Effects of Thermomechanical Densification and Heat Treatment on Density and Brinell Hardness of Scots Pine (*Pinus sylvestris* L.) and Eastern Beech (*Fagus orientalis* L.)

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The effects of thermomechanical densification (TMD) and heat treatment on density and Brinell hardness of Scots pine (*Pinus sylvestris* L.) and Eastern beech (*Fagus orientalis* L.) woods were investigated. Samples were densified using a specially designed hydraulic press with target compression ratios of 20 and 40%, and at 110 °C and 150 °C. Then, the heat treatment was applied to the samples at three different temperatures. To determine whether the changes occurred because of technological properties, tests of Brinell hardness and air-dry density were conducted. Increases of 42 and 35% were obtained for the density of Scots pine and beech samples, respectively. After the densification process, increases in radial and tangential hardness values were obtained. Decreases were observed in the density and hardness values of the samples because of the increase in temperature during heat treatment. After heat treatment, there were 4 and 5% decreases in the respective densities of Scots pine and beech, and decreases in their radial and tangential hardness values.

Keywords: Densification; Heat treatment; Scots pine; Eastern beech; Density; Brinell hardness

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INTRODUCTION

Wooden materials have been used throughout history in various ways to meet human needs. At present, wood, as an industrial product, has many uses in parallel with technological developments. The increase in human population and new application areas of wooden material has caused a heightened demand, which has increased the need for high-quality wood. This situation necessitates a more efficient use of existing resources, the modification of wood species of low resistance, and their use in this sector, as well as the production of different materials (Pelit 2014).

The resistivity, hardness, hydrophobicity, and dimensional stability of wooden materials can be increased by physical and chemical processes. These processes include treatment with water-based polymers or synthetic resins that do not dissolve after hardening, binding of cell wall polymers using organic chemicals or cross-link materials, polymerization of liquid monomers in wood cell lumens, densification of wooden materials by compressing or resin saturating, and heat treatment (Rowell and Konkol 1987). The density of wooden materials is an important factor that influences their other properties and potential uses. For example, the resistance, flexibility, and hardness of hardwoods are higher than those of softwoods. Additionally, hardwoods resist abrasives better (Örs and

Keskin 2008). A high density wooden material is necessary for applications where structural integrity and abrasion resistance are important. Wood types with lower densities are not attractive in terms of trade; however, they can be transformed into valuable, high performance products through modification with a densification process. Even the hardness and resistance properties of woods can be further improved by applying densification (Blomberg and Person 2004; Blomberg *et al.* 2005; Kutnar and Sernek 2007; Pelit 2014). Wooden materials can be densified using compression under pressure, impregnation of some chemicals into the cell walls, or the combination of compression and impregnation together (Rowell and Konkol 1987; Kutnar and Sernek 2007). In densification using chemicals, natural and artificial aqueous resins are saturated into the spaces within the wooden material and allowed to harden as a result of the chemical reaction or the cooling process. In this way, wooden materials with a higher density are obtained (Kamke 2006). On the other hand, in densification by compression, the space volume of the wooden material decreases and densification is realized by cell wall collapse (Kutnar *et al.* 2009). An important disadvantage of densification by the compressing method is that the wood resumes its initial dimensions before compressing when soaked in water or exposed to high relative humidity (Seborg *et al.* 1956; Kollmann *et al.* 1975; Kultikova 1999; Morsing 2000; Blomberg *et al*. 2006; Pelit 2014; Pelit *et al.* 2014). The usage of modification processes for wooden materials that involve the application of both heat and pressure are becoming more favorable; such processes are being used to extend the scope of use for various wood materials by enhancing some properties (dimensional stability, biological resistance, *etc*.).

The heat treatment process results in a slight modification in the molecular structure of the wooden material and thus improves its performance. The properties potentially improved by heat treatment are: biological resistance to fungi and insects, low equilibrium moisture content, increased dimensional stability with respect to the decrease in contraction and expansion, increased thermal insulation capacity, and increased resistance to weathering (Wikberg 2004; Enjily and Jones 2006; Korkut and Kocaefe 2009). However, an important disadvantage (based on mass loss and chemical degradation) of this application is the decrease in hardness and resistance properties of the wooden material (Yıldız 2002; Bekhta and Niemz, 2003; Esteves *et al.* 2007; Boonstra 2008; Aydemir and Gündüz 2009; Korkut and Kocaefe 2009; Şahin Kol 2010; Perçin 2012; Pelit 2014). Heat treatment of wood has been investigated for many years without any break in commercialization.

On the European market, several industrial heat treatment processes have been introduced. The most common processes are: the ThermoWood® process, the Plato process, the retification process, le Bois Perdure, and the oil-heat treatment (OHT) process. The total capacity for heat treated wood in Europe is approximately 200,000 m^3/year, and Finland alone has a production of 100,000 m^3/year (Sandberg *et al.* 2013).

It is considered that the decrease in density and resistivity of wooden materials that results from heat treatment can be compensated for by thermomechanical densification (TMD). It is also considered that new materials with improved properties can be produced by the use of these two modification methods in combination. In light of this information, the purpose of this study was to determine the density and the Brinell hardness of the new materials produced by heat treatment (ThermoWood® process) using Scots pine and Eastern beech densified by a thermomechanical method.

EXPERIMENTAL

Materials

Preparation of wooden materials

In this study, Scots pine (*Pinus sylvestris* L.) and Eastern beech (*Fagus orientalis* L.) woods, which are used in the woodworking industry in Turkey, were preferred. The Scots pine trees, from which the test samples were prepared, were obtained from Melet State Forestry Enterprise of the Mesudiye State Forestry Directorate in Ordu Province, Turkey, whereas the Eastern beech trees were obtained from Akkuş State Forestry Enterprise of Akkuş State Forestry Directorate. Round woods, having green moisture content, were cut from their sapwood with an automatically controlled band sawing machine. Cuts were determined by considering sample dimensions as annual rings parallel to the surface (tangent section) and these were transformed into timbers of rough scale. Attention was paid to ensure that no rot, knot, crack, color, or density differences were present in the samples (TS 2470 1976). Samples were initially dried to 12% moisture in an automatically controlled conventional drying furnace, and afterwards they were brought to the dimensions given in Table 1.

Table 1. Dimensions of Samples Before Densification

Target compression ratio (%)	Length - longitudinal direction (mm)	Width - tangential direction (mm)	Thickness - radial direction (mm)
Control	450	95	20
20	450	95	25
40	450	95	33.3

Before the densification process (according to TS 2471), samples were kept on hold in a conditioning cabin until they reached a stable weight with a relative humidity of 65 ± 3%, and temperature of 20 ± 2 °C. To prevent possible moisture changes that could occur after conditioning, samples were preserved in plastic bags until the time of densification (TS 2471 1976).

Densification

Densification of the samples using a thermomechanical densification (TMD) method was performed with a specially designed hydraulic press machine (Fig. 1) which could achieve pressure and temperature control and had pressing tray dimensions of 60 × 60 cm^2. The densification process was done by forming four different variations at target compression ratios of 20 and 40%, with temperatures of 110 ± 5 and 150 ± 5 °C. Densification variations are shown in Table 2.

Table 2. Densification Variations of the Treatments

Research code	Pressing temperature (°C)	Target compression ratio (%)	Duration (min.)
A1	110	20	Heating + 10
A2	110	40	Heating + 10
B1	150	20	Heating + 10
B2	150	40	Heating + 10

The samples were placed onto the bottom tray of the pressing machine and held under slight pressure. Heat transfer was achieved by placing the samples in contact with the heated bottom and top press tray. The samples were kept in this position until their internal temperatures reached the target temperature, by checking with a thermometer. Temperature control samples, which were located separately on the pressing tray, were used for controlling the internal temperature of the samples.

Afterwards, a compression process in the radial direction with automatic control at 60 mm/min loading speed was carried out. To obtain the proposed compression thickness (20 mm), metal stopping sticks were placed onto the pressing tray at particular intervals (Fig. 1). Compressed samples were held under pressure for 10 min. After this period, the samples were taken out from the press machine and cooled to room temperature under a pressure of 5 kg/cm^2, in order to minimize spring-back effects. After densification, the mean moisture amount was 5.2% for the samples densified at 110 °C, and 2.7% for the samples densified at 150 °C.

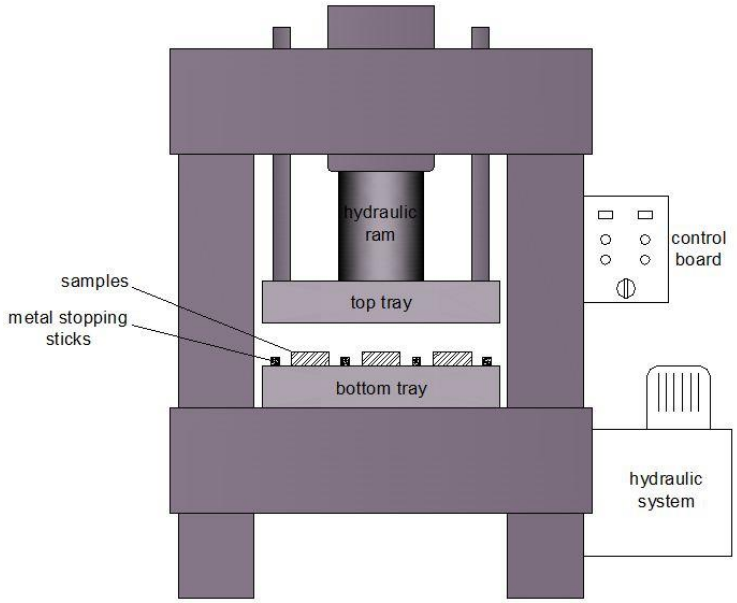

Fig. 1. Schematic diagram of the hot press used for densification of the samples

Heat treatment

Heat treatment, applied to the densified and control (undensified) samples, was carried out in 3 stages (1, drying at elevated temperature; 2, heat treatment; 3, cooling and conditioning) according to the method described in the ThermoWood Handbook (2003). In the first stage, samples were dried to approximately 0% moisture by increasing the furnace temperature with heat and steam. In the second stage, heat, at the proposed temperatures (190, 200, and 210 °C), was applied to the samples for 2 h. In the third stage (conditioning), the temperature was reduced and the moisture ratio of the samples was brought to 4 to 6% by applying water spray.

According to TS 2471 after the heat treatment process, samples remained at a temperature of 20 ± 2 °C and relative humidity of 65 ± 3% until they reached a stable weight (TS 2471 1976). To determine hardness values, samples were cut to the dimensions of 50 × 20 × 20 mm (length-longitudinal direction × width-tangential direction × thickness-radial direction). Furthermore, the test samples were prepared in the number as to ten repetition for each variable. To eliminate possible moisture differences after cutting,

samples remained in a conditioning cabin at 20 ± 2 °C and relative moisture was 65 ± 3% (TS 2471 1976). To prevent moisture changes after conditioning, samples were stored in plastic bags until the measurements were performed.

Methods

Determination of density

Densities were determined based on TS 2472 standards. Samples were stored in the conditioning cabin, with a temperature of 20 ± 2 °C and relative humidity of 65 ± 3%, until they reached a stable weight. The mass of each sample in this condition was measured on an analytical balance, with a sensitivity of (M_{12}) ± 0.01 grams. Dimensions (length, width, thickness) were measured with a vernier caliper having ±0.01 mm sensitivity, and volumes (V_{12}) were determined. The air-dry density (δ_{12}) was calculated according to Eq. 1.

$$\delta_{12} = M_{12} / V_{12} \qquad [g/cm^3] \qquad (1)$$

Determination of Brinell hardness

The TS 2479 standard was used in the determination of the radial and tangential Brinell hardness values. 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 seconds. The load was released in 15 seconds, and the diameter of the indentation made by the steel ball was measured using digital caliper with ± 0.01mm sensitivity and magnifier (Fig. 2). The Brinell hardness values (*HB)* were determined according to Eq. 2,

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

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).

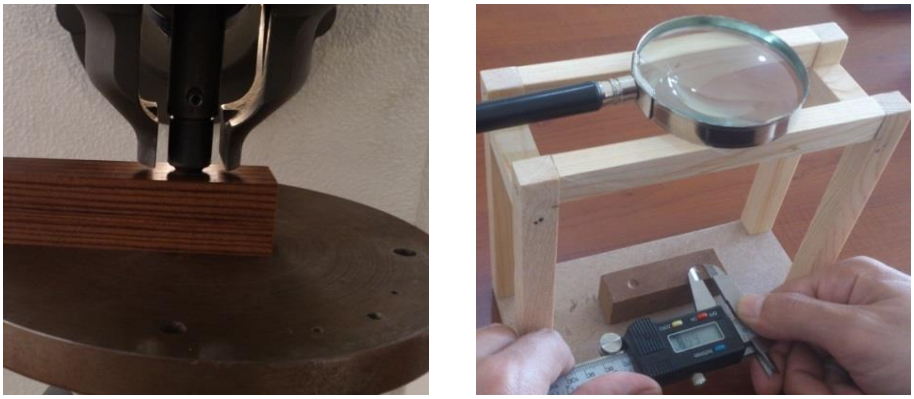

Fig. 2. Brinell hardness test and measurement of the indentation diameter

Statistical analysis

The MSTAT-C package program (Michigan State University, USA) was used for statistical evaluation. Multiple analysis of variance (ANOVA) tests were performed between process groups and control groups, and the differences between the Duncan test results and mean values were compared when significant differences were detected.

Therefore, success ranking among the factors included in the experiment was determined by separating them into homogeneity groups according to Least Significant Difference (LSD) critical values.

RESULTS AND DISCUSSION

Density

Analysis of variance results of air-dry density values from samples thermomechanically densified and heat treated are shown in Table 3.

Table 3. Analysis of Variance Results of Air-dry Density Values

Factors	Degrees of freedom	Sum of squares	Mean square	F-value	Level of significance (P≤0.05)
Wood type (A)	1	1.440	1.440	1785.6934	0.0000*
Densification (B)	4	2.776	0.694	860.4523	0.0000*
Heat treatment (C)	3	0.057	0.019	23.6143	0.0000*
Interaction (AB)	4	0.017	0.004	5.1976	0.0004*
Interaction (AC)	3	0.003	0.001	1.3198	ns**
Interaction (BC)	12	0.013	0.001	1.3280	ns**
Interaction (ABC)	12	0.002	0.000	0.2424	ns**
Error	360	0.290	0.001		
Total	399	4.600			

*Significant at 95% confidence level; **not significant

Table 4. Results of Air-dry Density Values Based on Wood Type, Densification, and Heat Treatment Level

Wood type	$\bar{x}$ (g/cm^3)	*HG*	*LSD*
Scots pine	0.652	B	± 0.0062
Eastern beech	0.772	A*	
Densification	$\bar{x}$ (g/cm^3)	*HG*	*LSD*
Undensified	0.597	E	± 0.0098
A1	0.686	C	
A2	0.824	A*	
B1	0.665	D	
B2	0.789	B	
Heat treatment	$\bar{x}$ (g/cm^3)	*HG*	*LSD*
Untreated	0.730	A*	± 0.0088
190 ºC	0.714	B	
200 ºC	0.707	B	
210 ºC	0.697	C	

A1: 110 °C / 20%; A2: 110 °C / 40%; B1: 150 °C / 20%; B2: 150 °C / 40%; $\bar{x}$: Average value; *HG:* Homogeneous group; *the highest air-dry density value

According to the results of multiple analysis of variance tests, wood type, densification, and heat treatment temperature factors, as well as the dual interaction of wood type-densification were found to have significant effects on the air-dry density values. However,

the dual interactions of wood type with heat treatment temperature and densification with heat treatment temperature, and the triple interaction of wood type with densification and heat treatment temperature were insignificant ($P \leq 0.05$). Mono comparison results of the Duncan Test conducted by using LSD critical values for wood type, densification, and heat treatment level are shown in Table 4.

According to the results of the comparisons, air-dry density values were higher in Eastern beech samples (0.772 g/cm^3) than Scots pine samples (0.652 g/cm^3). The highest air-dry density value (0.824 g/cm^3) was obtained in the samples densified under A2 conditions, and the lowest value (0.597 g/cm^3) was obtained in the undensified samples. Depending on the targeted compression ratios, the density of the samples was found to be highest in samples treated with a higher compression ratio (40%). This increase in density, in comparison to the control (undensified) samples, can be explained by the decrease in the cavity volume of the material and the increase in the amount of cell wall per unit volume. In different studies, it was stated that density increases with increasing compression ratio (Blomberg *et al.* 2005; Ünsal *et al.* 2011; Arruda and Menezzi 2013; Pelit *et al.* 2014).

In response to heat treatment, the highest air-dry density value (0.730 g/cm^3) was obtained in the samples without heat treatment and the lowest value (0.697 g/cm^3) was obtained in the samples for which heat treatment was applied at 210 ºC. The heat treatment process resulted in a decrease in the density of the test material. In addition, the amount of density decrease in the wooden materials increased with an increase in the heat treatment temperature. The decrease in density after heat treatment can be explained by a loss of mass in the wooden material and the decrease in the equilibrium moisture content. In the literature, it was stated that the main reasons for the decrease in wood density after heat treatment were: degradation of wood components (mainly hemicellulose) into volatile products which evaporate during treatment; evaporation of extractives; and a lower equilibrium moisture content of the wooden material, since heat-treated wood is less hygroscopic (Boonstra 2008).

The air-dry density values of Scots pine and Eastern beech are presented comparatively in Figs. 3 and 4.

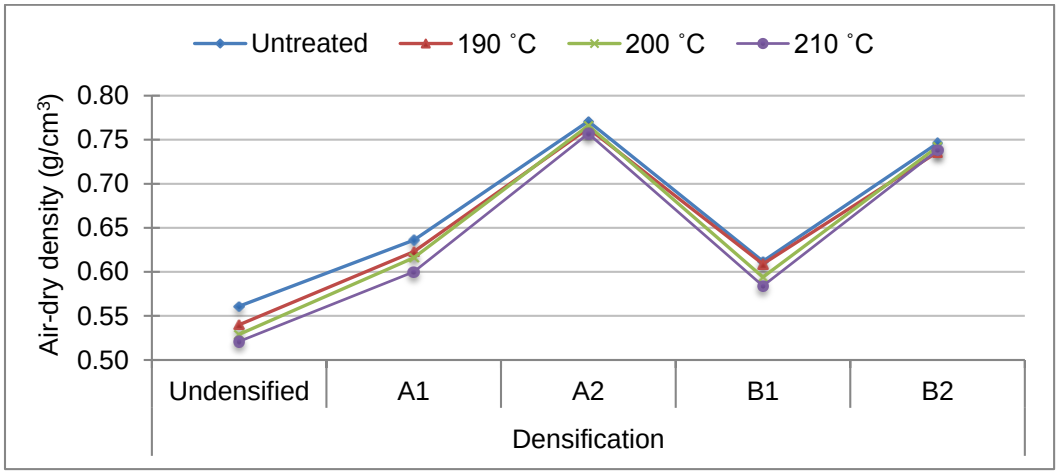

Fig. 3. Comparative appearance of air-dry density values in Scots pine

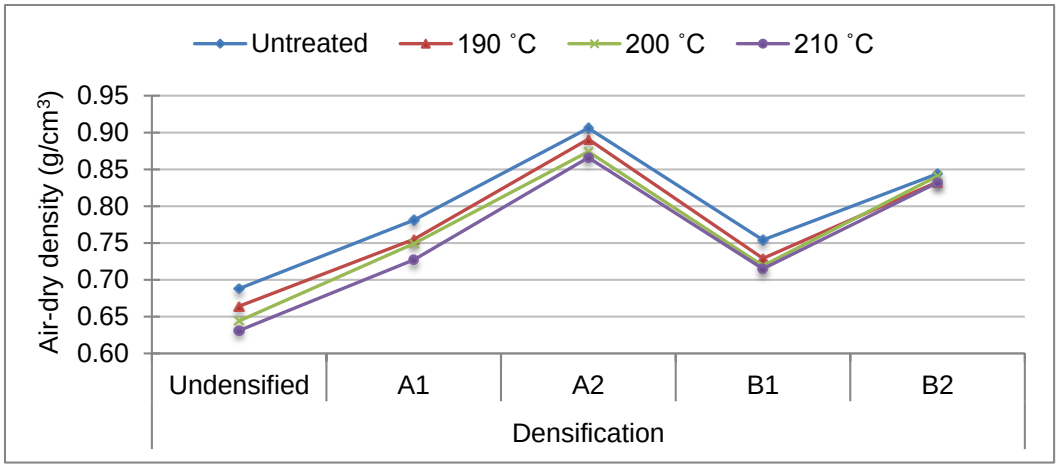

Fig. 4. Comparative appearance of air-dry density values in Eastern beech

Brinell Hardness in the Radial Compression Direction

Analysis of variance results from sample hardness measurements in the radial direction for thermomechanically densified and heat treated are shown in Table 5.

Table 5. Analysis of Variance Results for Radial Hardness

Factors	Degrees of freedom	Sum of squares	Mean square	F-value	Level of significance ($P \leq 0.05$)
Wood type (A)	1	4258.737	4258.737	1052.9356	0.0000*
Densification (B)	4	3835.656	958.914	237.0831	0.0000*
Heat treatment (C)	3	5311.052	1770.351	437.7038	0.0000*
Interaction (AB)	4	162.288	40.572	10.0311	0.0000*
Interaction (AC)	3	311.963	103.988	25.7100	0.0000*
Interaction (BC)	12	205.383	17.115	4.2316	0.0000*
Interaction (ABC)	12	63.712	5.309	1.3127	ns**
Error	360	1456.068	4.045		
Total	399	15604.859			

*Significant at 95% confidence level; **not significant

According to the analysis of variance results, a triple interaction for wood type with densification and heat treatment temperature was found to be not significant for the radial hardness. All other factors and their interactions were found to be significant ($P \leq 0.05$). Mono comparison results of the Duncan test was conducted by using LSD critical value for wood type, densification, and heat treatment level (Table 5).

According to results in Table 6, the radial hardness values obtained were higher in Eastern beech samples (31.93 N/mm^2) than Scots pine samples (25.40 N/mm^2). The fact that measured hardness was higher in the Eastern beech samples can be explained by the higher density and lower density difference between the annual rings. The highest radial hardness (32.44 N/mm^2 and 32.02 N/mm^2) was found in the samples densified under B2 and A2 conditions, whereas the lowest hardness value (24.49 N/mm^2) was found in undensified samples. Radial hardness increased in proportion to the compression ratios; for example, higher values were obtained for a compression ratio of 40%. In other studies, it was reported that the hardness of wooden material was closely related to the densification

ratio, and that hardness values increase with increasing densification ratios (Rautkari *et al.* 2009; Ünsal *et al.* 2011).

Table 6. Duncan Test for the Comparison of Radial Hardness with Wood Type, Densification, and Heat Treatment Level

Wood type	$\bar{x}$ (N/mm^2)	*HG*	*LSD*
Scots pine	25.40	B	± 0.3955
Eastern beech	31.93	A*	
Densification	$\bar{x}$ (N/mm^2)	*HG*	*LSD*
Undensified	24.49	D	± 0.6254
A1	26.64	C	
A2	32.02	A*	
B1	27.75	B	
B2	32.44	A*	
Heat treatment	$\bar{x}$ (N/mm^2)	*HG*	*LSD*
Untreated	34.49	A*	± 0.5594
190 ºC	28.80	B	
200 ºC	26.57	C	
210 ºC	24.82	D	

A1: 110 ˚C / 20%; A2: 110 ˚C / 40%; B1: 150 ˚C / 20%; B2: 150 ˚C / 40%; $\bar{x}$: Average value; *HG:* Homogeneous group; *The highest radial hardness value.

The highest radial hardness value in the heat treatment level was obtained in the samples without heat treatment (34.49 N/mm^2), whereas the lowest radial hardness value (24.82 N/mm^2) was obtained in the samples to which the heat treatment was applied at 210 ºC. After the heat treatment, radial hardness of the samples decreased and the hardness values gradually decreased with an increase in temperature. The decrease in hardness values can be attributed to the loss of mass and density in the wooden materials' components, as a result of thermal decomposition. In the literature, it is reported that the hardness of wooden materials decrease with increases in heat treatment temperature (Yıldız 2002; Korkut *et al.* 2008; Perçin 2012). In another study, it was reported that heat treatment applied after densification has a negative effect on the Brinell hardness values (Fang *et al.* 2012).

The radial hardness values of Scots pine and Eastern beech are presented comparatively in Figs. 5 and 6.

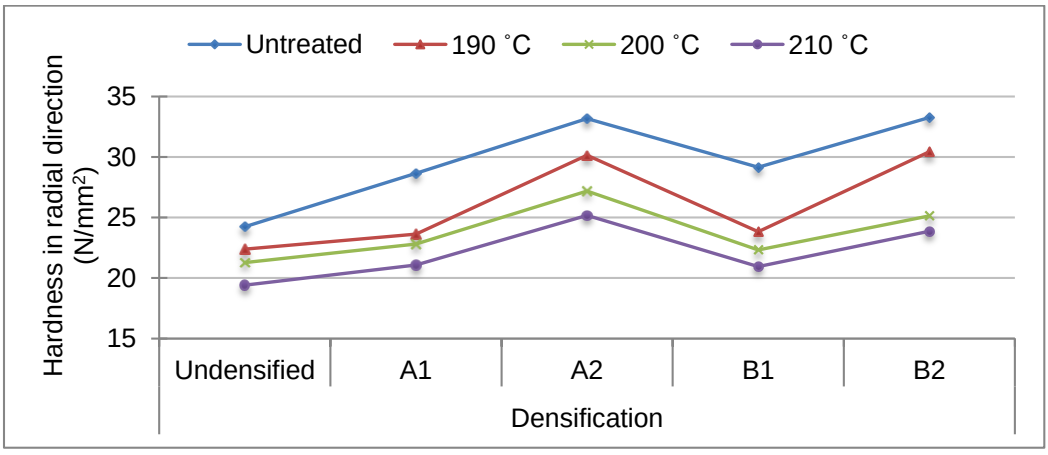

Fig. 5. Comparative appearance of radial hardness values in Scots pine

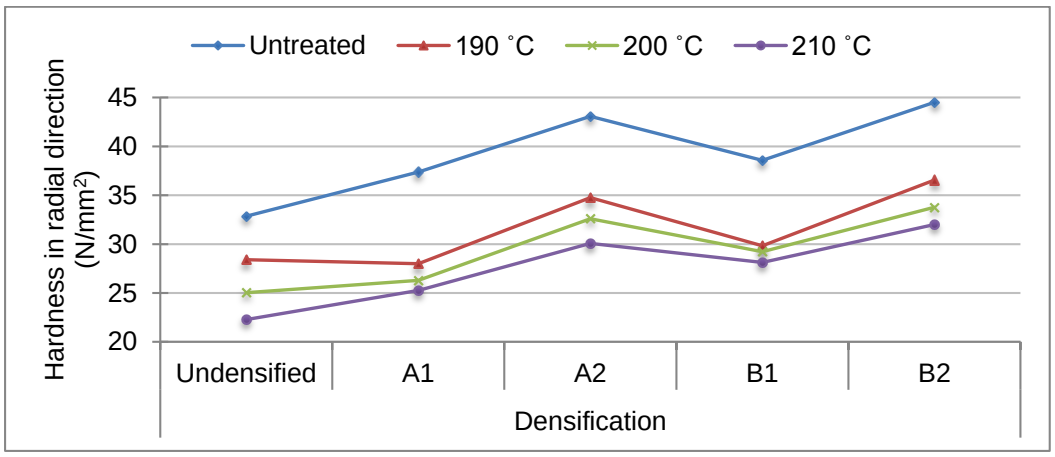

Fig. 6. Comparative appearance of radial hardness values in Eastern beech

Brinell Hardness in the Tangential Direction

Analysis of variance results of hardness values in the tangential direction of samples that were thermomechanically densified and heat treated are shown in Table 7.

Table 7. Analysis of Variance Results for Tangential Hardness

Factors	Degrees of freedom	Sum of squares	Mean square	F-value	Level of significance ($P \leq 0.05$)
Wood type (A)	1	8159.870	8159.870	1837.5990	0.0000*
Densification (B)	4	15354.022	3838.506	864.4296	0.0000*
Heat treatment (C)	3	8325.630	2775.210	624.9760	0.0000*
Interaction (AB)	4	647.924	161.981	36.4781	0.0000*
Interaction (AC)	3	872.378	290.793	65.4864	0.0000*
Interaction (BC)	12	510.621	42.552	9.5826	0.0000*
Interaction (ABC)	12	162.487	13.541	3.0493	0.0004*
Error	360	1598.582	4.441		
Total	399				

*Significant at 95% confidence level

The analysis of variance results showed that wood type, densification, heat treatment factors, and their reciprocal interactions on hardness values in the tangential direction were significant ($P \leq 0.05$). Mono comparison results using the Duncan test and LSD critical values for wood type, densification, and heat treatment level, are shown in Table 8.

According to the results, the tangential hardness values obtained were higher in Eastern beech samples (36.30 N/mm^2) than in Scots pine samples (27.27 N/mm^2). The highest hardness values (38.80 N/mm^2 and 38.64 N/mm^2) were found in the samples densified under B2 and A2 conditions. The lowest value (22.56 N/mm^2) was found in undensified samples. The tangential Brinell hardness values increased in proportion to the compression ratios; for example, higher values were obtained at higher compression ratios (40%). Density and strength increases resulting from the compression of wooden material largely depend on the level of compression with the densification method used and the properties of the wood types (Rautkari 2012).

Table 8. Duncan Test for the Comparison of Tangential Hardness with Wood Type, Densification, and Heat Treatment Level

Wood type	$\bar{x}$ (N/mm^2)	*HG*	*LSD*
Scots pine	27.27	B	± 0.4144
Eastern beech	36.30	A*	
Densification	$\bar{x}$ (N/mm^2)	*HG*	*LSD*
Undensified	22.56	C	± 0.6553
A1	29.49	B	
A2	38.64	A*	
B1	29.44	B	
B2	38.80	A*	
Heat treatment	$\bar{x}$ (N/mm^2)	*HG*	*LSD*
Untreated	39.32	A*	± 0.5861
190 ºC	31.34	B	
200 ºC	29.06	C	
210 ºC	27.43	D	

A1: 110 ˚C / 20%; A2: 110 ˚C / 40%; B1: 150 ˚C / 20%; B2: 150 ˚C / 40%; $\bar{x}$: Average value; *HG:* Homogeneous group; *the highest hardness value.

The highest compression ratio recovery value (39.32 N/mm^2) was obtained in the samples without heat treatment, whereas the lowest value (27.43 N/mm^2) was obtained in the samples in which heat treatment was applied at 210 ºC. Similar to the radial hardness, the tangential hardness of the samples decreased, and the hardness values gradually decreased with an increase in temperature. According to the results of studies on heat treatment using different temperatures and durations, technological properties of wooden materials are impaired by increasing heat treatment temperatures and durations (Korkut *et al.* 2008).

The tangential hardness values for Scots pine and Eastern beech are presented comparatively in Figs. 7 and 8.

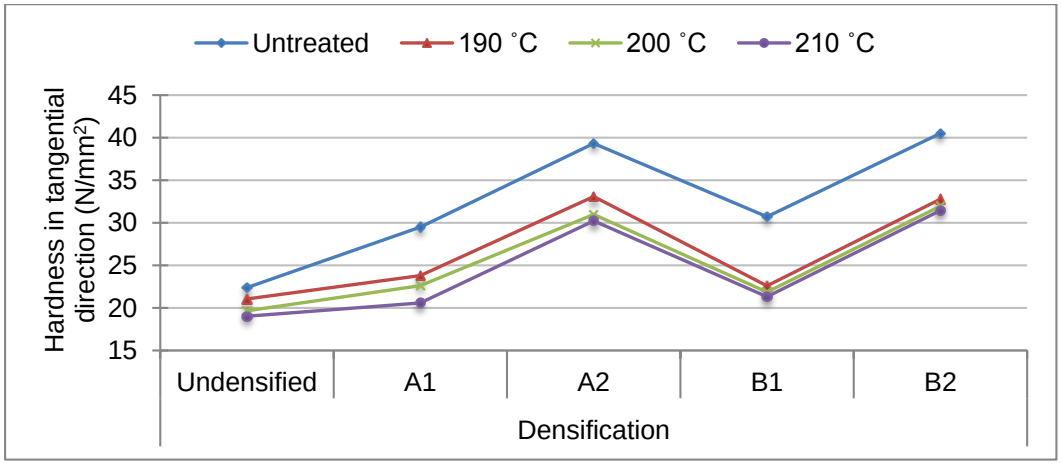

Fig. 7. Comparative appearance of tangential hardness values in Scots pine

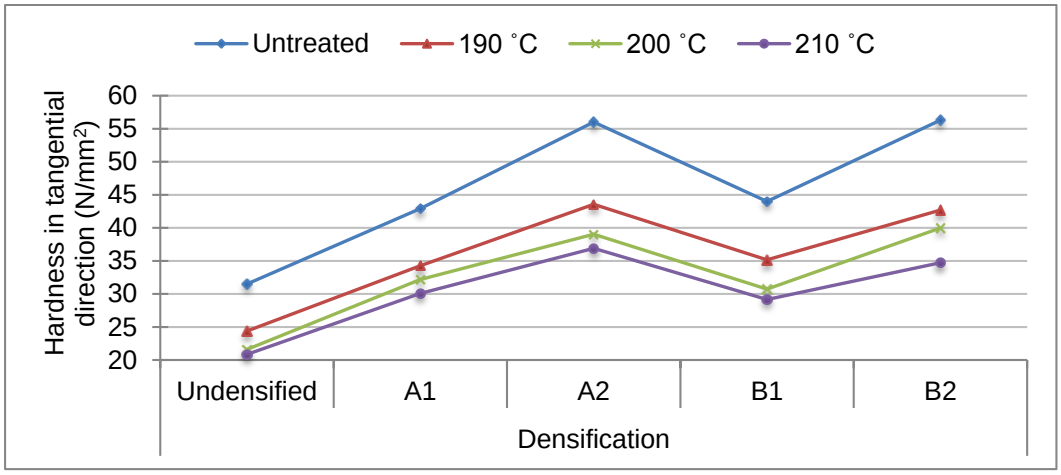

Fig. 8. Comparative appearance of tangential hardness values in Eastern beech

CONCLUSIONS

1. In this study, the effects of heat treatment applied using the ThermoWood® process on air-dry density and Brinell hardness of Scots pine and Eastern beech woods thermomechanically densified at different temperatures and compression ratios were investigated. After densification, an increase in density of less than or equal to 42% was observed in Scots pine samples in proportion to the compression ratios, and 35% in Eastern beech samples. After heat treatment, a decrease in density of approximately 4% in Scots pine and 5% in Eastern beech was observed in comparison with the control samples. Furthermore, at the same compression ratio (20 or 40%), higher density increases were obtained in the samples densified at 110 ºC with respect to the ones densified at 150 ºC.
2. After densification, there was an increase in the Brinell hardness values of Scots pine and Eastern beech samples with respect to the compression ratios. Higher increases in Brinell hardness values were obtained at a compression ratio of 40%. The effect of densification temperature on hardness was not significant. After densification, radial hardness values increased by 32% in Scots pine and 35% in Eastern beech samples, respectively. Significant increases of 66 and 78% were observed in tangential hardness values in Scots pine and Eastern beech samples, respectively.
3. A decrease was observed in the Brinell hardness values due to the heat treatment and an increase in temperature. After heat treatment, the radial hardness values of Scots pine and Eastern beech samples decreased from 12 to 26% and 20 to 30%, respectively, and their tangential hardness values decreased from 18 to 25% and 22 to 34%, respectively.
4. It was observed that the loss of density and resistivity in wooden materials is due to heat treatment and can be eliminated by TMD. Additionally, new materials with improved properties can be produced by the combination of these two modification methods.

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Article submitted: September 29, 2014; Peer review completed: March 28, 2015; Revisions accepted: April 3, 2015; Published: April 6, 2015.
DOI: 10.15376/biores.10.2.3097-3111