Effects of ThermoWood® Process Combined with Thermo-Mechanical Densification on some Physical Properties of Scots Pine (*Pinus sylvestris* L.)

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Effects of heat treatment on some physical properties of Scots pine (*Pinus sylvestris* L.) wood densified using a thermo-mechanical method were determined. Samples were densified in the radial direction with a specially designed hydraulic press machine with target compression ratios of 20% and 40%, and at 110 °C and 150 °C. Then, heat treatment was applied to the samples during 2 h at three different temperatures (190 °C, 200 °C, and 210 °C). In order to determine the changes occurring in physical properties, tests of actual compression ratio, spring-back, compression ratio recovery effect, swelling (TS 4084) in compression direction (radial), and density (TS 2472) were conducted. According to results of the research, at the same target compression ratio (20% or 40%), higher actual compression ratio and density increase were observed in the samples densified at 110 ºC in comparison to those densified at 150 ºC. While an increase of 42% in density was being obtained, small rates of decreases up to 4% were observed after heat treatment. Application of heat treatment and increase of treatment temperature significantly influenced dimensional stability of densified Scots pine. Furthermore in comparison to samples without heat treatment, effects of compression ratio recovery were reduced by 80%.

Keywords: Scots pine; Densification; Heat treatment; Physical properties

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INTRODUCTION

Most of the mechanical and physical properties of a wooden material are related to its density (Blomberg and Person 2004; Kamke 2006; Rautkari *et al.* 2010; Sandberg *et al.* 2013). A high density of a wooden material is necessary for applications in which structural applications and abrasion resistance are important. Wood types with low density and which are not attractive in terms of trade can be transformed into valuable and high performance products by modification with a densification process. Even the hardness and resistance properties of woods with high density can be further improved by applying densification (Blomberg and Person 2004; Blomberg *et al.* 2005; Kutnar and Sernek 2007). Wooden material can be densified by compressing under pressure with impregnation of some chemicals into cell wall or by combinations of compression and impregnation together. In densification by compressing, the natural elastic structure of the wood plays an important role, and compression properties mostly depend on the density, moisture, compression ratio, and direction of the wood (Rowell and Konkol 1987; Kutnar and Sernek 2007). Density and mechanical resistance properties can be improved by compressing wooden

material in the transverse direction. However, an important disadvantage of this approach is that the material reverts to its original dimensions before compression when the densified wood is soaked in water or exposed to high relative humidity. This situation is caused by the extension in cell wall, relaxation of internal stresses formed in material structure as a result of compression, and, in particular, the cell recovering to its original form (Seborg *et al*. 1956; Kollmann *et al.* 1975; Kultikova 1999; Morsing 2000; Blomberg *et al.* 2006). Usage of modification processes for wooden material involving application of both heat and pressure is increasing with the passage of time; such processes are being used to extend the usage fields of various wood materials by enhancing some properties (dimensional stability, biological resistance, *etc*.).

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

Heat treatment leads to permanent changes in molecular structure of the chemical compounds of wood. The fundamental idea underlying this application is to treat wooden material with heat above the temperatures of 150 °C where chemical reactions become accelerated (Cooper and Wang 2005; Boonstra 2008). Biological resistance and dimensional stability of the wooden material subjected to heat treatment increase and its color can be altered. However, an important disadvantage of this application is the decrease in mechanical resistance properties of the wood material (Yıldız 2002; Bekhta and Niemz 2003; Boonstra *et al.* 2006; Esteves *et al.* 2007; Boonstra 2008; Aydemir and Gündüz 2009; Korkut and Kocaefe 2009; Şahin Kol 2010; Perçin 2012). It is thought that some significant disadvantages of both methods can be eliminated by using the combination of thermo-mechanical densification and heat treatment (ThermoWood® process) methods. In view of the issues just mentioned, the purpose of this study is to determine some physical properties of the modified material (*Densified-ThermoWood*®) obtained by applying heat treatment to the Scots pine wood which had been densified with a thermo-mechanical process.

EXPERIMENTAL

Materials

Preparation of wood material

In this study, Scots pine (*Pinus sylvestris* L.) wood having relatively low density and which has been widely used in the woodworking industry in Turkey was employed. Trees that were used to prepare samples were obtained from the area of Melet Office of Mesudiye Forest Management in the city of Ordu in Turkey. Round woods having green moisture content were cut from their sapwood following TS 2470 standards with an automatically controlled band sawing machine by considering sample dimensions as annual rings to be parallel to the surface (tangent section). These were transformed into timbers of rough scale. Attention was paid to ensure that no rotten, 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. Before Densification Dimensions of Samples

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 until they reached stable weight in a conditioning cabin with a relative moisture of 65 ± 3% and temperature of 20 ± 2 °C. To prevent possible moisture changes that can occur after conditioning, samples were preserved in plastic bags until the densification moment (TS 2471 1976).

Densification

Densification of the samples with the thermo-mechanical (TM) method was performed with a specially designed hydraulic press machine of 100 tons capacity which can achieve pressure and temperature control and whose pressing tray dimensions are 60 × 60 cm^2. 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 given in Table 2.

Table 2. Densification Variations

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 a slight pressure by getting them in contact with the heated bottom and top press tray to provide heat transfer. The samples were kept in this position for a while until their internal temperature reached the target temperature, by checking with a thermometer. Temperature control samples, which were separately located on the pressing tray, were used for controlling internal temperature of the samples.

Afterwards, a compression process in radial direction with automatic control at 60 mm/min loading speed was carried out. To obtain 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, and after this period these 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, 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. 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 furnace temperature with using heat and steam. In the second stage, heat treatment at the proposed temperatures (190, 200, and 210 °C) was applied to the samples during 2 h. In the third stage (conditioning), the temperature was reduced and moisture ratio of the samples were provided to reach 4 to 6% by applying water spray.

According to TS 2471 after heat treatment, the samples were kept on hold at a temperature of 20 ± 2 °C and relative humidity of 65 ± 3% until they reached a stable weight (TS 2471 1976). To determine some physical properties, samples were cut as to have dimensions (thickness-radial direction remains constant) of 30 × 20 mm (length-longitudinal direction × width-tangential direction) and as to be repetitive for 10 times for each test variant. To be able to eliminate possible moisture differences after cutting, due to TS 2471, samples were again kept on hold 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 kept in plastic bags until the measurement was performed.

Methods

Determination of actual compression ratio, spring-back and compression ratio recovery

Samples were pressed at two different targeted ratios (20% and 40%). However, after the press machine pressure disappears, instantaneous spring-back, which is caused by the release of internal stresses, takes place. Additionally, moisture losses that occurred in the samples by the influence of temperature in compression caused a separate spring-back after the samples had been conditioned at 20±2 ˚C and at relative humidity of %65±3. It was found that variations (decreases) in the compression ratios targeted at pressing stage took place. The actual compression ratio (C_R), which forms as a result of these variations, was determined using Eq. 1, and spring-back (S_B) ratios were determined using Eq. 2. Furthermore, compression ratio recovery (C_{RR}) values of the Scots pine samples soaked to water at 20 ± 2 °C for 672 h, densified and heat treated, were determined with Eq. 3,

$$C_R = [(T_1 - T_3) / T_1] \times 100 \quad [\%] \qquad (1)$$

$$S_B = [(T_3 - T_2) / T_2] \times 100 \quad [\%] \qquad (2)$$

$$C_{RR} = [(T_4 - T_3) / (T_1 - T_3)] \times 100 \qquad [\%] \qquad (3)$$

where T_1 is the thickness of samples conditioned at 20 ± 2 °C and 65 ± 3% RH before compression, T_2 is the thickness of samples under pressure (load), T_3 is the thickness of samples conditioned at 20 ± 2 °C and 65 ± 3% RH for three weeks, and T_4 is the thickness of samples after soaking in water (Navi and Girardet 2000; Welzbacher *et al.* 2008; Dubey 2010). Thicknesses were determined with a vernier caliper of ± 0.01 mm sensitivity.

Determination of swelling in compression direction (radial)

Compression direction (radial) swelling ratios were determined according to TS 4084 standards. Samples were kept at 103 ± 2 °C in a drying furnace until they reached stable dimensions, and weight and thicknesses at this condition were found to be in (L_0) ± 0.01 mm sensitivity. Then, the samples were sunk into clean water and kept there until their dimensions in thickness direction became stable. Thicknesses at this condition (L_R) were again measured from the first measurement point, and maximum swelling (α_k) in compression (radial) direction was calculated based on Eq. 4.

$$\alpha_k = [(L_R - L_0) / L_0] \times 100 \qquad [\%] \qquad (4)$$

Determination of density

Densities were determined based on TS 2472 standards. Samples were kept in the conditioning cabin having a temperature of 20 ± 2 ºC and relative humidity of 65 ± 3% until they reached stable weight. Masses of the samples of this condition were measured on an analytical balance whose sensitivity was (M_{12}) ± 0.01 g, dimensions (length, width, thickness) were measured with a vernier caliper having ±0.01 mm sensitivity, and volumes (V_{12}) were determined. Air-dry density (δ_{12}) was calculated according to Eq. 5.

$$\delta_{12} = M_{12} / V_{12} \qquad [g/cm^3] \qquad (5)$$

Statistical analysis

The MSTAT-C package program was used in statistical evaluations. Multiple analysis of variance (ANOVA) was performed between process groups and control groups, and differences between Duncan test results and mean values were compared when significant differences were detected within obtained data. Therefore, success ranking among the factors included into the experiment was determined by separating them into homogeneity groups according to Least Significant Difference (LSD) critical values.

RESULTS AND DISCUSSION

Actual Compression Ratio

Actual compression ratios of Scots pine samples densified in different conditions, at air-dry moisture after densification are given in Table 3. The highest actual compression ratio (32.73%) was obtained for the samples densified under A2 conditions; the lowest value (15.49%) was obtained for the samples under B1 conditions. It was seen that actual compression ratios obtained after densification were different (20% and 40%) than the target compression ratios. Actual compression ratios being lower than the targeted ratios can be caused by the internal stresses formed within cell structure of the material at

compression stage, and moisture losses. Both factors result in some amount of spring-back in the samples. In the literature, it was stated that there are diversifications (decreases) in target compression ratios after densification because of the elastic behavior of wooden material (Welzbacher *et al.* 2008; Rautkari *et al.* 2011). In another study, due to elastic behavior of compressed wooden material, it has been emphasized that the material has tendency to regain its original shape after compression forces are released. Such behavior is defined as spring-back and results in changes in compressed dimension (Garcia-Romeu *et al.* 2007). Higher actual compression ratios were obtained in the samples that were densified at the same target compression ratios (20% or 40%) and at lower temperature (110 ºC). It can be said that this situation arises from the fact that spring-back values of the samples densified at 110 ºC are lower than those densified at 150 ºC.

Table 3. Mean Thicknesses and Actual Compression Ratio Results of Scots Pine Samples Before and After Densification

Densification	Thickness before densification (mm)	Target thickness (mm)	Thickness after densification (mm)	Actual compression ratio (%)
A1	25	20	20.89 (0.05)*	16.44 (0.21)*
A2	33.3	20	22.42 (0.32)*	32.73 (0.97)*
B1	25	20	21.13 (0.33)*	15.49 (1.31)*
B2	33.3	20	23.00 (0.67)*	31.01 (2.02)*

A1: 110 ˚C / 20%; A2: 110 ˚C / 40%; B1: 150 ˚C / 20%; B2: 150 ˚C / 40% *: Standard deviation

Spring-back

Analysis of variance results of spring-back values of Scots pine samples thermo-mechanically densified and heat treated are given in Table 4.

Table 4. Analysis of Variance Results of Spring-back Values

Factors	Degrees of freedom	Sum of squares	Mean square	F-value	Level of significance (P≤0.05)
Densification (A)	3	1522.050	507.350	344.1213	0.0000*
Heat treatment (B)	3	926.578	308.859	209.4906	0.0000*
Interaction (AB)	9	160.163	17.796	12.0704	0.0000*
Error	144	212.304	1.474		
Total	159	2821.095			

*: Significant at 95% confidence level

According to analysis of variance results, densification and heat treatment factors on spring-back values of Scots pine samples and their reciprocal interactions were found to be significant (P≤0.05). Mono comparison results of Duncan test, which was conducted by using LSD critical value at densification and heat treatment level, are given in Table 5.

Table 5. Comparison Results of Duncan Test Related to Spring-back Values at Densification and Heat Treatment Level

Densification	$\bar{x}$ (%)	*HG*	*LSD*
A1	2.286	D	± 0.5366
A2	7.926	B	
B1	3.345	C	
B2	9.693	A*	
Heat treatment	$\bar{x}$ (%)	*HG*	
Untreated	9.297	A*	
190 ºC	6.715	B	
200 ºC	4.158	C	
210 ºC	3.080	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 spring-back value

According to results shown in Table 5, the highest spring-back value (9.693%) at densification level was found in the samples densified under B2 conditions, whereas the lowest value (2.286%) was found in the samples densified under A1 conditions. Proportional to target compression ratios, the spring-back value at a high target compression ratio (40%) was found to be higher. This situation may arise from the internal stresses that formed within the wooden material, a phenomenon that was more pronounced at high target compression ratio. In the literature, it has been stated that the amount of stress that is formed within material structure shows a rapid increase as a result of reduced or eliminated void volumes during densification (Wolcott *et al.* 1989; Nairn 2006; Kutnar and Sernek 2007). Moreover, it was stated that more spring-back forms at higher compression ratios and this situation is caused by the stresses that are formed more within material structure (Laine *et al.* 2013). Higher spring-back value was obtained in the samples which were densified at the same target compression ratio (20% or 40%) and higher temperature (150 ºC). The fact that moisture losses occurred by the influence of temperature during densification period were found to be higher in the samples densified at 150 ºC can be influential on the results. This is because it was detested that increase of thickness in these samples after conditioning (20 ± 2 °C / 65 ± 3% RH) was found to be greater. In the literature, it is stated that when spring-back occurs in the thermally compressed wooden panels, this situation is caused by natural thermoplastic structure of the lignin and decreased amount of moisture in wooden panels by the effect of temperature during compression (Kollmann *et al.* 1975).

The highest spring-back value in heat treatment level was obtained in the samples without heat treatment (9.297%), whereas the lowest value (3.080%) was obtained in the samples to which heat treatment was applied at 210 ºC. Progressive decreases in spring-back value were encountered with application of heat treatment and increasing heat treatment temperature. In the literature, it has been stated that heat treatment applied after densification considerably reduces spring-back property and hygroscopicity, and both higher heat treatment temperature and longer application durations give better results in dimensional stability (Cai *et al.* 2012). Moreover, it has been stated that heat treatment applied to the densified wooden material causes degradation of susceptible components such as hemicellulose in wooden material polymers and accordingly internal stresses formed in the structure can be eliminated by relaxation during densification process, and

the decrease in spring-back ratios can be explained with this reason (Dwianto *et al.* 1997; Dubey 2010).

Table 6. Comparison Results of Duncan Test Related to Spring-back Values at Densification-Heat Treatment Dual Interaction Level

Densification	Heat treatment							
	Untreated		190 ºC		200 ºC		210 ºC	
	$\bar{x}$ (%)	*HG*	$\bar{x}$ (%)	*HG*	$\bar{x}$ (%)	*HG*	$\bar{x}$ (%)	*HG*
A1	4.450	FG	2.519	H	1.150	I	1.025	I
A2	12.120	B	8.925	C	6.320	DE	4.340	FG
B1	5.639	E	4.030	G	2.070	HI	1.640	HI
B2	14.981	A*	11.385	B	7.090	D	5.315	EF
LSD ± 1.073								

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 spring-back value

According to a comparison of the results (Table 6), the highest spring-back value (14.981%) was obtained in specimens without heat treatment that were densified under B2 conditions, and the lowest value (1.150 and 1.025%) was obtained in the specimens for which heat treatment was applied at 200 and 210 ºC and they were densified under A1 conditions.

Compression Ratio Recovery

Analysis of variance results of compression ratio recovery values of Scots pine samples that were thermo-mechanically densified followed by application of heat treatment are given in Table 7.

Table 7. Analysis of Variance Results of Compression Ratio Recovery Values

Factors	Degrees of freedom	Sum of squares	Mean square	F-value	Level of significance (P≤0.05)
Densification (A)	3	3312.348	1104.116	87.2631	0.0000*
Heat treatment (B)	3	152300.172	50766.724	4012.3140	0.0000*
Interaction (AB)	9	2599.092	288.788	22.8242	0.0000*
Error	144	1821.993	12.653		
Total	159	160033.605			

*: Significant at 95% confidence level

According to analysis of variance results, (Table 7) densification, heat treatment factors, and their reciprocal interactions on compression ratio recovery values of Scots pine samples were found to be significant (P≤0.05).

Mono comparison results of Duncan test, which was conducted by using the LSD critical value at densification and heat treatment level, are given in Table 8. According to the results of comparative tests, the highest compression ratio recovery value (50.14%) at densification level was found in the samples densified under B1 conditions, whereas the lowest value (38.50%) was found in the samples densified under B2 conditions.

Table 8. Comparison Results of Duncan Test Related to Compression Ratio Recovery Values at Densification and Heat Treatment Level

Densification	$\bar{x}$ (%)	*HG*	*LSD*
A1	47.81	B	± 1.572
A2	42.43	C	
B1	50.14	A*	
B2	38.50	D	
Heat treatment	$\bar{x}$ (%)	*HG*	
Untreated	96.97	A*	
190 ºC	36.83	B	
200 ºC	26.49	C	
210 ºC	18.59	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 compression ratio recovery value

In the samples where actual compression ratio after densification was found to be low, compression ratio recovery values were found to be higher. This situation possibly can be caused by more fractures occurring, more cellular collapse, and more closure of lumens in the structures of samples having high compression ratio. Furthermore, it can be explained as compressed samples exhibiting the tendency of recovering to their initial dimensions before densification can reach this target more easily at a low compression ratio. In the literature it has been stated that collapses start with fracture or bending of cell walls in densification; deformation formed under low stress conditions has linear and elastic characteristics. Deformation increases more under the stresses above the fracture point, and collapsed cell walls make intimate contacts with each other at this stage (Ahmed *et al.* 2013). Moreover, it was stated that the set-recovery effect takes place as a result of relaxation of internal stresses formed in material structure during compression in case of compressed wooden material is subjected to water or moisture (Morsing 2000).

The highest compression ratio recovery value (96.97%), in heat treatment level was obtained in the samples without heat treatment, whereas the lowest value (18.59%) was obtained in the samples which heat treatment applied to at 210 ºC. The compression ratios in the samples without heat treatment after soaking in water were almost fully lost. Progressive decreases in compression ratio recovery value were obtained with heat treatment application and increase of heat treatment temperature. This situation can be explained with the relaxation by heat treatment of internal stresses formed in cell wall polymers of wooden material in the densification process and destruction of crosslinks that are responsible for shape memory effect as a result of heat treatment (Navi and Heger 2004; Inoue *et al.* 2008; Dubey 2010; Laine *et al.* 2013).

Comparison results of Duncan Test conducted by using LSD critical value at densification-heat treatment dual interaction level are given in Table 9. According to the tabulated results, the highest compression ratio recovery value (110.64%) was obtained in samples without heat treatment that were densified under A1 conditions, while the lowest value (15.26%) was obtained in the samples for which heat treatment was applied at 210 ºC and densified under B2 conditions.

Swelling In Compression Direction (Radial)

Analysis of variance results of compression direction (radial) swelling values of heat-treated Scots pine samples thermo-mechanically densified are given in Table 10.

Table 9. Comparative Results of Duncan Test Related to Compression Ratio Recovery Values at Densification-Heat Treatment Dual Interaction Level

Densification	Heat treatment							
	Untreated		190 ºC		200 ºC		210 ºC	
	$\bar{x}$ (%)	*HG*	$\bar{x}$ (%)	*HG*	$\bar{x}$ (%)	*HG*	$\bar{x}$ (%)	*HG*
A1	110.64	A*	35.21	FG	27.84	IJ	17.54	MN
A2	88.88	C	35.67	F	26.39	J	18.80	LM
B1	103.67	B	44.04	E	30.09	HI	22.77	K
B2	84.71	D	32.40	GH	21.65	KL	15.26	N
LSD ± 3,144								

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 compression ratio recovery value

Table 10. Analysis of Variance Results of Compression Direction Swelling Value

Factors	Degrees of freedom	Sum of squares	Mean square	F-value	Level of significance (P≤0.05)
Densification (A)	4	9858.184	2464.546	1300.9094	0.0000*
Heat treatment (B)	3	11948.175	3982.725	2102.2794	0.0000*
Interaction (AB)	12	4156.699	346.392	182.8426	0.0000*
Error	180	341.006	1.894		
Total	199	26304.065			

*: Significant at 95% confidence level

According to analysis of variance results; densification, heat treatment factors, and their reciprocal interactions were found to have significant effect on swelling values of Scots pine samples (P≤0.05) (Table 10).

Mono comparison results of Duncan tests conducted by using LSD critical value at densification and heat treatment level are given in Table 11. The highest compression direction (radial) swelling value (23.710%) at densification level was obtained in the samples densified under A2 conditions, and the lowest value (3.884%) was obtained in the samples which were undensified (Table 11). Proportional to the target compression ratios, greater swelling ratio was obtained at high target compression ratio (40%). It can be said that this situation is caused by the wooden materials having the tendency of recovering to their initial dimensions before compression. Furthermore, results showed parallelism with the actual compression ratios at after densification; swelling value was found to be higher in the samples where high actual compression ratio values had been obtained. In different studies it is stated that compression ratio remarkably influences dimensional stability of samples, and samples with higher compression ratios tend to swell more (Ünsal *et al.* 2011; Cai *et al.* 2012).

Table 11. Comparative Results of Duncan Test Related to Compression Direction Swelling Values at Densification and Heat Treatment Level

Densification	$\bar{x}$ (%)	*HG*	*LSD*
Undensified	3.884	D	± 0.6072
A1	11.899	C	
A2	23.710	A*	
B1	11.882	C	
B2	20.450	B	
Heat treatment	$\bar{x}$ (%)	*HG*	*LSD*
Untreated	27.396	A*	± 0.5431
190 ºC	12.677	B	
200 ºC	9.685	C	
210 ºC	7.702	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 compression direction (radial) swelling value

The highest compression direction (radial) swelling value (27.396%) at heat treatment level were obtained in the samples without heat treatment, whereas the lowest value (7.702%) was obtained in the samples which heat treatment was applied at 210 ºC. Progressive decreases were encountered with heat treatment application and increasing heat treatment temperature. In the literature it is stated that several permanent changes take place in chemical and physical structure of wooden material with heat treatment. The main cause of these changes are thermal degradation of hemicelluloses; such degradation provides dimensional enhancement in heat treated wood with respect to normal wood because of the reduction of hydroxyl groups which keeps water within the wood (ThermoWood Handbook 2003). It was noted that dimensional stability of the wooden material was developed as a result of the decrease in hygroscopicity of wooden material after heat treatment; furthermore it was also stated that esterification of hydroxyl groups and cross-link reactions are influential in the decrease of hygroscopicity (Tjeerdsma and Militz 2005).

Comparative results of the Duncan test conducted by using the LSD critical value at densification-heat treatment dual interaction level are given in Table 12.

Table 12. Comparative Results of Duncan Test Related to Compression Direction Swelling Values at Densification-Heat Treatment Dual Interaction Level

Densification	Heat treatment							
	Untreated		190 ºC		200 ºC		210 ºC	
	$\bar{x}$ (%)	*HG*	$\bar{x}$ (%)	*HG*	$\bar{x}$ (%)	*HG*	$\bar{x}$ (%)	*HG*
Undensified	4.837	LM	4.113	MN	3.411	N	3.174	N
A1	24.103	C	9.661	I	7.933	J	5.897	KL
A2	46.034	A*	20.717	D	15.542	F	12.546	GH
B1	21.265	D	11.538	H	8.068	J	6.659	K
B2	40.742	B	17.354	E	13.471	G	10.233	I
LSD ± 1.214								

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 compression direction (radial) swelling value

According to results shown in Table 12, the highest compression direction (radial) swelling value (46.034%) was obtained in without heat treatment samples that were densified under A2 conditions, whereas the lowest value (3.411 and 3.174%) was obtained in the samples which heat treatment applied at 200 and 210 ºC but undensified.

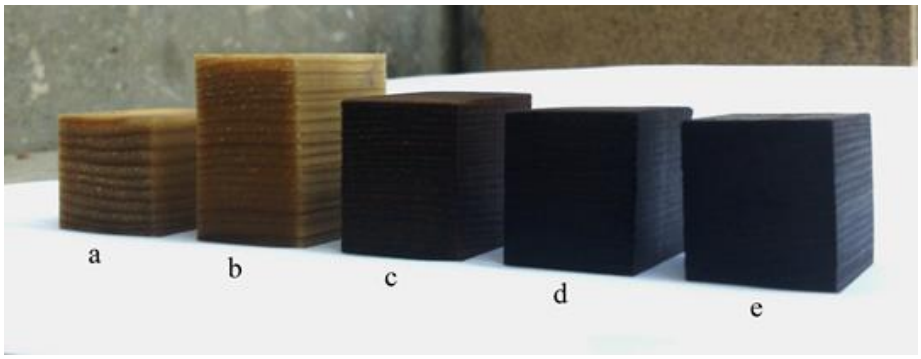

Fig. 2. The appearance of samples after soaking in water (a: control; b: densified; c: densified + treated at 190 ºC; d: densified + treated at 200 ºC; e: densified + treated at 210 ºC)

Density

Analysis of variance results of air-dry density values of Scots pine samples thermo-mechanically densified and heat treated are given in Table 13.

Table 13. 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)
Densification (A)	4	1.498	0.374	411.3279	0.0000*
Heat treatment (B)	3	0.017	0.006	6.2373	0.0005*
Interaction (AB)	12	0.006	0.001	0.5608	ns**
Error	180	0.164	0.001		
Total	199	1.685			

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

According to analysis of variance results, densification and heat treatment factors on air-dry density values of Scots pine samples were found to be significant, but their reciprocal interactions were not significant (P≤0.05). Mono comparison results of Duncan Test conducted by using LSD critical value in densification and heat treatment level are given in Table 14. According to results, the highest air-dry density value (0.764 g/cm^3) at densification level was obtained in the samples densified under A2 conditions, and the lowest value (0.538 g/cm^3) was obtained in the undensified samples. Results showed parallelism with the actual compression ratios after densification, such that air-dry density values were found to be higher in the groups where high compression ratios were obtained. These increases in densities can be explained by noting that the void volume of the wooden material decreased and number of cell walls per unit volume increased after compression. In different studies, it was stated that density shows increases with increasing compression ratio (Blomberg *et al.* 2005; Ünsal and Candan 2008; Ünsal *et al.* 2011; Arruda and Menezzi 2013). It was reported that the density increase obtained by compression of wooden material to a large extent depends on the level of compression with the used densification method and the properties of the wood types (Rautkari 2012).

Table 14. Comparative Results of Duncan Test Related to Air-dry Density Values at Densification and Heat Treatment Level

Densification	$\bar{x}$ (g/cm^3)	*HG*	*LSD*
Undensified	0.538	E	± 0.0139
A1	0.618	C	
A2	0.764	A*	
B1	0.600	D	
B2	0.741	B	
Heat treatment	$\bar{x}$ (g/cm^3)	*HG*	*LSD*
Untreated	0.665	A*	± 0.0124
190 ºC	0.654	AB	
200 ºC	0.649	BC	
210 ºC	0.640	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

The highest air-dry density value (0.665 g/cm^3) in heat treatment level was obtained in the samples without heat treatment; the lowest value (0.640 g/cm^3) was obtained in the samples for which heat treatment was applied at 210 ºC. Small scale decreases in air-dry density values have been encountered with heat treatment application and increased heat treatment temperature. These decreases can be explained based on an understanding that mass losses took place in the samples and decreases in amount of equilibrium moisture after application of heat treatment. In the literature it is stated that mass losses play a significant role in density losses, depending on heat treatment (Fengel and Wegener 1989; Yıldız 2002; Perçin 2012). Moreover, it has been stated that the main reasons for the decrease of the density of wood after heat treatment are: degradation of wood components (mainly hemicelluloses) 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).

CONCLUSIONS

1. In this study, effects of heat treatment applied with ThermoWood® process on some physical properties of Scots pine (*Pinus sylvestris* L.) wood thermo-mechanically densified at different temperatures and target compression ratios, have been investigated. Heat treatment applied at various temperatures was found to remarkably influence the physical properties of densified Scots pine.
2. There were observed losses of up to 22% in the target compression ratios due to spring-back and hygroscopic properties of the wooden material. At the same target compression ratio (20% or 40%), higher actual compression ratio and higher density increases were obtained in the samples densified at 110 ºC with respect to the ones densified at 150 ºC. While density of Scots pine samples exhibited increases from 11% to 42% depending on compression ratios after densification processes, small decreases between 1 to 4% in density occurred after heat treatment.
3. The compression ratio recovery value before heat treatment was found to be close to 100%. Even in the samples densified at the 20% target compression ratio, values above

100% were obtained for the compression ratio recovery value. The effect of compression ratio recovery was considerably reduced with heat treatment and increased application temperature, and an improvement of 80% was provided with respect to the samples without heat treatment. Furthermore, an improvement of 67% was obtained in spring-back effect, and an improvement of 72% was obtained in compression direction (radial) swelling with the heat treatment applied at 210 ºC.

4. It has been seen that heat treatment applied with ThermoWood® process will be useful in improving some properties (dimensional stabilization) of wooden material which was thermo-mechanically densified, and properties of the product (*Densified-ThermoWood*®) obtained by using both methods together can be enhanced.

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