

EFFECT OF HEAT TREATMENT ON THE PHYSICAL AND MECHANICAL PROPERTIES OF COMPRESSION AND OPPOSITE WOOD OF BLACK PINE

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The effect of commercial heat treatment on physical and mechanical properties of compression wood (CW) and opposite wood (OW) of black pine (*Pinus nigra* Arnold) was investigated. Black pine logs containing CW were cut parallel to the pith and separated into CW and OW sections. A commercial heat treatment process was applied to pine lumber at 180 and 210 °C for 3 hours. Water absorption (WA), contact angle (CA), swelling, modulus of rupture (MOR), modulus of elasticity (MOE), and impact bending strength (IBS) were measured. The results showed that heat treatment decreased water absorption and swelling of the CW and OW of black pine. Heat treatment at 210 °C temperature decreased the longitudinal swelling of CW by 51.4%. Higher immersion time lowered the effect of heat treatment on the WA values. The CA values of the CW and OW increased due to heat treatment. Heat treatment reduced the MOR, MOE, and IBS values. The results indicated that MOR, MOE, and CA values were highly affected in the CW; on the other hand, the IBS value was highly affected in the OW by heat treatment compared to control groups. The results indicate that heat-stabilized CW can be used more widely and effectively in the forest products industry.

Keywords: Compression wood; Opposite wood; Water absorption; Swelling; Mechanical properties; Wettability

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INTRODUCTION

Reaction wood is an abnormal type of wood tissue formed in the living stems of both hardwoods and softwoods, apparently as a result of abnormal growing conditions. In softwood, it is termed compression wood (CW). The area opposite to the CW region is termed opposite wood (OW) (Timell 1986). The OW has similar properties to the normal wood. The CW is characterized by relatively wide, eccentric growth rings that contain an abnormally large proportion of latewood. Many of the anatomical, chemical, physical, and mechanical properties of reaction wood differ between normal and opposite wood (Timell 1986; Haygreen and Bowyer 1996). Density of the CW is commonly 30 to 40% greater than that of normal wood. Compression wood tracheids are about 30% shorter than normal. The S₂ layer of the CW tracheid has a larger microfibrillar angle compared to a normal wood tracheid. Therefore, CW shrinks and swells more in the longitudinal direction and less in the transverse direction than normal wood with changes in moisture content. In addition, CW contains about 10% less cellulose and 8 to 9% more lignin and hemicelluloses than normal wood (Bowyer *et al.* 2003).

The abnormal properties of CW make it an undesirable material for commercial lumber (Timell 1986), wood-based panels (Akbulut *et al.* 2004), and pulp and paper manufacture (Ban *et al.* 2004). Previous studies reported that the water resistance and mechanical properties of wood-based panels such as particleboard and fiberboard, when made from furnishes containing compression wood, was decreased compared with that of the panels made from normal wood (Akbulut *et al.* 2004; Lehmann and Geimer 1974; Coleman and Biblis 1977).

Heat treatment is one of the processes used to modify the properties of wood. Heat-treated wood is considered to be an eco-friendly alternative to chemically impregnated wood materials. During heat-treatment, a large number of chemical changes occur in the wood components, including significantly lower hemicelluloses content (Pavlo and Niemz 2003). The hemicelluloses degrade first (between 160 and 260 °C) due to their low molecular weight and their branching structure (Fengel and Wegener, 1984). Heat treatment decreases the equilibrium moisture content of wood (Nakano and Miyazaki 2003; Ates *et al.* 2009; Wang and Cooper 2005; Metsa-Kortelainen *et al.* 2006), improves its dimensional stability (Yildiz 2002; Bekhta and Niemz 2003; Gonzalez-Pena *et al.* 2004; Wang and Cooper 2005), but decreases mechanical properties (Ates *et al.* 2009; Kim *et al.* 1998; Kubojima *et al.* 2000; Bengtsson *et al.* 2002; Bekhta and Niemz 2003; Shi *et al.* 2007). Although wettability decreases (Petrisans *et al.* 2003; Follrich *et al.* 2006; Hakkou *et al.* 2005), the gluing process can be adapted for treated wood (Militz 2002).

In previous studies, the effect of heat treatment on the properties of normal wood was investigated in different wood species (Metsa-Kortelainen *et al.* 2006; Yildiz 2002; Shi *et al.* 2007; Akyildiz *et al.* 2007; Santos 2000). Akyildiz *et al.* (2009) investigated the technological and chemical properties of heat treated black pine wood. They found that the modulus of rupture (MOR) and the modulus of elasticity (MOE) values of black pine normal wood were decreased due to heat treatment. Metsa-Kortelainen *et al.* (2006) investigated the effect of heat treatment on water absorption of sapwood and heartwood of Scots pine and Norway spruce. To our knowledge, there is no information about the effect of heat treatment on wettability, swelling, water absorption, and mechanical properties of CW. The objective of this study was to determine the effect of heat treatment with commercial method on some physical and mechanical properties of CW and OW in black pine (*Pinus nigra* Arnold).

EXPERIMENTAL

Black pine (*Pinus nigra* Arnold) logs containing CW were obtained from Bahcekoy Forest Enterprises in Istanbul, Turkey. The logs were cut parallel to the pith and the CW and OW sections were separated. Each section was sawn into timbers with dimensions of 20 × 1000 mm (thickness×length) and different width. CW and OW sections were divided into 3 treatments groups. One of them was kept as an untreated control group and two others were thermally treated at 180 °C and 210 °C. The commercial Thermowood method patented by International Thermowood Association was applied to timbers in NOVA Forest Products Inc., Bolu, Turkey. The process was carried out in three main phases. Firstly, wood temperature was raised rapidly using heat and steam to a level around 100 °C. Thereafter the temperature was increased steadily to 130 °C, and the moisture content was reduced to nearly zero. Whenever high heat drying

was occurring, the temperature was increased to a level of 180 °C (first treatment group) and 210 °C (second treatment group) and held constant through 3 hours. In the final stage, the temperature was reduced to 50 to 60 °C by using a water spraying system. This process was continued until the moisture content of wood samples reached 4 to 6%.

Following the thermal treatment, small clear test specimens were prepared from the timbers of CW and OW according to the related standard method for determining the physical and mechanical properties. All of the samples were conditioned at 20±2 °C temperature and 65±5% relative humidity until they reached a constant weight.

The MOR, the MOE, the impact bending strength (IBS), and the swelling (Sw) measurements were carried out based on the ISO 3133 (1975), ISO 3349 (1975), ISO 3131 (1975), and ISO 4859-4860 (1982), respectively.

The water absorption (WA) was determined for 1 h, 2 h, 4 h, 6 h, 24 h, and 48 h water immersion times. The WA test was carried out on the same samples with the swelling. The samples firstly were dried to oven-dry moisture content at 103°C ±2 and weighed. They were then immersed in distilled water at 20 °C. At given interval times, samples were removed and weighted to determine weight gain. The WA values were calculated as a percentage of oven-dry weight. In addition to the WA values, the Water Repellent Effectiveness (WRE) values were calculated using following equation:

$$WRE (\%) = \frac{(WA \text{ of the control samples} - WA \text{ of the treated samples})}{WA \text{ of the control samples}} \times 100$$

Furthermore, Anti-Swell Effectiveness (ASE) was also calculated based on the swelling values by the following formula:

$$ASE (\%) = \frac{(Sw \text{ of the control samples} - Sw \text{ of the treated samples})}{Sw \text{ of the control samples}} \times 100$$

The contact angle (CA) values were obtained using a KSV Cam-101 Scientific Instrument (Helsinki, Finland). The CA is determined from the tangent with the sessile drop profile at the point of contact with the solid surface. After a 5 µL droplet of distilled water was placed on the sample surface, CA values were measured at 1-s time intervals up to 30-s total. CA values were obtained from the average of the measurements over the 30 s period.

A one-way ANOVA test was employed to determine the effect of heat treatment on the properties of CW and OW. The significant differences between the treatments were evaluated with Duncan's multiple comparison tests.

RESULTS AND DISCUSSION

Table 1 shows the WA and WRE values of CW and OW at different immersion times. The results indicated that, in both CW and OW, the thermal treatment significantly reduced ($p < 0.01$) the moisture uptake of wood samples. WA values of heat-treated groups at all immersion time were lower than those of the control group. This was expected because of the chemical decomposition of carbohydrates occurring at treatment temperature, which are responsible for the wood-water interactions. Especially in short

immersion times, the reducing effect of the thermal treatment on the WA was more remarkable compared to long immersion times (Figs. 1a and 1b). The WRE values were decreased considerably with increasing immersion time (Fig. 1c). It should also be noted that the WA values of heat-treated groups increased more rapidly than those of control groups, depending upon the immersion time (Figs. 1a and 1b). This result indicated that the heat treatment is more effective in reducing moisture uptake in short-duration exposure to water, but if the exposure time is prolonged, it loses its relative influence. This was probably due to a lowered fiber saturation point (FSP) of thermally treated wood. At the early stage of water soaking, water is located within the cell wall as bound water until the FSP is reached. After this point, water is located in the cell lumen as free water. The WRE values increased with increasing temperature of thermal treatment. These increases were found to be significant for OW ($p < 0.05$), while it was insignificant for CW. Furthermore, in OW, the effect of heat treatment at 180 °C temperature on the WA was not statistically significant at immersion times of 4h and up when compared to the control group.

Table 1. Water Absorption (WA) and Water Repellent Efficiency (WRE) of Thermal Treatment in Black Pine CW and OW

Wood Type	Group	Exposure Time (hours)					
		1-h	2-h	4-h	6-h	24-h	48-h
WA (%)							
CW	Control	63.9 (16.7) a	74.0 (15.2) a	77.0 (14.5) a	81.3 (15.5) a	95.5 (17.7) a	110.9 (19.1) a
	180	37.7 (15.6) b	49.9 (14.1) b	54.9 (12.1) b	62.5 (13.3) ab	79.0 (11.1) ab	91.6 (12.1) ab
	210	22.7 (7.7) b	35.6 (12.6) b	42.8 (15.2) b	49.2 (16.1) b	69.1 (15.7) b	84.8 (14.8) b
OW	Control	72.7 (9.3) a	80.5 (8.0) a	85.4 (11.8) a	89.4 (12.3) a	108.1 (15.3) c	125.2 (16.3) a
	180	50.6 (6.2) b	62.8 (4.2) b	73.4 (3.4) a	79.9 (3.4) a	104.3 (3.6) c	116.7 (6.8) a
	210	24.6 (8.7) c	38.4 (9.5) c	44.7 (10.7) b	50.5 (11.5) b	74.3 (9.4) d	90.1 (8.1) b
WRE (%)							
CW	180	41.0	32.6	28.7	23.1	17.3	17.4
	210	65.5	51.9	44.4	39.5	27.6	23.5
OW	180	30.4	22.0	14.1	10.6	3.5	6.8
	210	66.2	52.3	47.7	43.5	31.2	28.0

Groups with the same letters in column indicate there was no statistical difference ($p < 0.05$) between the samples according to Duncan's multiple range test. Values in parentheses are standard deviations. The values are an average of 10 samples.

CW had a lower WA value when compared to OW for all immersion times. However the lower WA of CW was found significant only at 24-h immersion time. CW has lower cellulose and higher lignin content compared to normal wood and opposite wood (Bowyer *et al.* 2003, Timell 1986). CW contained approximately 37% greater lignin and 43% lower cellulose than did normal and opposite wood (Tarmian and Azadfallah 2009). The lower WA of CW might be related to lower amounts of hygroscopic materials, *i.e.* cellulose and hemicelluloses, within its cell walls.

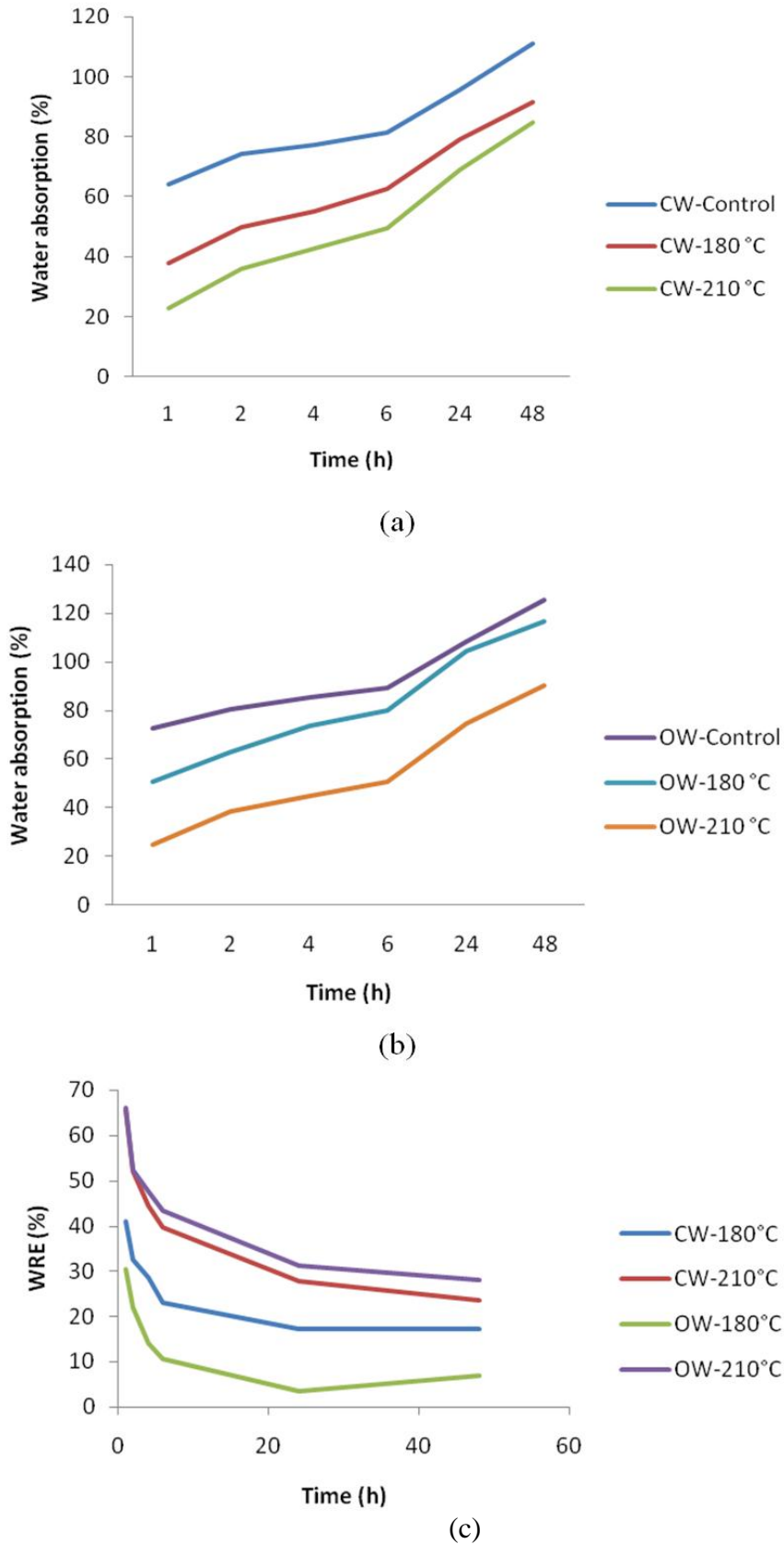


Fig. 1. WA and WRE values of CW and OW of black pine, depending on immersion time

Table 2 shows the radial, tangential, longitudinal, and volumetric swelling values of black pine CW and OW. The ASE values were also given in Table 2. As was expected, the control group of CW showed significantly higher longitudinal and lower transverse swelling than that of OW as a result of microfibril alignment in the S2 layer of the cell wall. The ANOVA results showed that the thermal treatment reduced the swelling significantly in both CW and OW. The treatment temperature also had significant effect on the swelling values. In general, swelling values decreased with increasing treatment temperature in both CW and OW. The volumetric ASE of thermal treatment at 180°C was 24.0% and 28.2% for CW and OW, respectively. When the treatment temperature was raised to 210 °C, the ASE also increased to 37.6% for CW and 43.7% for OW (Table 2). This was expected due to decreasing the hygroscopicity of wood with the thermal decomposition of holocellulose. It should be pointed out that the longitudinal swelling value, which is probably the most important quality problem of the CW, decreased remarkably with the thermal treatment when compared to the control samples. The treatment at 210 °C reduced the longitudinal swelling by 51.4% for CW (Table 2). Decreases of the longitudinal swelling values in the CW were higher than those of OW at both temperatures. Furthermore, the longitudinal swelling of CW and OW became more equal after the heat treatment. Thus, it could be concluded that the stability problems of timbers containing compression wood caused by its excessive and unequal shrinkage or swelling in longitudinal direction could be minimized with thermal modification.

Table 2. Swelling (Sw) Values and Antiswell Efficiency (ASE) of Thermal Treatment in Black Pine CW and OW

Wood Type	Group	Radial		Tangential		Longitudinal		Volumetric	
		Sw (%)	ASE (%)	Sw (%)	ASE (%)	Sw (%)	ASE (%)	Sw (%)	ASE (%)
CW	Control	3.75 (0.92) a	-	4.86 (1.00) a	-	1.77 (0.25) a	-	10.39 (1.62) a	-
	180	2.87 (0.44) b	23,5	3.84 (1.12) b	21,0	1.20 (0.30) b	32,2	7.90 (1.18) b	24,0
	210	2.44 (0.65) b	34,9	3.18 (0.85) b	34,6	0.86 (0.20) c	51,4	6.48 (0.99) c	37,6
OW	Control	4.48 (0.59) c	-	8.14 (0.44) c	-	1.26 (0.40) d	-	13.88 (0.48) d	-
	180	3.75 (1.33) c	16,3	5.19 (1.11) d	36,2	1.02 (0.41) de	19,0	9.96 (2.00) e	28,2
	210	2.38 (0.43) d	46,9	4.64 (0.49) d	43,0	0.78 (0.25) e	38,1	7.81 (0.94) f	43,7

Groups with same letters in column indicate that there was no statistical difference ($p < 0.05$) between the samples according to Duncan's multiple range test. Values in parentheses are standard deviations. The values are an average of 10 samples.

The CA values of the CW and OW are shown in Table 3. The CA values of the heat treated wood were found to be considerably higher than those of untreated wood. This means that the heat treatment significantly decreased the wettability of CW and OW. Since wood is a hygroscopic material, a cohesion force exists between wood and water in contact with it. Thermal decomposition of hygroscopic content, *i.e.* hemicellulose and cellulose, of wood with the thermal treatment may lead to reduced wettability of wood. Decreasing the effect of heat treatment on the wettability was also observed by Hakkou *et al.* (2005) and Kocaefe *et al.* (2008). Petrisans *et al.* (2003) suggested that one of the possible reasons for decrease of wettability could be the increase of cellulose crystallinity. The results also indicated that the CA values increased as the treatment temperature increased. The effect of the treatment temperature on the CA value was found to be significant ($p < 0.01$) in the CW, while it was not significant in the OW. It

should also be noted that there was no significant difference between the wood types in terms of the CA values.

Table 3. CA Values of Black Pine CW and OW

Wood Type	Group	CA (°)
CW	Control	39.6 (12.0) a
	180	78.0 (14.5) b
	210	89.8 (15.7) c
OW	Control	38.1 (11.5) a
	180	82.3 (16.5) b
	210	87.9 (9.3) b

Groups with same letters in a column indicate that there was no statistical difference ($p < 0.05$) between the samples according to Duncan's multiply range test. Values in parentheses are standard deviations. The values are average of 30 and 20 samples for CW and OW, respectively.

The MOR, the MOE, and the IBS values of the CW and OW are shown in Table 4. It was observed that the thermal treatment resulted in reduction in the strength of both CW and OW. The heat treatment at 210 °C reduced the MOR by 36.0% and 46.0% compared to the control group in CW and OW, respectively. Similarly the MOE was decreased by 19.1% for CW and 20.0% for OW compared to the control group by the thermal treatment at 210 °C. As for IBS, the percentage of reduction with the thermal treatment at 210 °C was 53.5% for the CW and 43.9% for the OW compared to the control group. Similar worsening effects of heat treatment were reported by several researchers in previous studies (Shi *et al.* 2007; Bengtsson *et al.* 2002; Yildiz 2002; Santos 2000). Vernois (2001) stated that the degree of decreases in the strength is very dependent on the wood species to be treated. It was also observed that the increasing treatment temperature reduced strength values slightly but these reductions were not found significant at a confidence level of 99% for both CW and OW (Table 4). The decreases in the strength properties with the thermal treatment can be explained by the rate of thermal degradation and losses of substance after treatment. The decrease in strength is mainly due to the depolymerization reactions of wood polymers (Kotilainen 2000; Wikberg and Maunu 2004).

Table 4. Results of ANOVA and Duncan's Mean Separation Tests for Mechanical Properties of Black Pine CW and OW

Wood Type	Group	MOR (N/mm ²)	MOE (N/mm ²)	IBS (J/cm ²)
CW	Control	61.4 (12.2) a	5606.8 (915.2) a	3.89 (1.06) a
	180	43.0 (6.4) b	4783.5 (1085.2) b	2.01 (0.53) b
	210	39.3 (7.3) b	4537.5 (977.0) b	1.81 (0.38) b
OW	Control	59.1 (11.3) c	5466.0 (903.9) a	3.12 (1.06) a
	180	37.3 (4.7) d	4730.1 (448.2) b	1.92 (0.62) b
	210	31.9 (10.5) d	4375.3 (870.1) b	1.75 (0.21) b

Groups with same letters in column indicate that there was no statistical difference ($p < 0.05$) between the samples according to Duncan's multiply range test. Values in parentheses are standard deviations. The values are average of 30 and 20 samples for CW and OW, respectively.

The primary reason for the strength loss is the degradation of hemicelluloses, which are less resistant to heat than cellulose and lignin. Changes in or loss of hemicelluloses play key roles in the strength properties of wood heated at high-temperatures (Hillis 1984). In addition, there was no significant difference observed between the strength of CW and OW except MOR. CW is higher in density than normal wood of the same species. Hygreen and Bowyer (1996) stated that, because of its higher density, it might be expected that CW also would have higher strength than normal wood. However, CW is about equal in strength to normal mature wood of the same species.

CONCLUSIONS

Heat treatment decreased the moisture uptake and the volumetric swelling of black pine CW and OW. Especially, in CW, heat treatment at 210 °C temperature decreased the longitudinal swelling by 51.4%. Higher immersion time lowered the impact of heat treatment on the WA values. CW had lower WA value compared to OW for all immersion times. The increasing treatment temperature also had a positive effect on the moisture uptake and dimensional stability of wood. The heat treatment reduced the wettability of CW and OW of black pine. The CA values increased as the treatment temperature increased. The effect of the applied temperature on the CA value was significant in the CW while it was not significant in the OW. The MOR, the MOE, and the impact bending strength decreased as the temperature increased. Effects of the heat treatment temperature on the MOR, MOE, and impact bending strength were not significant for both CW and OW. Wood type had a significant effect on the swelling values, while the WA, CA, and the strength values were not influenced considerably by the wood type. As a consequence, the present results imply that heat treatment can minimize the stability problem of CW caused by the excessive shrinkage or swelling in the longitudinal direction, and thus heat-stabilized CW can be used more widely and effectively in the forest products industry.

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