The Effects of Densification and Heat Post-Treatment on Hardness and Morphological Properties of Wood Materials

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This study investigated the effects of densification and heat post-treatment on the Janka hardness and microscopic structure of relatively low-density Uludağ fir, linden, and black poplar woods. Wood samples were densified with compression ratios of 25% and 50% at 100 °C and 140 °C, respectively. Heat post-treatment was then applied to the samples at 185 °C and 212 °C for 2 h. The hardness in the radial and tangential directions was determined, and morphological changes in the cell structures were analyzed using scanning electron microscopy (SEM). The hardness values in the radial and tangential directions of the densified samples increased depending on the compression rate and treatment temperature. The hardness values in both directions were higher in the 50% compressed samples. For samples compressed at 140 °C, the hardness values were higher in the tangential direction, whereas the samples compressed at 100 °C were higher in the radial direction. After the heat post-treatment process, the hardness values of all samples decreased. As the treatment temperature increased, more adverse effects on the hardness was noted. According to the SEM analyses, the densification and heat post-treatment deteriorated the cell structure of the samples. The more cell deformation was observed in the samples densified at 100 °C with compression ratio 50% and high heat post-treatment temperature.

Keywords: Densification; Heat post-treatment; Wood; Hardness; Morphological properties

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INTRODUCTION

Wood is an engineered material with excellent features. It has easy machinability, aesthetic appearance, and high strength properties, as well as being light-weight, sustainable, and recyclable. Because wood is an organic material, it can be degraded by the environment from water, light, fire, and microorganisms.

The properties of wood depend on its chemical and structural characteristics. These can be modified with different wood modification techniques. Thus, wood can be made more resistant to destructive environmental factors (Bami and Mohebby 2011). Heat treatment is a common modification technique used commercially to improve the undesirable properties of wood. Heat treatment leads to permanent changes in the structure of the chemical components of wood (hemicelluloses, cellulose, and lignin) (Kamdem *et al.* 2002; Tjeerdsma and Militz 2005; Boonstra *et al.* 2006; Yang *et al.* 2007; Kocaefe *et al.* 2008; Esteves and Pereira 2009; Tumen *et al.* 2010; Aydemir *et al.* 2011). As a result,

the equilibrium moisture content (EMC) and hygroscopicity of heat-treated wood decreases. In this way, the dimensional stability (Aydemir *et al.* 2011; Pelit *et al.* 2014, 2016; Aytin *et al.* 2015; Kocaefe *et al.* 2015) and decay resistance (Kamdem *et al.* 2002; Lekounougou and Kocaefe 2014; Yalçın and Şahin 2015) of wood increases. Some of the strength properties (modulus of elasticity, bending strength, *etc*.) of heat-treated wood may decline because of thermal degradation, which is the primary disadvantage of heat treatment (Bekhta and Niemz 2003; Yıldız *et al.* 2006; Boonstra 2008; Korkut *et al.* 2008; Perçin *et al.* 2015). Additionally, the natural appearance of wood changes (Mitsui *et al.* 2001; Bekhta and Niemz 2003; Gündüz *et al.* 2010).

The mechanical properties of wood positively correlate with its density, and the mechanical strength can be improved by increasing the density. An increment of wood density is particularly important for low-density wood species (Laine *et al.* 2013; Sandberg *et al.* 2013). Wood can be densified by applying mechanical high-pressure compression with heat and/or steam. In addition, wood can be densified by saturating its pore volume with natural or synthetic resins (Kollmann 1936; Stamm 1964). A major disadvantage of mechanically densified wood is the recovery of its initial dimensions after exposure to water or heat. The dimensional stability of mechanically densified wood can be significantly improved by heat post-treatments (Welzbacher *et al.* 2008; Fang *et al.* 2011; Cai *et al.* 2013; Pelit *et al.* 2014, 2016). In the mechanical densification process, wood materials under low temperature, low moisture content, and short process time exhibit glassy behavior. This form can be described as hard and brittle. Under higher temperatures, higher moisture content, and longer processing time, wood exhibits elastic behavior. This form is described as flexible. The transition temperature in between these two states is related to the phase-changing origin and is called the "glass transition temperature." The viscoelastic structure of the wood plays an important role in mechanical densification. When the structure of hemicellulose and lignin is glassy, deep cracks can occur in the wood and the polymers are in a fragile state. When the wood material temperature is above the glass transition temperature, amorphous polymers are rearranged without any major deformation or fracture in the cell structure (Wolcott *et al.* 1990, 1994; Kutnar and Sernek 2007). The stress relaxation of wood is highly dependent on the loading conditions; for example, higher temperature and increased moisture content enhance stress relaxation (Kelley *et al.* 1987).

This study determined the changes in the microscopic structure of newly-tested Uludağ fir (*Abies bornmulleriana* Mattf.), linden (*Tilia grandifolia* Ehrh.), and black poplar (*Populus nigra* L.) woods after mechanical densification at different compression ratios, temperatures, and heat post-treatments to prevent dimensional recovery. Additionally, the hardness properties of the mechanically densified and heat post-treated samples were determined.

EXPERIMENTAL

Wood Materials

Uludağ fir (*Abies bornmulleriana* Mattf.), linden (*Tilia grandifolia* Ehrh.), and black poplar (*Populus nigra* L.) trees were supplied as timber from a lumber yard in Düzce, Turkey. The sapwood was cut from the logs with an automatic band saw. Rough-scale planks were formed, and the cuts were determined by considering the annual rings parallel to the surface (tangent section) and the sample dimensions. No rot, knot, crack, color, or

density differences were present in the samples (TS 2470 1976). The samples were initially subjected to a natural drying process to approximately 12% moisture content and then cut to the dimensions given in Table 1. Before the densification process, the samples were held in a conditioning cabin with a relative humidity of 65 ± 3% and a temperature of 20 ± 2 °C until they reached a stable weight (TS 2471 1976).

Table 1. Dimensions of Samples before Densification

Compression Ratio (%)	Length - Longitudinal Direction (mm)	Width - Tangential Direction (mm)	Thickness - Radial Direction (mm)
Control	400	95	20
25	400	95	27
50	400	95	40

Thermo-Mechanical Densification and Heat Post-Treatment

The thermo-mechanical densification process was done with a hydraulic press at the compression ratios of 25% and 50%, with temperatures of 100 ± 5 °C and 140 ± 5 °C for 10 min. After thermo-mechanical densification, the heat post-treatment was carried out on the wood samples to provide a measure of dimensional stability. The heat treatment was conducted under the protection of water vapor at the two proposed temperatures (185 °C and 212 °C) for 2 h. The thermo-mechanical densification and heat post-treatment processes have been described in detail by Pelit *et al.* 2016. After the heat post-treatment, the samples remained at a temperature of 20 ± 2 °C and relative humidity of 65 ± 3% until a stable weight was achieved. The densified and heat post-treated samples were then cut into smaller samples with the dimensions of 50 × 20 × 20 mm^3 (longitudinal × tangential × radial). Test samples were prepared in a number sufficient to accommodate 10 repetitions for each variable.

Determination of Janka Hardness

The Janka hardness was accessed in the radial and tangential directions according to the procedures described in TS 2479 (1976). Janka hardness values (Hj) were calculated according to the following equation (Eq. 1),

$$H_j = KP \ (N/mm^2) \quad (1)$$

where P is the load needed for the loading tip moving at 3 to 6 mm/min to penetrate to a certain depth (N), and K is the coefficient equal to 1 when the loading tip penetrates to a depth of 5.64, while equal to 4/3 when it penetrates to a depth of 2.82 mm.

At the end of the tests, the moisture content of the samples was determined according to TS 2471 (1976), and the moisture content of the samples, which deviated from 12%, was determined. Strength values were corrected (revised to 12% moisture content) according to the following equation (Eq. 2),

$$\sigma_{12} = \sigma_m \ [1 + \alpha \ (m - 12)] \quad (N/mm^2) \quad (2)$$

where σ_{12} is the hardness at a 12% moisture content (N), σ_m is the hardness at the moisture content deviating from 12% (N), α is the constant value showing the relationship between hardness and moisture content (α = 0.025), and m is the moisture content (%) during the tests.

Scanning Electron Microscopy (SEM) Analysis

Changes in the anatomical structure of the untreated, densified, and densified and heat-treated wood samples were investigated using the FEI Quanta FEG 250 scanning electron microscope (Dawson Creek Drive Hillsboro, Oregon, USA). Small wood samples with the dimensions of 10 × 10 × 10 mm^3 (longitudinal × tangential × radial) were prepared for SEM analysis, and the wood sample surfaces were sputter coated with gold. Microscopic images at different magnifications were obtained from cross-sectional segments of the fir, linden, and poplar wood samples.

Statistical Analysis

Analysis of variance (ANOVA) testing was carried out to determine the effect of heat post-treatment on the Janka hardness values of the densified fir, linden, and poplar woods at the 0.05 significance level. Significant differences between the groups were compared using Duncan's test.

RESULTS AND DISCUSSION

Hardness

The ANOVA results of the Janka hardness measurements in the radial and tangential directions from samples densified and heat post-treated are given in Table 2. The effects of wood type, densification, and heat treatment factors on the Janka hardness in the radial and tangential directions were statistically significant ($P \leq 0.05$).

Table 2. Analysis of Variance Results for Janka Hardness

Test	Factors	Degrees of freedom	Sum of squares	Mean square	F-value	Level of significance ($P \leq 0.05$)
Hardness (radial direction)	Wood type (A)	2	6927.052	3463.526	380.0330	0.0000*
	Densification (B)	4	33815.746	8453.937	927.6023	0.0000
	Heat treatment (C)	2	2399.803	1199.902	131.6584	0.0000
	Interaction (AB)	8	597.785	74.723	8.1989	0.0000
	Interaction (AC)	4	16.815	4.204	0.4612	ns
	Interaction (BC)	8	515.793	64.474	7.0744	0.0000
	Interaction (ABC)	16	109.193	6.825	0.7488	ns
	Error	405	3691.069	9.114		
	Total	449	48073.257			
Hardness (tangential direction)	Wood type (A)	2	12365.255	6182.628	945.7134	0.0000
	Densification (B)	4	47987.752	11996.94	1835.088	0.0000
	Heat treatment (C)	2	3172.281	1586.140	242.6208	0.0000
	Interaction (AB)	8	3810.742	476.343	72.8628	0.0000
	Interaction (AC)	4	93.447	23.362	3.5735	0.0070
	Interaction (BC)	8	50.047	6.256	0.9569	ns
	Interaction (ABC)	16	76.307	4.769	0.7295	ns
	Error	405	2647.699	6.538		
	Total	449	70203.530			

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

Duncan's one-way comparison results conducted for the factors of wood type, densification, and heat treatment are given in Table 3. The highest hardness value in the radial direction (39.89 N/mm^2) regarding wood type was obtained from black poplar samples; lower hardness values of 31.75 N/mm^2 and 31.39 N/mm^2 in the radial direction were seen in the Uludağ fir and linden samples, respectively. Regarding densification conditions, the highest radial hardness value (45.61 N/mm^2) was observed in the samples densified under D2 conditions, and the lowest hardness value (21.94 N/mm^2) was observed in the undensified samples. According to the heat treatment level, the highest radial hardness value (37.37 N/mm^2) was observed in the untreated samples, while the lowest radial hardness value (31.76 N/mm^2) was observed in the samples subjected to heat treatment at 212 °C.

Table 3. Duncan's Test Results for Mean Values

Factors	Hardness (radial direction) (N/mm^2)		Hardness (tangential direction) (N/mm^2)	
	Mean	HG	Mean	HG
Wood type				
Uludağ fir	31.75	*b*	33.02	*b*
Linden	31.39	*b*	32.12	*c*
Black poplar	39.89	*a*	43.66	*a*
Densification				
Undensified	21.94	*e*	19.39	*e*
D1	31.76	*c*	32.68	*d*
D2	45.61	*a*	46.13	*b*
D3	29.82	*d*	35.15	*c*
D4	42.59	*b*	47.89	*a*
Heat treatment				
Untreated	37.37	*a*	39.67	*a*
185 ºC	33.90	*b*	35.93	*b*
212 ºC	31.76	*c*	33.20	*c*

D1: 100 °C/25%; D2: 100 °C/50%; D3: 140 °C/25%; D4: 140 °C/50%; HG*:* homogeneous group

With respect to wood type, the highest hardness (43.66 N/mm^2) was observed in the tangential direction of black poplar samples, and the lowest hardness value (32.12 N/mm^2) was observed in the radial direction of linden samples. Regarding densification conditions, the highest tangential hardness value (47.89 N/mm^2) was observed in the samples densified under D4 conditions, and the lowest tangential hardness value (19.39 N/mm^2) was observed in the undensified samples. For the heat treatment level, the highest tangential hardness value (39.67 N/mm^2) was observed in the samples without heat treatment, while the lowest (33.20 N/mm^2) was observed in the samples subjected to heat treatment at 212 °C (Table 3).

A comparison of the hardness values in the radial and tangential directions of densified and heat-treated Uludağ fir, linden, and black poplar samples are given in Figs. 1 and 2. According to Fig. 1, the highest hardness values in the radial direction were obtained from poplar samples, followed by fir and linden samples, respectively. There was no significant statistical difference between the hardness values of the fir and linden samples. Radial hardness values increased depending on the compression ratio, and higher

hardness values were obtained from samples densified at the highest compression ratio (50%). The effect of the compression temperature on the radial hardness value was significant. The hardness value of samples compressed at 100 °C was higher than the samples compressed at 140 °C.

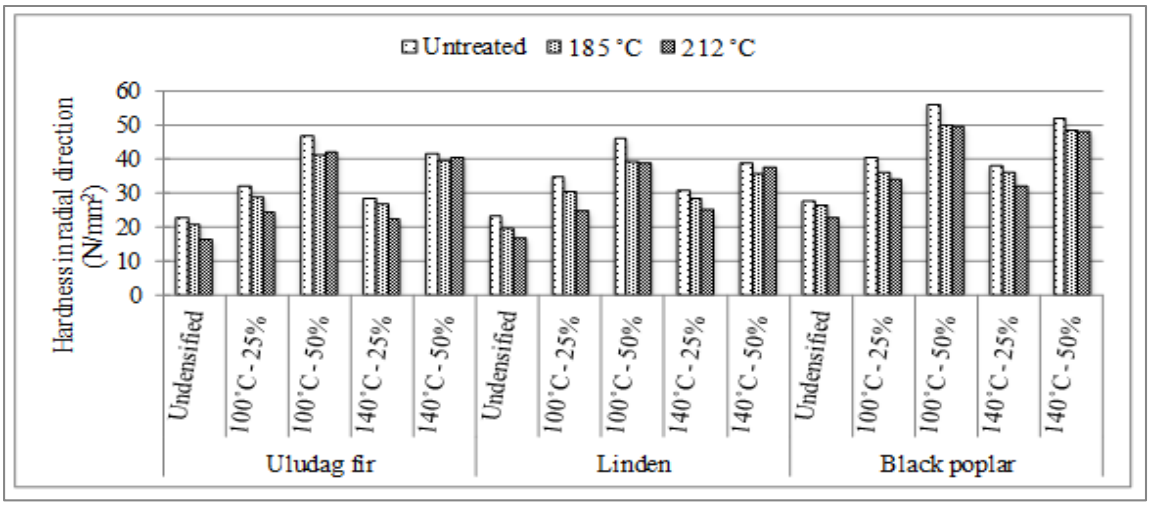

Fig. 1. Comparison of the hardness measurements in the radial direction

After the densification process, the radial hardness values of the Uludağ fir, linden, and black poplar samples increased by up to 105%, 97%, and 103%, respectively, compared with the control samples (Table 4). Several studies on the densification of wood material using different methods stated that the increment of hardness of the samples depended on an increase in the compression ratio and density (Morsing 2000; Navi and Girardet 2000; Blomberg *et al.* 2005; Rautkari *et al.* 2009; Ünsal *et al.* 2011; Fang *et al.* 2012; Ülker *et al.* 2012; Laine *et al.* 2013; Pelit *et al.* 2015). Radial hardness values decreased in all heat post-treated samples. Moreover, these decreases were generally associated with increasing heat post-treatment temperature. However, the increasing heat post-treatment process temperature exhibited no effect on the radial hardness value of samples densified at the 50% compression ratio. Although the radial hardness values of densified samples decreased with increasing heat post-treatment temperature, these values were still significantly higher than undensified samples without heat treatment (controls) (Table 4). In a similar study, it was reported that the heat treatment process, performed at different temperatures and processing times, exhibited an adverse effect on the hardness values of densified poplar veneers; however, these hardness values were 2 to 3 times higher compared to the control samples (Fang *et al.* 2012). Another study indicated that thermal processes applied to densified Scots pine samples had no significant effect on the hardness values (Rautkari *et al.* 2013).

According to Fig. 2, the hardness values in the tangential direction, similar to the radial direction, were higher in the poplar samples. After the densification process, the hardness values in the tangential direction of the samples increased depending on the compression ratio. Compared to the control (undensified) samples, tangential hardness values increased by 121%, 110%, and 147%, respectively, for the 50% compressed Uludağ fir, linden, and black poplar samples (Table 4). For the compression temperatures, the samples compressed at 140 °C exhibited higher hardness values in the tangential direction than those compressed at 100 °C. This situation was different for the hardness values in the

radial direction. According to SEM analysis (Figs. 3c, 4c, and 5c), a greater amount of deformations (breakage, cracking, *etc*.) of the cell structure occurred at the lower temperature (100 °C), which may have influenced the results. A previous study reported that wood cell deformation occurred more frequently in samples densified at a lower temperature and a higher compression ratio, thus decreasing the resistance properties (Tabarsa and Chui 1997). Moreover, the type and amount of cell deformation had a significant effect on the physical and mechanical properties of the densified wood material (Kultikova 1999; Navi and Girardet 2000; Kutnar *et al.* 2009). The hardness values in the tangential direction of the densified fir, linden, and poplar samples were reduced after heat-treatment, and as the processing temperature increased, the hardness values gradually decreased. However, the hardness values in the tangential direction of all densified (especially at 50% compression ratio) samples were higher than the untreated control samples (Table 4).

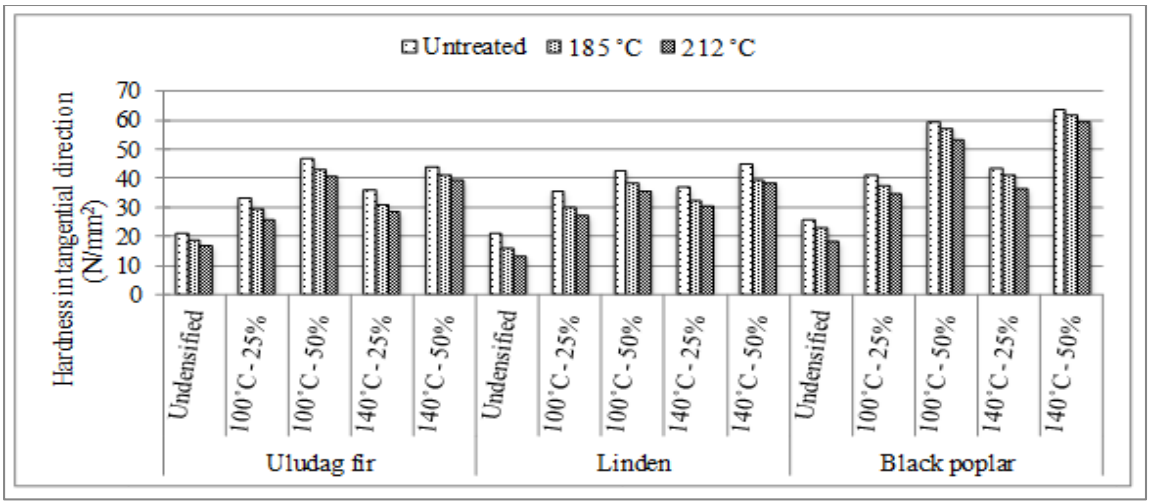

Fig. 2. Comparison of the hardness measurements in the tangential direction

Table 4. Rates of Change in the Radial and Tangential Hardness Values of Wood Types after Densification and Heat Treatment (%)

Densification	Heat treatment (°C)	Wood type					
		Uludağ fir		Linden		Black poplar	
		Radial	Tangential	Radial	Tangential	Radial	Tangential
Undensified	185	-9.09	-10.31	-15.55	-25.88	-4.21	-9.80
	212	-27.15	-19.06	-27.54	-38.68	-17.83	-28.08
100 °C/25%	Untreated	40.23	58.27	48.91	66.99	45.68	59.39
	185	25.62	41.11	30.66	39.80	29.79	45.16
	212	6.91	21.77	5.95	28.18	22.91	33.88
100 °C/50%	Untreated	105.12	121.10	97.43	99.53	102.56	130.73
	185	80.89	103.66	68.01	80.26	80.51	120.93
	212	84.52	93.30	66.21	66.53	78.35	107.66
140 °C/25%	Untreated	24.53	71.72	31.73	74.31	36.67	68.49
	185	17.23	46.15	21.88	50.59	30.30	59.43
	212	-0.74	35.74	7.79	43.51	14.95	42.71
140 °C/50%	Untreated	82.99	108.03	65.87	109.94	88.22	147.30
	185	73.98	96.48	53.36	84.72	74.32	139.52
	212	77.35	86.26	61.84	79.04	73.23	130.14

Scanning Electron Microscopy

The SEM micrographs of cross-sections from untreated and heat-treated linden, black poplar, and Uludağ fir samples at different magnifications are shown in Figs. 3, 4, and 5.

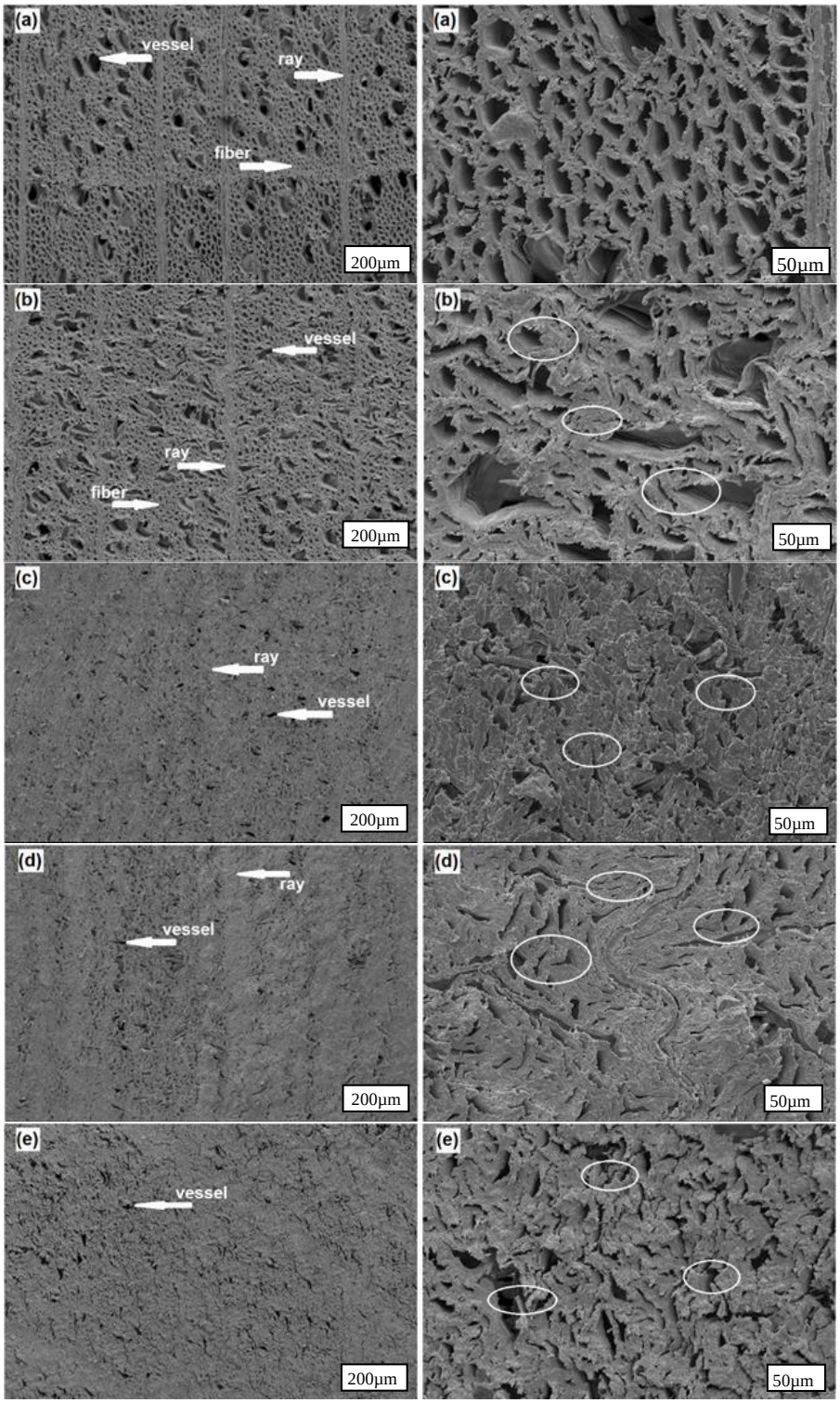

Fig. 3. Scanning electron microscope images of cross-sections of linden wood samples: a) control (untreated); b) 25% compressed at 100 °C; c) 50% compressed at 100 °C; d) 50% compressed at 140 °C; e) 50% compressed at 140 °C and heat post-treated at 212 °C

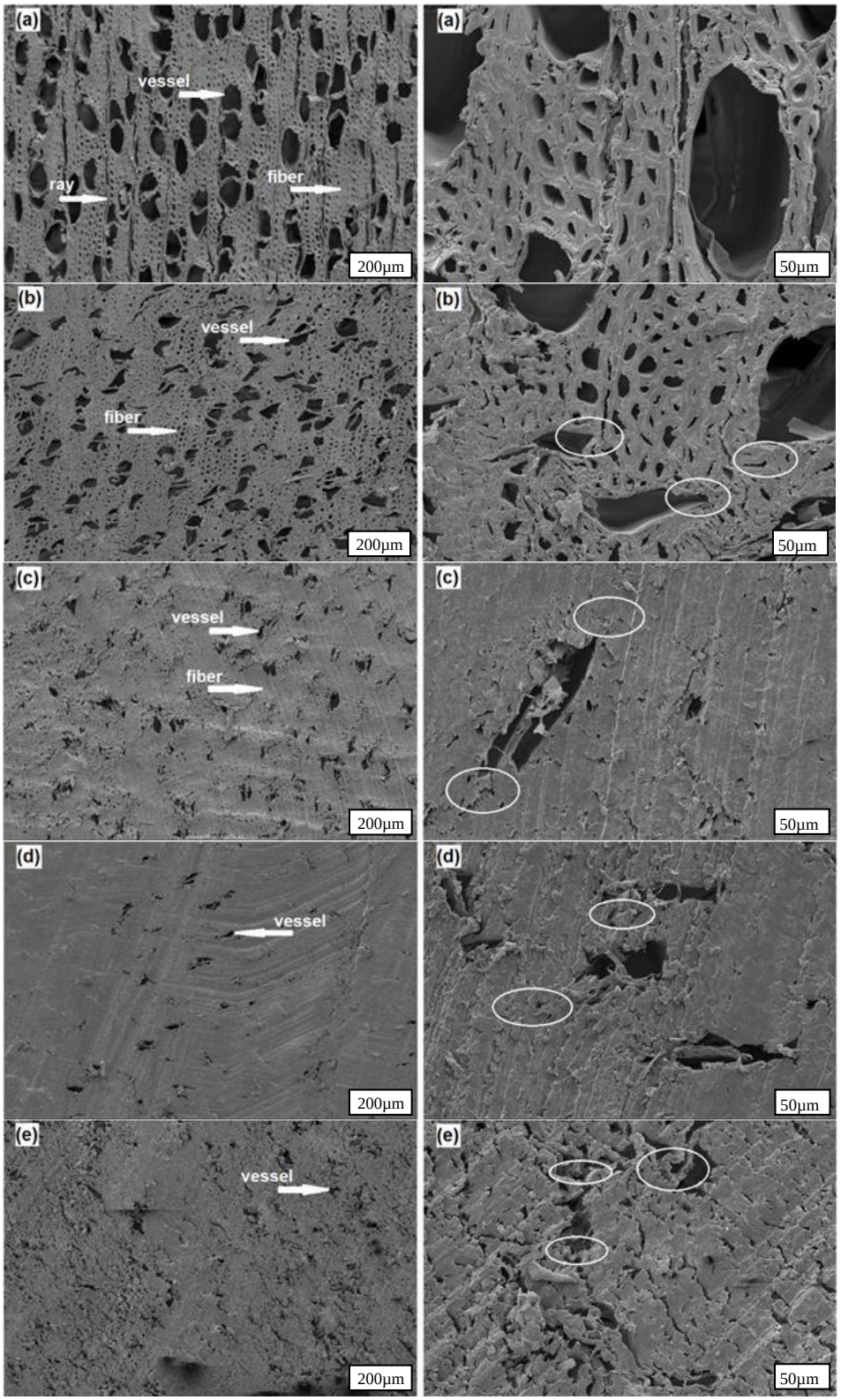

Fig. 4. Scanning electron microscope images of cross-sections of black poplar wood samples: a) control (untreated); b) 25% compressed at 100 °C; c) 50% compressed at 100 °C; d) 50% compressed at 140 °C; e) 50% compressed at 140 °C and heat post-treated at 212 °C

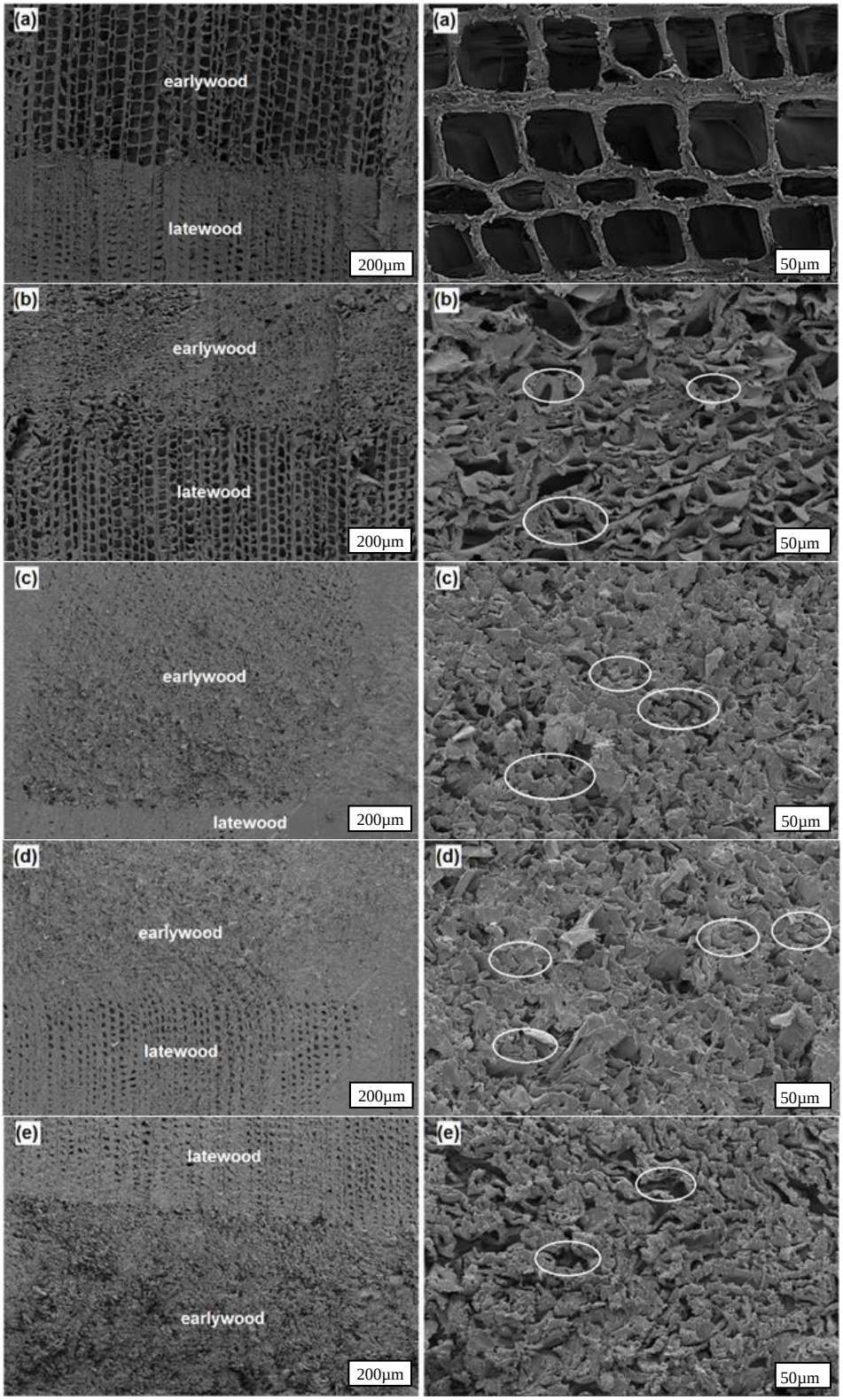

Fig. 5. Scanning electron microscope images of cross-sections of Uludağ fir wood samples: a) control (untreated); b) 25% compressed at 100 °C; c) 50% compressed at 100 °C; d) 50% compressed at 140 °C; e) 50% compressed at 140 °C and heat post-treated at 212 °C

According to the SEM analysis, densification and heat treatment applications resulted in a significant difference in the anatomical structure of the Uludağ fir, linden, and black poplar wood samples. Depending on the compression ratio and the processing temperature, the void volume decreased in all wood types and cell deformation occurred. Cell deformations (breakage, cracking, *etc*.) from compression occurred more frequently at the 50% compression ratio than the 25% compression ratio. No significant deformations were observed in the cell wall of the 25% compressed samples. The changes in the cell wall structure of the samples took the form of elastic buckling instead of breaking or cracking (Figs. 3b, 4b, and 5b). In previous studies of solid wood and veneer densification, the compression ratio had an effect on the cell deformation, thus increasing the compression ratio resulted in collapses, breakage, and cracking in the cell wall (Tabarsa and Chui 1997; Kultikova 1999; Doğu *et. al* 2010; Ahmed *et al.* 2013; Bekhta *et al.* 2015). Furthermore, it was observed during the densification process that the internal stress increased significantly as a result of the elimination of the void spaces and the compression of the wood by the concentration of the collapsed cell walls. Depending on the circumstances of densification and the structure of the cell wall material, cell collapses from elastic buckling, plastic yielding, or brittle crushing also contributed to increased stress (Wolcott *et al.* 1989; Kutnar and Sernek 2007).

Regarding the compression temperature, cell deformation occurred at both 100 °C and at 140 °C. However, cell deformations were observed primarily in samples compressed at 100 °C. Cell wall deformations primarily took the form of breaking or cracking. This situation was more obvious in the linden and Uludağ fir samples (Figs. 3c and 5c). Thus, the cell wall was not highly damaged in the samples compressed at the higher compression ratio (50%) and at 140 °C. In particular, this condition was demonstrated in the linden wood samples (Fig. 3d). In the literature, the viscoelastic nature of wood plays a major role in compression and densification. When the wood temperature rises above the glass transition temperature of its amorphous polymers, considerable deformation can occur without fractures (Kutnar and Sernek 2007). In a different study, microscopic images of cross-sections showed that thermo-mechanical densification created vessel collapse, however the cells were not entirely deformed and the lumens remained open (Navi and Girardet 2000). When the cell structures of the densified post heat-treated samples were analyzed, the heat treatment at a high temperature caused degradation in the cell wall in addition to the deformation by compression (Figs. 3e, 4e, and 5e). In previous studies, the heat process applied to densified wood was reported to lead to more degradation, especially to the hemicellulose in the polymers, than in normal wood (Dwianto *et al.* 1997; Dubey 2010). The void volume of the heat-treated samples after densification increased slightly compared to samples that were only densified. This situation indicates that a portion of the cell wall components had evaporated from the wood under the effect of high temperature, decreasing the amount of water and resulting in the reduction in the EMC.

According to the SEM images derived from a larger area (200 µm/300X magnification) of the densified samples, the thin-walled earlywood zone was more compressed than the thick-walled latewood zone. Moreover, cell deformations were more numerous in the earlywood zone, as shown in the Uludağ fir samples (Fig. 5b, 5c, 5d, and 5e). In densification studies using different methods, collapses occurred most frequently in earlywood. In previous microscopic observations of compressed wood samples, the densification process especially deformed earlywood (Tabarsa and Chui 2001; Kutnar *et al.* 2009; Doğu *et al.* 2010; Kultikova 1999; Ahmed *et al.* 2013; Standfest *et al.* 2013).

According to the SEM images of samples that were only heat-treated (Fig. 6), the cell structure in the samples heat-treated at 185 °C and 212 °C changed in comparison with the controls (Figs. 3a, 4a, and 5a). Heat treatment resulted in degradation of the cell structure of the wood samples. For the heat-treatment applied at the highest temperature (212 °C), deformations, such as ruptures and fragmentations, were observed in the cell walls of these samples (Figs. 6b, 6d, and 6f), reducing the hardness values.

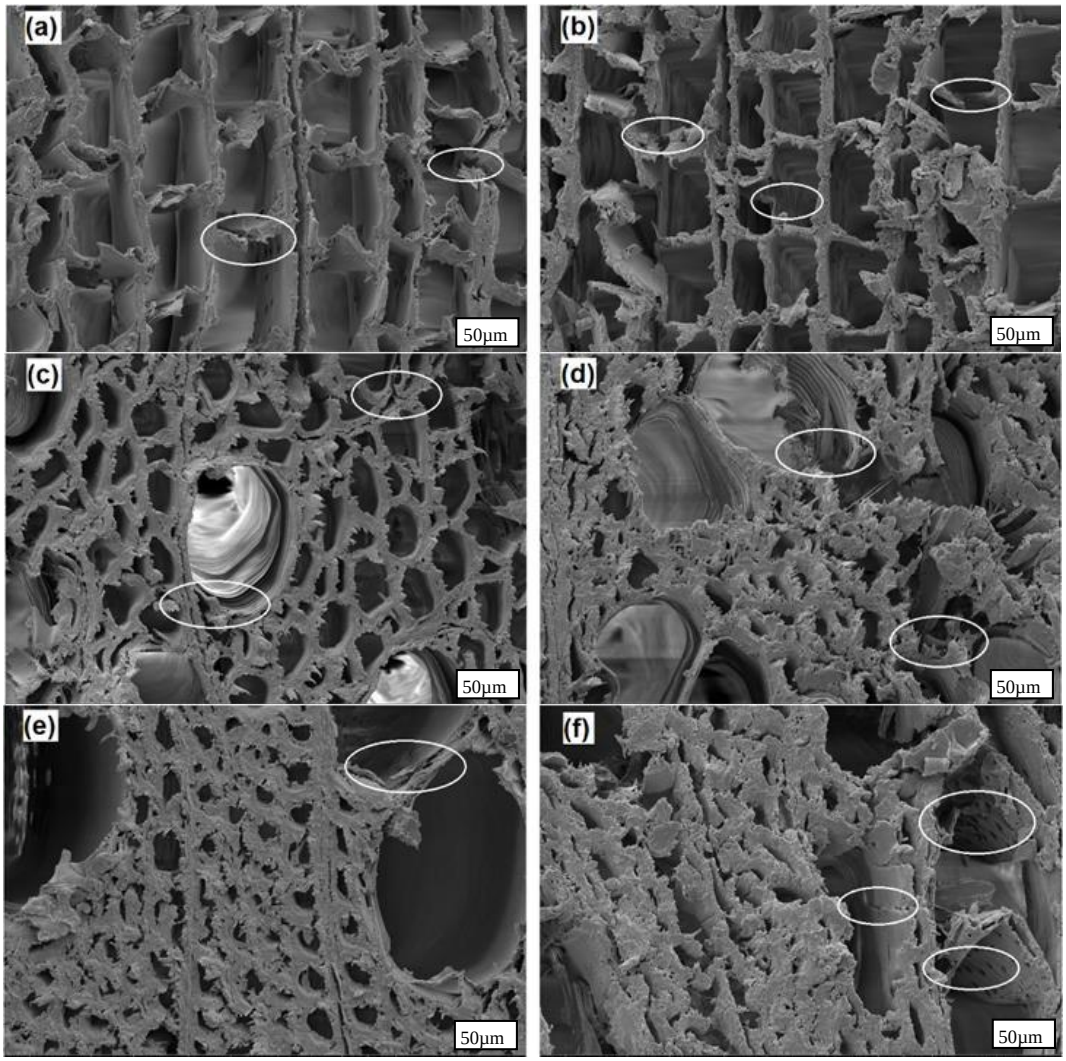

Fig. 6. Scanning electron microscope images in cross-sections of samples subjected only to heat treatment: a) Uludağ fir heat-treated at 185 °C; b) Uludağ fir heat-treated at 212 °C; c) linden heat-treated at 185 °C; d) linden heat-treated at 212 °C; e) black poplar heat-treated at 185 °C; f) black poplar heat-treated at 212 °C

CONCLUSIONS

1. The effects of densification and heat post-treatment on the Janka hardness and the morphological properties of Uludağ fir, linden, and black poplar wood samples were investigated. The Janka hardness values increased in the densified samples depending on the compression ratio and processing temperature. Higher hardness values were

obtained in the samples densified at the highest compression ratio (50%). The effect of compression temperature on the hardness values in the radial and tangential directions was found to be significant. The hardness values in the radial direction of the samples were negatively correlated with the compression temperature.

2. The increase in hardness value after the densification process was higher in the tangential direction. The hardness values of Uludağ fir, linden, and black poplar samples increased up to 121%, 110%, and 147%, respectively. After the heat post-treatment process, the hardness values of all samples decreased; thus, the increase in heat post-treatment temperature had a negative effect on the hardness values in the radial and tangential directions of the samples. However, after heat post-treatment, the hardness values of the densified (especially at the 50% compression ratio) samples were significantly higher than the controls (undensified and without heat treatment) samples.
3. Scanning electron microscopy analysis revealed that the densification and heat treatment applications caused deformations in the cell walls. Greater cell wall deformation was observed in the samples compressed at a lower temperature (100 °C) with the higher compression ratio (50%) and the application of the highest heat post-treatment temperature (212 °C). In addition, it was determined that the earlywood zone was more easily compressed during the densification process and more deformations were observed in this zone compared to the latewood zone.

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