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INFLUENCE OF THERMAL PRETREATMENTS ON DIMENSIONAL CHANGE AND HUMIDITY SENSITIVITY OF DENSIFIED SPRUCE AND POPLAR WOOD

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ABSTRACT

The effect of thermal pretreatments on the dimensional change and humidity sensitivity of densified spruce (*Picea orientalis*) and poplar (*Populus nigra*) wood were investigated. A thermal pretreatment was applied on the wood specimens at 140 °C, 160 °C, 180 °C, and 200 °C for 7 h and 9 h. Wood specimens were then compressed at ratios of 20 % and 40 % at a temperature of 150 °C. The results showed that spring-back and thickness swelling increased in all specimens (thermally pre-treated and untreated) depending on the increase in compression ratio. However, set-recovery was determined higher at 20 % compression ratio. The equilibrium moisture content values of untreated specimens and thermally pre-treated specimens at low temperatures (140 ºC and 160 ºC) were found lower than uncompressed specimens. The impact of compression ratio on equilibrium moisture content was not clear. Thermal pretreatments significantly affected the dimensional stability and hygroscopicity of densified specimens (especially poplar wood). Depending on the increase in thermal pretreatment temperature and duration, spring-back, set-recovery and thickness swelling in wood specimens decreased up to 31 %, 67 % and 62 %, respectively. In addition, equilibrium moisture content and water absorption decreased with the increase in thermal pretreatment temperature and duration. Moreover, the thermal treatment temperature was more important than duration on the investigated properties.

Keywords: Densification, dimensional stability, hygroscopicity, thermal treatment, wood material.

INTRODUCTION

The numerous superior properties of wood make it usable in many structural and non-structural applications. However, since wood is a natural material, it can be easily degraded by various environmental factors (biotic and abiotic). On the other hand, the difficulties in finding wood with good characteristics and the increase in less durable and faster growing species have accelerated the modification studies aimed at improving wood properties (Rowell 2012, Sandberg *et al.* 2017). In wood modification processes, it is aimed to produce materials that are non-toxic during use and do not create any toxic residues when disposed of at the end of its life. Wood modification is essentially methods applied to improve some undesirable characteristics of wood such as low resistance to biodegradation, hygroscopic structure, low dimensional stability, low hardness and low resistance to weathering (Hill 2006, Gérardin 2016, Jones *et al.* 2019).

Density is an important parameter for determining suitable usage areas of wood and giving an idea about many properties of wood. In structural applications and where resistance is important, high-density wood species are generally preferred because of their high mechanical properties. However, wood species with these properties are difficult to obtain due to their limited and high cost. Wood densification, an alternative modification method, it gives improved new properties to wood materials with low-quality and insufficient strength characteristics. Thus, the economic value and usage area of wood species that are less used in the sector can be increased (Sandberg *et al.* 2013, Song *et al.* 2018, Fang *et al.* 2019, Laskowska 2020). There is a growing interest in the use of densified wood. Increased wood density is achieved by compressing the wood material, usually in the radial direction and under suitable conditions (moisture and temperature) to improve its especially mechanical properties. The main purpose of wood densification processes is to improve the hardness and mechanical strength properties of wood species with particularly low density (Laine *et al.* 2013, Báder *et al.* 2018, Cencin *et al.* 2021, Xu *et al.* 2021). In addition to the compression process, wood material can be densified by filling the cavity structure of wood impregnated with different resins or by combining compression and impregnation (Seborg *et al.* 1962, Kollmann *et al.* 1975, Inoue *et al.* 1993, Fukuta *et al.* 2008, Gabrielli and Kamke 2010, Lykidis *et al.* 2020, Pelit and Emiroglu 2020). However, the densified wood obtained by these methods may contain toxic effects depending on the resin properties. The most important issue associated with wood densified by compressing is the fixation of the compressed thickness. Compressed wood produced without any deformation fixation treatment is sensitive to moisture. After compression, the densified wood has a tendency to return to its initial dimensions when exposed to liquid water or humid environments. This undesirable phenomenon is defined as set-recovery and is the main disadvantage of compressed wood (Morsing 2000, Navi and Heger 2004, Rautkari *et al.* 2010, Kutnar and Kamke 2012, Gao *et al.* 2019).

Thermal modification is a generally accepted procedure used to improve some undesirable properties of wood at a temperature greater than 160 °C, without chemical additives and in a limited oxygen environment (Militz 2005, Torniainen *et al.* 2021). The thermal modification of wood is accepted environmentally friendly and is known as the most commercial wood modification process to date (Sandberg *et al.* 2021). Thermal treatments are a physical process. However, it causes chemical changes of the basic components of wood (cellulose, hemicelluloses and lignin), which affects properties such as hygroscopicity, dimensional stability, permeability, and decay resistance in wood (Boonstra 2016). Thermal treatments are a widely used wood modification method, especially to increase dimensional stability and decay resistance (Esteves and Pereira 2009, Ünsal *et al.* 2009, Sandberg *et al.* 2017, Hill *et al.* 2021). The equilibrium moisture content (EMC) of thermally treated wood is reduced and stability resistance is significantly increased by decreasing shrinking and swelling due to ambient conditions (Militz 2002, Bekhta and Niemz 2003, Esteves *et al.* 2007, Korkut and Guller 2008, Kaygın *et al.* 2009, Aydemir *et al.* 2011, Kocaefe *et al.* 2015, Boonstra 2016). This property change is mainly associated with the thermal degradation of hemicelluloses and generally the changes persist as the temperature increases (Hill 2006). In addition to dimensional stability, wood's resistance to biodegradation increases as a result of thermal treatment, especially its resistance to fungi (Boonstra *et al.* 2007a, Dubey *et al.* 2012, Lekounougou and Kocaefe 2014, Yalçın and Şahin 2015, Ayata *et al.* 2017). Moreover, the color of the thermally treated wood can be changed homogeneously to more interesting dark tones (Thompson *et al.* 2005, González-Peña and Hale 2009, Pleschberger *et al.* 2014, Toker *et al.* 2016, Pelit 2017, Sikora *et al.* 2018, Sivrikaya *et al.* 2019, Torniainen *et al.* 2021). However, as an important disadvantage, mechanical strength properties of thermally treated wood generally decreases and wood becomes more fragile and rigid, depending on the processing conditions (treatment temperature, treatment time, ambient condition), wood species, and properties of its anatomical structure, or the moisture content of the wood (Poncsák *et al.* 2006, Yıldız *et al.* 2006, Boonstra *et al.* 2007b, Korkut *et al.* 2008, Perçin *et al.* 2016, Pelit and Yorulmaz 2019). The reduced mechanical strength limits the use of thermally treated wood, especially in structural applications (Esteves and Pereira 2009).

In our previous study, the effect of densification modification on the mechanical properties of thermally treated wood specimens was studied (Pelit and Yorulmaz 2019). The results showed that the hardness and mechanical strength, which were reduced by thermal treatment, improved significantly due to the compression ratio after densification. The goal of this study presented was to investigate the effect of thermal pretreatments on the dimensional stability and hygroscopicity behaviors of densified wood specimens. For this reason, poplar and spruce woods were thermally treated at four different temperatures and at two different durations were densified with two different compression ratios. Spring-back, thickness swelling, set-recovery, equilibrium moisture content (EMC) and water absorption tests were performed to determine the stability and hygroscopicity properties of wooden specimens in this condition.

MATERIALS AND METHOD

Wood material

In this study, Eastern spruce (*Picea orientalis* (L.) Link.) and black poplar (*Populus nigra* L.) wood, which have relatively low densities, were used. Wood materials were supplied as round wood from a timber company in Istanbul, Turkey. Round wood was cut from the sapwood with a band sawing machine taking into account the study methodology. Wood materials were subjected to natural drying (approximately 12 % moisture content), and then cut with a tolerance of 15 % to 20 % from the draft dimensions of the specimens to be used for densification.

Thermal treatment of wood specimens

Thermal treatments were conducted in a laboratory-type oven and at atmospheric pressure. The oven used has a capacity of 48 liters and internal dimensions of 420 × 350 × 330 mm. Sixty test specimens were subjected to heat treatment at one time. Thermal treatment processes of wood specimens are given in Table 1.

Table 1: Thermal treatment processes.

Thermal treatment stages	Temperature (°C)	Duration (h)	Total duration (h)
Drying	103	30 to 36	40 to 47
Thermal treatment	140, 160, 180, and 200	7 and 9	
Cooling	-	2 to 4	

The specimens were separately thermally treated at target temperatures for 7 h and 9 h. After thermal treatment, wood specimens hold in a conditioning cabin (relative humidity (RH) 65 ± 3 % and 20 ± 2 °C) until they reached a stable weight, and then they were cut to the dimensions of 20 mm × 320 mm (tangential direction × longitudinal direction) and thicknesses 20 mm (for non-compressed specimens), 25 mm, and 33,3 mm (radial direction). Wood thicknesses were prepared differently in order to achieve the targeted compression ratios (20 % and 40 %).

Densification of wood specimens

The thermally pre-treated specimens were densified *via* custom-made metal molds in laboratory test press. The compression parameters used for the densification process of the wood specimens are given in Table 2.

Table 2: Densification parameters.

Parameter	**Value**
Compression temperature (°C)	150
Compression ratio (%)	20 and 40
Pre-heating time (min)	20
Closing rate (mm min^{-1})	60
Compressed holding time (min)	10

The wood specimens were first placed in the channels opened on the surfaces of the metal molds (Figure 1). Before compression, specimens were preheated for 20 min. at target temperature. The specimens were then compressed with a speed of 60 mm min^{-1} and in the radial direction. The load was applied until the metal molds made contact to reach the target thickness of 20 mm in wood specimens (Figure 2). The compressed spruce and poplar specimens were hold at 150 °C for 10 minutes. The specimens were then cooled to room temperature under pressure to minimize the spring-back formation.

Densified specimens were conditioned at RH 65 % and 20 °C until they reached a stable weight. The thermally pre-treated and densified specimens were then sized in line with the standards of the applied tests and in a number providing ten repetitions ($n = 10$) for each group.

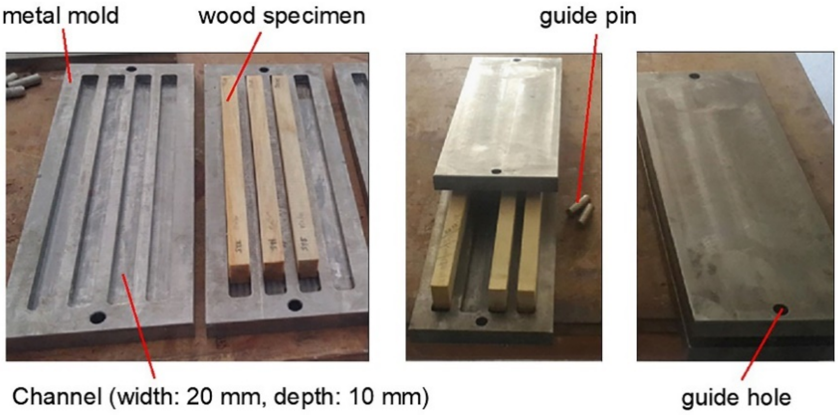

Figure 1: Position of wood specimens in metal molds.

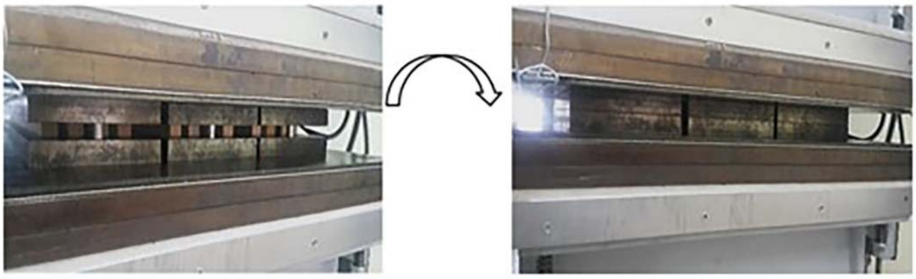

Figure 2: Compression of wood specimens with metal molds in a hot press.

Determination of dimensional change and hygroscopicity properties

Compressed wood tends to return to its initial dimensions after the opening of press due to shape memory effect. This phenomenon is referred to as spring-back and causes a change in the target compression ratio. The actual compression ratio (or compression-set) of the compressed wood specimens was calculated using equation 1, and spring-back values were calculated using equation 2:

$$\text{Compression ratio (\%)} = \frac{T_0 - T_c}{T_0} \times 100 \qquad (1)$$

$$\text{Spring} - \text{back (\%)} = \frac{T_c - T_t}{T_t} \times 100 \qquad (2)$$

where T_o is the initial thickness (mm) of specimens before compression, T_c is the thickness (mm) of specimens kept at 20 ºC ± 2 °C and 65 % ± 3 % RH for eight weeks (until reaching a stable weight) after compression, T_t is the thickness (mm) under pressure (target).

Equilibrium moisture content (EMC) was determined in line with ISO 13061-1 (ISO 2014), and calculated using by equation 3:

$$\text{EMC}\ (\%) = \frac{W_c - W_d}{W_d} \times 100 \tag{3}$$

where W_c is the weight (g) of specimens conditioned at 20 ºC ± 2 °C and RH 65 % ± 3 % for eight weeks and W_d is the weight (g) of specimens after keeping in a heated oven (103 ºC ± 2 °C) for 72 h.

The set-recovery values of the densified specimens after soaking in water was determined using equation 4:

$$\text{Set} - \text{recovery}\ (\%) = \frac{T_m - T_c}{T_o - T_c} \times 100 \tag{4}$$

where T_m is the thickness (mm) of specimens after immersion in water for two weeks.

Thickness swelling of test specimens was analyzed in line with ISO 13061-15 (ISO 2017), and calculated using equation 5:

$$\text{Thickness swelling}\ (\%) = \frac{T_m - T_d}{T_d} \times 100 \tag{5}$$

where T_d is the thickness (mm) of specimens after waiting at 103 ºC ± 2 °C for 72 h. Water absorption was calculated using equation 6:

$$\text{Water absorption}\ (\%) = \frac{W_m - W_d}{W_d} \times 100 \tag{6}$$

where W_m is the weight (g) of specimens after immersion in water for two weeks.

Statistical analysis

Analysis of variance (ANOVA) tests were performed to determine the impact of thermal pretreatments on the dimensional stability and hygroscopicity of densified spruce and poplar specimens at the 0,05 significance level. Then the mean values of the tested properties of the modified wood specimens were compared separately.

RESULTS AND DISCUSSION

Thickness of the specimens before and after the compression, actual compression ratio, spring-back and density values of thermally pre-treated and densified wood specimens are given in Table 3 and Table 4. The results showed that spring-back occurred at different rates depending on the thermal pretreatment conditions and compression ratios after the pressing process in the densified specimens. There was a decrease in the targeted compression ratios depending on the spring-back ratios in the wood specimens. Spring-back values increased with increasing compression ratio in both thermally pre-treated and untreated wood specimens. Spring-back increased by 68 % and 92 %, respectively, in 40 % compressed spruce and poplar specimens compared to 20 %. Due to the increase in compression ratio, the internal stresses increase in densified wood, thus leading to higher spring-back values (Laine *et al.* 2013, Nairn 2006, Pelit *et al.* 2014, Wolcott *et al.* 1989).

Thermal pretreatment conditions affected the springback ratios of the densified wood specimens differently. At lower temperatures (140 ºC and 160 ºC), the springback ratios of the thermally treated specimens generally tend to increase compared to the untreated specimens. These results were similar to results obtained by Kariz *et al.* (2017). However, as of the 180 ºC limit, the spring-back values of the specimens decreased due to the increase in thermal treatment temperature and time. Compared to the untreated samples, the spring-back ratio decreased up to 10 % and 31 %, respectively, in the spruce and poplar specimens that were thermally treated at 200 °C for 9 h (Table 3 and 4). Reducing the internal stresses that occur in wood specimens due to the effect of high pressure during the pressing stage by thermal pretreatments and the decrease in the EMC of the thermally pre-treated specimens may affect the results. Furthermore, previous studies noted that thermal degradations that occur especially in hemicellulose compound with the effect of temperature play a key role in the elimination of spring-back in densified wood (Dwianto *et al.* 1997, Heger *et al.* 2004, Morsing 2000).

Density values of densified wood specimens increased depending on thermal pretreatment conditions and compression ratio. As a result of the increase in the compression ratio, the density of the spruce and poplar specimens increased by 45 % and 46 %, respectively, compared to the control samples (Tables 3 and 4). The rate of increase in the density of mechanically compressed wood generally depends on the level of compression, spring-back effect and characteristics of the wood species (Rautkari 2012, Pelit *et al.* 2014).

Table 3: Properties of thermally pre-treated and densified spruce wood (*n*=10).

Thermal treatment	**Compression ratio (%)**	**Initial thickness (mm)**	**Final thickness (mm)**	**Actual compression ratio (%)**	**Spring-back (%)**	**Density (kg/m^3)**	**Increase in density (%)**
Untreated	Non-compressed	20	20	-	-	382 (37)	-
	20	24,98 (0,22)	21,31 (0,17)	14,68 (0,74)	6,55 (0,84)	434 (27)	13,6
	40	32,86 (0,24)	22,03 (0,22)	32,94 (0,85)	10,16 (1,08)	555 (75)	45,3
140 °C / 7 h	20	24,87 (0,30)	21,29 (0,16)	14,38 (1,08)	6,46 (0,78)	426 (26)	11,5
	40	32,66 (0,25)	22,25 (0,21)	31,88 (1,02)	11,24 (1,07)	530 (59)	38,7
140 °C / 9 h	20	24,91 (0,25)	21,17 (0,12)	15,02 (1,11)	5,85 (0,59)	438 (24)	14,7
	40	32,56 (0,19)	22,14 (0,17)	32,01 (0,67)	10,69 (0,87)	541 (55)	41,6
160 °C / 7 h	20	24,84 (0,25)	21,33 (0,13)	14,13 (0,66)	6,67 (0,67)	432 (36)	13,1
	40	32,51 (0,36)	22,24 (0,20)	31,57 (1,04)	11,21 (0,98)	525 (74)	37,4
160 °C / 9 h	20	24,78 (0,24)	21,30 (0,09)	14,04 (0,98)	6,50 (0,43)	417 (48)	9,2
	40	32,62 (0,25)	22,16 (0,22)	32,07 (1,03)	10,79 (1,12)	516 (33)	35,1
180 °C / 7 h	20	24,81 (0,20)	21,28 (0,08)	14,22 (0,56)	6,41 (0,41)	417 (28)	9,2
	40	32,49 (0,55)	22,07 (0,20)	32,04 (1,47)	10,37 (1,00)	519 (27)	35,9
180 °C / 9 h	20	24,69 (0,25)	21,21 (0,10)	14,07 (0,72)	6,06 (0,49)	414 (25)	8,4
	40	32,37 (0,52)	22,08 (0,13)	31,76 (1,33)	10,39 (0,64)	512 (63)	34,0
200 °C / 7 h	20	24,64 (0,27)	21,23 (0,08)	13,86 (0,83)	6,14 (0,42)	411 (29)	7,6
	40	32,46 (0,41)	21,98 (0,19)	32,27 (0,67)	9,91 (0,95)	495 (45)	29,6
200 °C / 9 h	20	24,70 (0,21)	21,18 (0,07)	14,25 (0,78)	5,88 (0,34)	411 (41)	7,6
	40	32,49 (0,34)	22,00 (0,20)	32,27 (0,79)	10,02 (1,01)	492 (58)	28,8

All values are measurement results of specimens conditioned at RH 65 % and 20 °C.
Values in parentheses are standard deviations.

Table 4: Properties of thermally pre-treated and densified poplar wood (*n*=10).

Thermal treatment	**Compression ratio (%)**	**Initial thickness (mm)**	**Final thickness (mm)**	**Actual compression ratio (%)**	**Spring-back (%)**	**Density (kg/m^3)**	**Increase in density (%)**
Untreated	Non-compressed	20	20	-	-	404 (10)	-
	20	25,19 (0,15)	21,46 (0,09)	14,78 (0,55)	7,31 (0,46)	472 (22)	16,8
	40	33,39 (0,08)	22,65 (0,34)	32,18 (0,92)	13,23 (1,72)	589 (30)	45,8
140 °C / 7 h	20	25,09 (0,07)	21,44 (0,10)	14,52 (0,48)	7,22 (0,50)	463 (25)	14,6
	40	33,29 (0,13)	22,93 (0,33)	31,13 (0,98)	14,64 (1,63)	570 (29)	41,1
140 °C / 9 h	20	25,09 (0,04)	21,57 (0,09)	14,03 (0,41)	7,87 (0,46)	452 (19)	11,9
	40	33,21 (0,22)	22,90 (0,25)	31,05 (0,91)	14,49 (1,26)	573 (38)	41,8
160 °C / 7 h	20	25,02 (0,11)	21,42 (0,19)	14,37 (0,84)	7,11 (0,93)	461 (25)	14,1
	40	33,22 (0,12)	22,86 (0,28)	31,17 (0,76)	14,31 (1,39)	571 (23)	41,3
160 °C / 9 h	20	24,97 (0,12)	21,52 (0,11)	13,82 (0,77)	7,62 (0,54)	461 (28)	14,1
	40	33,21 (0,09)	22,78 (0,35)	31,42 (1,05)	13,89 (1,73)	560 (30)	38,6
180 °C / 7 h	20	24,92 (0,17)	21,32 (0,13)	14,45 (0,63)	6,60 (0,65)	461 (29)	14,1
	40	33,11 (0,10)	22,46 (0,26)	32,17 (0,79)	12,31 (1,29)	556 (29)	37,6
180 °C / 9 h	20	24,90 (0,12)	21,17 (0,14)	14,98 (0,44)	5,83 (0,71)	456 (24)	12,9
	40	33,06 (0,16)	22,40 (0,18)	32,26 (0,41)	11,99 (0,88)	557 (25)	37,9
200 °C / 7 h	20	24,69 (0,18)	21,06 (0,09)	14,68 (0,59)	5,32 (0,46)	438 (27)	8,4
	40	32,84 (0,18)	22,03 (0,17)	32,92 (0,52)	10,14 (0,85)	545 (25)	34,9
200 °C / 9 h	20	24,73 (0,11)	21,01 (0,08)	15,03 (0,45)	5,05 (0,40)	435 (30)	7,7
	40	32,85 (0,13)	21,96 (0,11)	33,15 (0,40)	9,78 (0,56)	535 (36)	32,4

All values are measurement results of specimens conditioned at RH 65 % and 20 °C.
Values in parentheses are standard deviations.

It was observed that the thermal pretreatment temperature and time affected the density values of the wood specimens. Density values generally tend to decrease with increasing temperature and time. This is more evident in 40 % compressed wood specimens. Compared to the untreated samples, the density of the densified spruce and poplar specimens, which were thermally pre-treated for 9 h at 200 °C, decreased by 11 % and 9 %, respectively (Table 3 and 4). The density reduction of thermally treated wood is mostly due to the destruction of hemicelluloses and mass losses as a result of evaporation of extractives (Boonstra 2008; Esteves and Pereira 2009). In addition, it can be said that the lower initial density and decreases in the EMC of thermally treated wood specimens has an effect on the air-dry density results (Kariz *et al.* 2017, Pelit and Yorulmaz 2019).

The ANOVA results for EMC, water absorption, thickness swelling, and set-recovery values of thermally pre-treated and densified specimens are shown in Table 5. According to the results, the effect of thermal treatment conditions and compression ratio on tested properties of spruce and poplar specimens was statistically significant ($p \leq 0,05$). However, only for spruce wood, the effect of thermal treatment conditions on water absorption was no found to be significant (Table 5).

Table 5: ANOVA results for tested properties of thermally pre-treated and densified wood specimens.

Tests	Source	Spruce		Poplar	
		F ratio	p value	F ratio	p value
EMC	Thermal treatment (A)	458,5648	0,0000*	810,5218	0,0000*
	Compression ratio (B)	232,0087	0,0000*	99,0298	0,0000*
	Interaction (A×B)	20,0875	0,0000*	21,9468	0,0000*
Water absorption	Thermal treatment (A)	1,1963	ns	17,3151	0,0000*
	Compression ratio (B)	104,1394	0,0000*	48,6578	0,0000*
	Interaction (A×B)	0,8497	ns	0,4909	ns
Thickness swelling	Thermal treatment (A)	133,0360	0,0000*	339,3149	0,0000*
	Compression ratio (B)	11128,3401	0,0000*	11443,0455	0,0000*
	Interaction (A×B)	37,5094	0,0000*	101,9875	0,0000*
Set-recovery	Thermal treatment (A)	105,9681	0,0000*	483,6466	0,0000*
	Compression ratio (B)	62,1283	0,0000*	33,2390	0,0000*
	Interaction (A×B)	1,3664	ns	4,0171	0,0000*
*: significant at 95 % confidence level; ns: not significant					

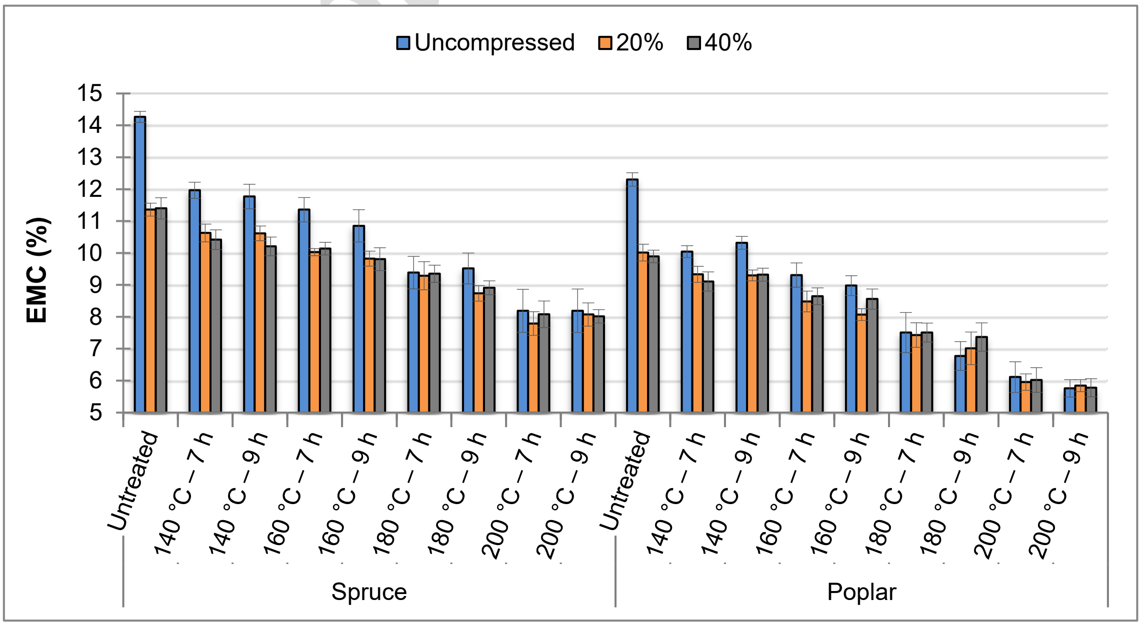

Figure 3: EMC values of thermally pre-treated and densified spruce and poplar specimens (n=10).

Figure 3 results showed that EMC values were generally lower in densified specimens compared to uncompressed specimens. This was quite evident in untreated and thermally pre-treated specimens at low temperatures (140 ºC and 160 ºC). However, no obvious difference was observed between the EMC values of the thermally pre-treated uncompressed and densified specimens at 180 ºC and especially at 200 ºC. Also, the compression ratio (20 % or 40 %) has no significant effect on the EMC values of both thermally treated and untreated specimens (Figure 3).

On the other hand, EMC values of spruce and poplar specimens decreased with increasing thermal pretreatment temperature and time. It was observed that the temperature effect was more important than the time effect on the results. Thermal treatment showed a similar effect in both uncompressed and densified wood specimens (Figure 3). Compared to untreated specimens, the mean EMC of spruce and poplar wood thermally treated at 200 °C decreased by 43 % and 52 % for uncompressed specimens, and 30 % and 41 % for densified specimens, respectively. For EMC, the effect of thermal pretreatment is more pronounced in poplar wood. The hygroscopic behavior of wood is related to the -OH groups in the cell wall structure. -OH groups are present in hemicelluloses, cellulose, and lignin as alcohols, as well as in lignin as phenolic groups. Hemicelluloses are the component with the highest sorptive properties, followed by cellulose and lignin. Thermal modification of wood causes a reduction of OH content (mainly due to hemicelluloses degradation); therefore, it is expected that the EMC will be lower compared to untreated wood (Boonstra 2016, Gérardin 2016, Hill *et al.* 2021).

According to Figure 4, water absorption of both thermally pre-treated and untreated spruce and poplar specimens were higher than uncompressed specimens after densification. In addition, water absorption was detected higher in specimens densified with high compression ratio (40 %). This effect is more pronounced in spruce specimens.

For both wood species, the effect of compression ratio on water absorption tended to decrease with the increase in thermal pretreatment temperature, and closer values were measured in both compression ratios. In the study reported by Pelit *et al.* (2016), thermal post-treatments were applied to thermo-mechanically densified fir and poplar wood specimens. Similarly, water absorption increased with increasing compression ratio in densified specimens. However, contrary to the present study, especially in specimens densified at high compression ratio, lower water absorption was obtained after thermal post-treatment. Thus, it has been observed that the thermal treatment application stage (before or after densification) affects the water absorption results.

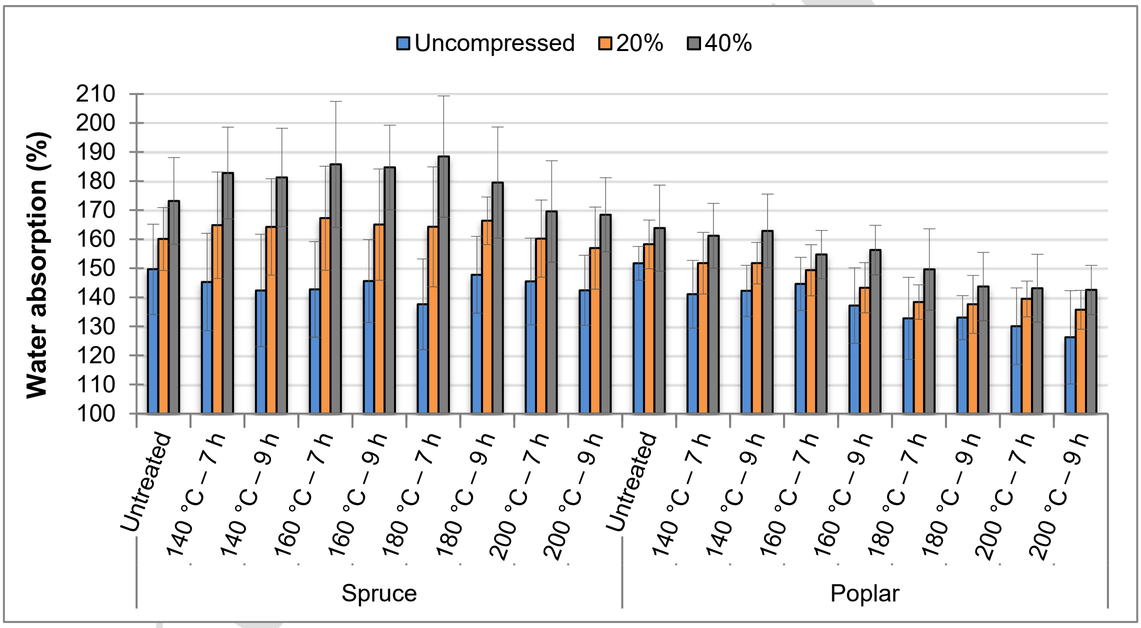

Figure 4: Water absorption values of thermally pre-treated and densified spruce and poplar specimens (n=10).

For spruce wood, thermal treatment applications caused a decrease in water absorption values of uncompressed specimens. However, water absorption tends to increase in densified spruce specimens as a result of the increase in thermal pretreatment temperature up to 200 ºC. In spruce specimens pre-treated at 200 ºC, water absorption was slightly decreased compared to untreated specimens (Figure 4). However, ANOVA results showed that the effect of thermal treatments on water absorption of spruce specimens was not significant (Table 5). For poplar wood, both uncompressed and densified specimens showed a decrease in water absorption values depending on increase in thermal treatment temperature (Figure 4). Compared to untreated specimens, water absorption was decreased by 17 % and 14 %, respectively, in uncompressed and densified poplar specimens thermally treated at 200 °C. In addition, it was determined that the thermal treatment time had a limited effect on the water absorption values of the specimens. The main components (cellulose, hemicelluloses and lignin) of the wood cell wall contain free hydroxyl groups (OH) that attract and hold water by hydrogen bonding. The accessibility of OH groups in the chemical components of wood (especially hemicelluloses) plays an important role in the desorption and water adsorption process (Boonstra 2016). Thermal treatment of wood results in a reduction in the accessible OH content and reduces the content of bound water held in the wood cell wall (Hill *et al.* 2021). On the other hand, the most important parameters in thermal modification are treatment temperature and time, but on many wood properties, temperature is more dominant than treatment time (Bekhta and Niemz 2003, Tjeerdsma and Militz 2005, Kartal *et al.* 2007, Esteves *et al.* 2008).

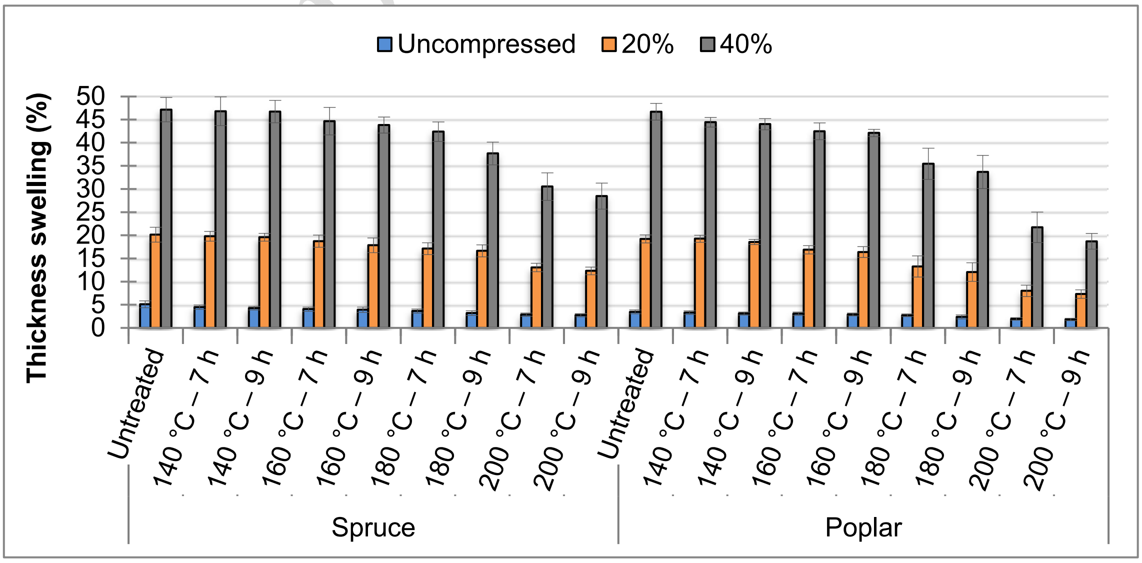

Figure 5: Thickness swelling values of thermally pre-treated and densified spruce and poplar specimens (*n*=10).

Thickness swelling values were found to be significantly higher in densified wood specimens compared to uncompressed specimens. Also, thickness swelling increased with increasing compression ratio in all densified specimens (Figure 5). These determined results are compatible with the findings of previous studies (Cai *et al.* 2013, Pelit *et al.* 2016, Pelit and Emiroglu 2020, Budakçı *et al.* 2021). It is known that the compressed wood tends to revert to its initial dimensions before compression under high humidity conditions or in contact with water. This is caused by the extension of the cell wall, the relaxation of stresses occurring in the wood structure due to compression, and the tendency of the cell wall to revert to its original state, especially due to shape memory (Kollmann *et al.* 1975, Morsing 2000, Seborg *et al.* 1956).

The thickness swelling of both uncompressed and densified specimens decreased and dimensional stability increased depending on the increase in thermal treatment temperature and time. More successful results were obtained in thermally pre-treated poplar specimens. In addition, it was determined that the temperature effect was more significant on the thickness swelling than the thermal treatment time (Figure 5). Compared to untreated specimens, thickness swelling was reduced by up to 40 % and 62 %, respectively, in spruce and poplar specimens that were thermally pre-treated for 9 hours at 200 °C. Kocaefe *et al.* (2015) reported several factors that cause an decrease in the dimensional change of thermally treated wood. These include the mass loss of hygroscopic hemicellulose polymers causing reduction of hydroxyl groups, cross-linking of aromatic rings in lignin, and cross-linking or bridging of cellulose chains due to separation of two hydroxyl groups on adjacent cellulose chains. Also, the overall swelling of the wood is reduced as a result of the reduction in water absorption after thermal treatment, thus increasing its dimensional stability (Boonstra 2016).

The set-recovery was higher in the specimens that were densified at 20 % compression ratio compared to the specimens densified with 40 % compression ratio in both untreated and thermally pre-treated wood specimens. The higher set-recovery at the lower compression ratio is similar to the results of previous studies reported (Kariz *et al.* 2017, Pelit *et al.* 2016, Pelit and Emiroglu 2020). However, the effect of compression ratio on set-recovery decreased due to temperature increase in thermal pre-treated specimens and closer values were determined for both compression ratios (Figure 6).

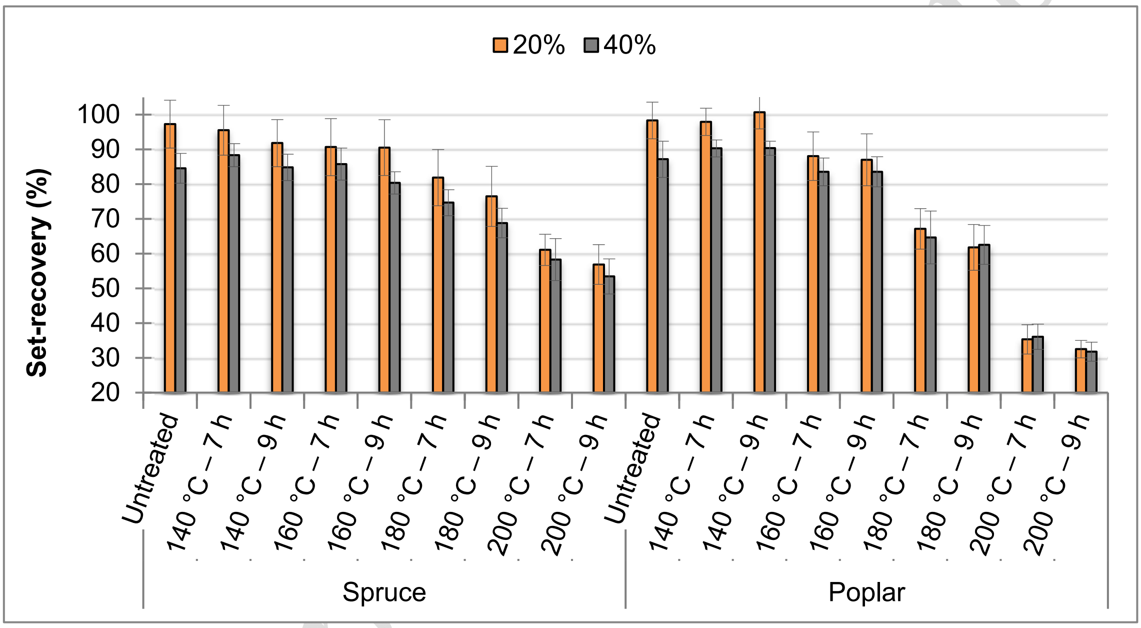

Figure 6: Set-recovery values of thermally pre-treated and densified spruce and poplar specimens (n=10).

Regarding the thermal pretreatment conditions, the set-recovery values of the untreated and low-temperature thermally treated specimens were found to be quite high, and the densified specimens almost completely reached their initial dimensions before compression. However, with the increase in thermal pretreatment temperature and time, set-recovery decreased significantly, especially in poplar wood specimens. As in the other test results, it was determined that the thermal treatment temperature had a more significant effect on the set-recovery than the thermal treatment time (Figure 6). Compared to untreated specimens, set-recovery was reduced by up to 42 % and 67 %, respectively, in spruce and poplar specimens that were thermally pre-treated for 9 hours at 200 °C. It can be said that the break-down of the cross-links, which are responsible for the shape memory of wood, by thermal treatments is effective on the results (Inoue *et al.* 2008, Laine *et al.* 2013, Navi and Heger 2004, Pelit *et al.* 2014). In addition, the fact that the rate of internal stresses occurring in the wood during the pressing process is lower in thermally pre-treated specimens may have an effect on the set-recovery.

CONCLUSIONS

In the present study, the impact of thermal pretreatments on the dimensional change and humidity sensitivity of densified spruce and poplar wood specimens were analyzed. Spring-back and thickness swelling values increased due to the increase in compression ratio in both thermally pre-treated and untreated wood specimens. On the other hand, set-recovery was determined higher in specimens densified at 20 % compression ratio compared to 40 % compression ratio. However, the effect of compression ratio on set-recovery decreased as the thermal pretreatment temperature increased. Water absorption values increased significantly in all densified specimens (especially spruce specimens) depending on the compression ratio. After densification, the EMC of the untreated and thermally pre-treated specimens at low temperatures (140 ºC and 160 ºC) were significantly lower than the uncompressed samples. However, the EMC values of the thermally pre-treated specimens (uncompressed ve densified), especially at 200 ºC, were found to be similar. In addition, compression ratio had no significant effect on EMC values. After densification, the density of the spruce and poplar specimens increased up to 45 % and 46 %, respectively, with the increase in compression ratio.

Thermal pretreatments have a significant effect on the tested properties of densified wood specimens. Spring-back, set-recovery and thickness swelling decreased and dimensional stability of specimens (especially poplar) increased depending on the increase in thermal pretreatment temperature and time. Compared to the untreated specimens, spring-back decreased up to 10 % and 31 %, set-recovery up to 42 % and 67 %, and thickness swelling up to 40 % and 62 %, respectively, in the spruce and poplar specimens, which were thermally pre-treated at 200 °C for 9 h. On the other hand, EMC and water absorption values of spruce and poplar specimens decreased with increase in thermal pretreatment temperature and time. However, water absorption tends to increase in thermally pre-treated spruce samples at low temperatures. The mean EMC of thermally pre-treated spruce and poplar wood at 200 °C decreased by 30 % and 41 %, respectively. All test results showed that thermal treatment temperature has a more significant effect than thermal treatment time.

AUTHORSHIP CONTRIBUTIONS

H. P.: Conceptualization, Formal Analysis, Funding acquisition, Investigation, Methodology, Project administration, Resources, Supervision, Visualization, Writing – original draft, Writing – review & editing. **R. Y.:** Investigation, Resources, Writing – original draft, Writing – review & editing.

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