects of pressurized superheated-steam heat treatment on set recovery and mechanical properties of surface-compressed wood," *BioResources* 14(1), 1718-1730. DOI: 10.15376/biores.14.1.1718-1730

Gündüz, G., Aydemir, D., and Korkut, S. (2010). "The effect of heat treatment on some mechanical properties and color changes of Uludağ fir wood," *Dry. Technol.* 28(2), 249-255. DOI: 10.1080/07373930903530162

Hill, C. A. S. (2006). *Wood Modification: Chemical, Thermal and Other Processes*, Wiley, Chichester, United Kingdom.

ISO 13061-17 (2017). "Physical and mechanical properties of wood - Test methods for small clear wood specimens - Part 17: Determination of ultimate stress in compression parallel to grain," International Organization for Standardization, Geneva, Switzerland.

Kariz, M., Kuzman, M. K., Sernek, M., Hughes, M., Rautkari, L., Kamke, F. A., and Kutnar, A. (2017). "Influence of temperature of thermal treatment on surface densification of spruce," *Eur. J. Wood Wood Prod.* 75(1), 113-123. DOI: 10.1007/s00107-016-1052-z

Kaygın, B., Gündüz, G., and Aydemir, D. (2009). "Some physical properties of heat-treated paulownia (*Paulownia elongata*) wood," *Dry. Technol.* 27(1), 89-93. DOI: 10.1080/07373930802565921

Kocaefe, D., Huang, X., and Kocaefe, Y. (2015). "Dimensional stabilization of wood," *Curr. For. Rep.* 1(3), 151-161. DOI: 10.1007/s40725-015-0017-5

Kocaefe, D., Poncsak, S., and Boluk, Y. (2008). "Effect of thermal treatment on the chemical composition and mechanical properties of birch and aspen," *BioResources* 3(2), 517-537. DOI: 10.15376/biores.3.2.517-537

Kollmann, F. F. P., Kuenzi, E. W., and Stamm, A. J. (1975). *Principles of Wood Science and Technology. Vol. II: Wood Based Materials*, Springer-Verlag, New York, NY, USA.

Korkut, S., Kök, M. S., Korkut, D. S., and Gürleyen, T. (2008). "The effects of heat treatment on technological properties in red-bud maple (*Acer trautvetteri* Medw.) wood," *Bioresource Technol.* 99(6), 1538-1543. DOI: 10.1016/j.biortech.2007.04.021

Korkut, S., and Aytin, A. (2015). "Evaluation of physical and mechanical properties of wild cherry wood heat-treated using the thermowood process," *Maderas- Cienc. Tecnol.* 17(1), 171-178. DOI: 10.4067/S0718-221X2015005000017

Kutnar, A., Kamke, F. A., and Sernek, M. (2008). "The mechanical properties of densified VTC wood relevant for structural composites," *Holz Roh Werkst.* 66(6), 439-446. DOI: 10.1007/s00107-008-0259-z

Kutnar, A., Kamke, F. A., and Sernek, M. (2009). "Density profile and morphology of viscoelastic thermal compressed wood," *Wood Sci. Technol.* 43(1-2), 57-68. DOI: 10.1007/s00226-008-0198-1

Laine, K., Rautkari, L., Hughes, M., and Kutnar, A. (2013). "Reducing the set-recovery of surface densified solid Scots pine wood by hydrothermal post-treatment," *Eur. J. Wood Wood Prod.* 71(1), 17-23. DOI: 10.1007/s00107-012-0647-2

Lekounougou, S., and Kocaefe, D. (2014). "Durability of thermally modified *Pinus banksiana* (Jack pine) wood against brown and white rot fungi," *Int. Wood Prod. J.* 5(2), 92-97. DOI: 10.1179/2042645313Y.0000000057

Navi, P., and Heger, F. (2004). "Combined densification and thermo-hydro-mechanical processing of wood," *MRS Bull.* 29(5), 332-336. DOI: 10.1557/mrs2004.100

Özçifçi, A., Altun, S., and Yapıcı, F. (2009). "Effects of heat treatment on technological properties of wood," in: *5th International Advanced Technologies Symposium (IATS'09)*, Karabük, Turkey, pp. 1-5.

Pelit, H. (2017). "The effect of different wood varnishes on surface color properties of heat treated wood materials," *J. Fac. For. Istanbul Univ.* 67(2), 262-274. DOI: 10.17099/jffiu.300010

Pelit, H., Budakçı, M., and Sönmez, A. (2018). "Density and some mechanical properties of densified and heat post-treated Uludağ fir, linden and black poplar woods," *Eur. J. Wood Wood Prod.* 76(1), 79-87. DOI: 10.1007/s00107-017-1182-y

Pelit, H., Sönmez, A., and Budakçı, M. (2015). "Effects of thermomechanical densification and heat treatment on density and Brinell hardness of Scots pine (*Pinus sylvestris* L.) and Eastern beech (*Fagus orientalis* L.)," *BioResources* 10(2), 3097-3111. DOI: 10.15376/biores.10.2.3097-3111

Pelit, H., and Yalçın, M. (2017). "Resistance of mechanically densified and thermally post-treated pine sapwood to wood decay fungi," *J. Wood Sci.* 63(5), 514-522. DOI: 10.1007/s10086-017-1641-4

Perçin, O., and Altınok, M. (2017). "Some physical and mechanical properties of laminated veneer lumber reinforced with carbon fiber using heat-treated beech veneer," *Eur. J. Wood Wood Prod.* 75(2), 193-201. DOI: 10.1007/s00107-016-1125-z

Sandberg, D., Haller, P., and Navi, P. (2013). "Thermo-hydro and thermo-hydro-mechanical wood processing: An opportunity for future environmentally friendly wood products," *Wood Materials Science & Engineering* 8(1), 64-88. DOI: 10.1080/17480272.2012.751935

Sikora, A., Kačík, F., Gaff, M., Vondrová, V., Bubeníková, T., and Kubovský, I. (2018). "Impact of thermal modification on color and chemical changes of spruce and oak wood," *J. Wood Sci.* 64(4), 406-416. DOI: 10.1007/s10086-018-1721-0

Song, J., Chen, C., Zhu, S., Zhu, M., Dai, J., Ray, U., Li, Y., Kuang, Y., Li, Y., Quispe, N., *et al.* (2018). "Processing bulk natural wood into a high-performance structural material," *Nature* 554, 224-228. DOI: 10.1038/nature25476

Şahin, H. İ., and Güler, C. (2018). "Effect of heat treatment on the dimensional stability of ash (*Fraxinus angustifolia* Vahl.) wood," *Forestist* 68(1), 42-52. DOI: 10.5152/forestist.2018.005

Şahin Kol, H., and Sefil, Y. (2011). "The thermal conductivity of fir and beech wood heat treated at 170, 180, 190, 200, and 212 C," *J. Appl. Polym. Sci.* 121(4), 2473-2480. DOI: 10.1002/app.33885

Şenol, S. (2018). *Determination of Physical, Mechanical and Technological Properties of Some Wood Materials Treated with Thermo-vibro-mechanical (TVM) Process*, Ph.D. Dissertation, Duzce University, Düzce, Turkey.

Toker, H., Baysal, E., Kötekli, M., Türkoğlu, T., Kart, Ş., Şen, F., and Peker, H. (2016). "Surface characteristics of Oriental beech and Scots pine woods heat-treated above 200 °C," *Wood Res.* 61(1), 43-54.

TS 2470 (1976). "Wood - Sampling methods and general requirements for physical and mechanical tests," Turkish Standards Institute, Ankara, Turkey.

TS 2471 (1976). "Wood - Determination of moisture content for physical and mechanical tests," Turkish Standards Institute, Ankara, Turkey.

TS 2472 (1976). "Wood - Determination of density for physical and mechanical tests," Turkish Standards Institution, Ankara, Turkey.

TS 2474 (1976). "Wood - Determination of ultimate strength in static bending," Turkish Standards Institution, Ankara, Turkey.

TS 2479 (1976). "Wood - Determination of static hardness," Turkish Standards Institute, Ankara, Turkey.

Tümen, İ., Aydemir, D., Gündüz, G., Üner, B., and Çetin, H. (2010). "Changes in the chemical structure of thermally treated wood," *BioResources* 5(3), 1936-1944. DOI: 10.15376/biores.5.3.1936-1944

Ülker, O., İmirzi, Ö., and Burdurlu, E. (2012). "The effect of densification temperature on some physical and mechanical properties of Scots pine (*Pinus sylvestris* L.)," *BioResources* 7(4), 5581-5592. DOI: 10.15376/biores.7.4.5581-5592

Yalçın, M., and Şahin, H. İ. (2015). "Changes in the chemical structure and decay resistance of heat-treated narrow-leaved ash wood," *Maderas- Cienc. Tecnol.* 17(2), 435-446. DOI: 10.4067/S0718-221X2015005000040

Yang, H., Yan, R., Chen, H., Lee, D. H., and Zheng, C. (2007). "Characteristics of hemicelluloses, cellulose and lignin pyrolysis," *Fuel* 86(12-13), 1781-1788. DOI: 10.1016/j.fuel.2006.12.013

Yıldız, S., Gezer, E. D., and Yıdız, Ü. C. (2006). "Mechanical and chemical behavior of spruce wood modified by heat," *Build. Environ.* 41(12), 1762-1766. DOI: 10.1016/j.buildenv.2005.07.017