

A Comparative Study of Some of the Mechanical Properties of Pine Wood Heat Treated in Vacuum, Nitrogen, and Air Atmospheres

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The use of heat treatment to modify wood using different treatment heat transfer media, such as nitrogen, vegetable oil, steam, and vacuum, is preferable in many respects to other methods that use chemical treatments. However, the results of the heat treatment differ based on the heat transfer media that are used. In this study, the thermal modification of black pine wood in vacuum, nitrogen, and air atmospheres was studied. The heat treatments were conducted at temperatures of 180 °C, 200 °C, and 220 °C. After the heat treatments, the density, mass loss, modulus of rupture, modulus of elasticity, and impact of bending of heat-treated black pine wood were determined. The results indicated that the density, modulus of rupture, and impact of bending decreased as the temperature increased. In addition, the greatest decrease in the mechanical properties of the wood occurred in the test samples that were treated in air. The vacuum atmosphere was least harmful to the mechanical properties of the wood, and the differences in the mechanical properties of the wood that were heat treated in vacuum and nitrogen were unnoticeable.

Keywords: Heat treatment; Vacuum; Nitrogen; Mechanical properties

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INTRODUCTION

Heat treatment is an industrial method used to modify many materials, such as glass, metal, plastic, ceramic, and solid wood. Treating these materials at high temperatures improves some of their properties (URL1 2016). In the wood industry, high temperatures have been used extensively as an attractive method for modifying the characteristics of wood.

It has been reported that both the swelling and shrinkage of wood decrease after heat treatment (Esteves and Pereira 2009; Aydemir *et al.* 2011; Kaymakci and Akyıldız 2011). Other reports have indicated that after heat treating, the mechanical properties of wood decrease (Metsä-Kortelainen *et al.* 2006; Severo *et al.* 2012; Candelier *et al.* 2013b), its biological durability increases (Kamdem *et al.* 2002; Esteves and Pereira 2009; Candelier *et al.* 2013a), and its color parameters and lightness change (Bekhta and Niemz 2003; Ayata *et al.* 2017).

Steam, nitrogen, and oil are used as the heat transfer media in these processes (Wang and Cooper 2005). These heat transfer media and some other factors affect the results of the treatment.

The effects of these factors have been noted by researchers, *e.g.*, temperature, duration, the heat transfer media (Esteves and Pereira 2009), the species of wood (Aydemir *et al.* 2011; Araújo *et al.* 2016), the initial moisture content (Gaff and Gasparik 2013), the

density of the wood (Bal 2013a), the maturity of the wood (Bal and Bektaş 2012; Severo *et al.* 2012), and the extractives content (Metsä-Kortelainen *et al.* 2006; Bal 2013b).

Heat treatment in vacuum is a relatively new method, and some researchers have studied the effects of this method on the chemical, biological, physical, and mechanical properties of wood. For example, Gao *et al.* (2016) studied the degradation resistance and the physical, mechanical, and durability properties of heat-treated poplar wood in vacuum. The results showed that the vacuum heat treatment improved poplar wood's degradative resistance, dimensional stability, and durability, but it reduced the wood's mechanical strength. Jebrane *et al.* (2017) studied two heat treatment methods conducted in steam and vacuum, and they determined similar results for the wood's durability as well as its chemical, physical, and mechanical properties.

Wang *et al.* (2016) studied the effect of vacuum heat treatment on the chemical composition of larch wood, and they noted that the relative percentage contents of lignin and extractives increased after heat treatment, and they also noted that holocellulose, cellulose, and hemicelluloses decreased as a result of the thermo-vacuum treatment. Candelier *et al.* (2013b) investigated the effects of vacuum *versus* nitrogen on the mechanical properties of heat-treated beech wood, and the results showed that the mechanical properties were degraded less when the wood samples were treated in vacuum even though the mass losses were similar at the end of the heat treatment.

Mitchell (2007) studied the heat treatment of loblolly pine wood under air, oxygen and nitrogen, and reported that oxygen-dependent degradation was most visible for modulus of rupture and modulus of elasticity. Araújo *et al.* (2016) conducted an experiment that indicated that the heat treatment of wood under vacuum and nitrogen significantly lowered the equilibrium moisture content with only a small improvement in the dimensional stability.

Conversely, Allegretti *et al.* (2012) investigated the effects of vacuum treatment on the mechanical and biological durability of fir and spruce woods, and they determined that there were no significant decreases in their mechanical properties. These studies that have been mentioned revealed important information about the heat treatment of wood in vacuum. However, it must be recognized that several variables can affect the results of such studies, including the extent of the vacuum, the type of wood, the dimensions of the wood, the types of oven and heat transfer (conduction using metal plate and convection using air), and the operation of removing the test samples from the oven at the end of the test and holding the test samples until they cool.

Therefore, the previous studies clearly indicate that different researchers reported different results concerning the effects of the heat treatment of woods in vacuum and nitrogen. The differences could have resulted from using the heat transfer method in a vacuum atmosphere by using conduction and convection.

It is well known that heat transfer using conduction is a more effective method than convection due to the physical contact. However, there has not been sufficient research of the effect of the vacuum atmosphere on the heat treatment of wood. Therefore, in this study, mass loss, density, and some of the mechanical properties of heat-treated pine wood in vacuum, nitrogen, and air atmospheres were investigated using the convection heat transfer method.

EXPERIMENTAL

Materials

For this study, black pine (*Pinus nigra*) logs were obtained from a lumberyard in Kahramanmaraş, Turkey. The logs were divided into planks as shown in Fig. 1. The planks from sap wood were selected. The planks were dried for two months in an open shed, and then the planks were divided into slats with the dimensions of 2 cm× 2 cm× 120 cm (width, height, and length). Flawless slats without cracks and knots were chosen. The samples were prepared from these slats to be tested for their mechanical properties. One control and three test groups were prepared from the same slats. Twenty test samples were prepared for each mechanical test with the width, thickness, and length dimensions of 2 cm, 2 cm, and 30 cm, respectively.

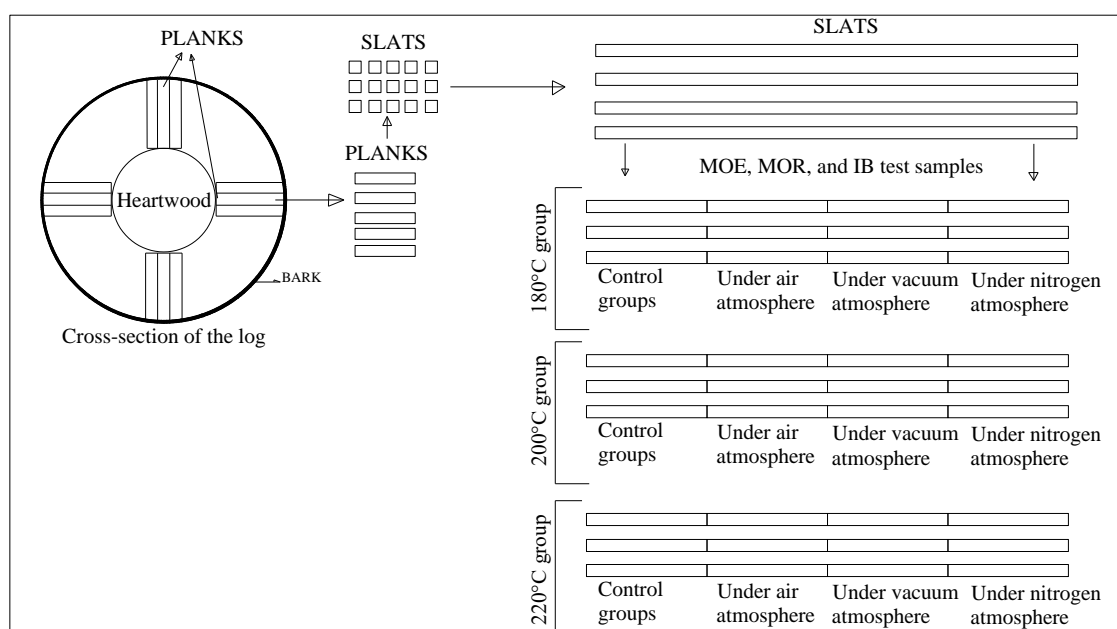


Fig. 1. Preparation of the test samples

Methods

After the test samples were prepared, they were oven-dried at 103 °C until they reached a constant weight. Then, the samples were weighed and their dimensions were measured to determine their mass loss and their oven-dried density. Next, the samples were heat treated in vacuum, nitrogen, and air atmospheres. Heat treatment under vacuum was applied at 500 mBar using a vacuum oven (JSR-JSVO 60T; JS Research Inc., Gongju, Republic of Korea) and a vacuum pump (Value vdr-16; Zhejiang Value Mechanical & Electrical Products Co., Ltd., Zhejiang, China). Heat treatment in nitrogen was conducted in the same oven by adding nitrogen gas. During the treatment, the door of the oven was closed, and air was removed from the oven by using a vacuum, and nitrogen gas was introduced into the oven. During the treatment, the door to the oven was always closed. Heat treatment in air was conducted in the same oven, but no vacuum and no nitrogen were used. Heating time of the oven was 30, 35, 40 ±2 minutes for temperatures of 180, 200 and 220 °C, respectively, and heat treatment was 150 minutes for each group.

After the tests, all of the samples were weighed to determine their mass losses and densities, and they were quickly placed in nylon bags to prevent contact with oxygen until they were cooled. Lastly, the samples to be used to determine the mechanical properties of the wood were stored in a climatic chamber at 20 °C and 65% relative humidity for five weeks, after which the tests were conducted.

Density (D), modulus of rupture (MOR), modulus of elasticity (MOE), and impact bending (IB) were tested according to Turkish standards TS 2471 (1976), TS 2474 (1976), TS 2478 (1976), and TS 2477 (1976), respectively. The MOR and MOE were determined using a universal testing machine (ALŞA Laboratuvar Cihazları, İstanbul, Turkey) (50 kN) at a speed of 2 mm/min, and IB was determined using a pendulum-impact testing machine (ALŞA Laboratuvar Cihazları, İstanbul, Turkey).

After the tests, the data were evaluated with the SPSS (IBM, SPSS 13, New York, USA) program using a one-way analysis of variance (ANOVA) and Duncan test.

RESULTS AND DISCUSSION

The arithmetic means, standard deviations, percentage decreases, and ANOVA results are shown in Table 1. The ANOVA results are shown at the bottom of the table. The analysis of the mass loss (ML) data given in the table clearly indicates that the ML values increased as the temperature increased. Mass loss occurs when the thermal degradation reaction of the wood occurs. It has been stated that ML is the indicator value of the heat treatment (Esteves and Pereira 2009; Candelier *et al.* 2013b). As the values of ML increase, the chemical constituents change (Aydemir *et al.* 2011; Severo *et al.* 2012; Wang *et al.* 2016), the physical properties are modified (Bal 2014; Gao *et al.* 2016), the biological durability increases (Candelier *et al.* 2013a), and the mechanical properties decrease (Candelier *et al.* 2013b; Jebrane *et al.* 2017). The highest ML value was measured in the air atmosphere group. The differences in the ML values between the vacuum and nitrogen groups were not significant ($p > 0.05$) for any of the temperature groups. According to these ML results, it was apparent that there was less effect on the wood in vacuum and nitrogen atmospheres than in the air atmosphere. However, there were no significant differences in the ML values for the treatments in vacuum and the nitrogen atmosphere. In a similar study, Candelier *et al.* (2013a, 2014) reported that heat treatment under nitrogen presented higher Klason lignin and carbon contents and lower hemicelluloses and neutral monosaccharide contents than wood that was heat treated in vacuum, but there were no differences in the ML between the two treatments. In another study, the ML of the low-density wood (bracatinka) under a vacuum atmosphere was higher than that in a nitrogen atmosphere. The ML of the high-density wood (cumaru) in a vacuum atmosphere was lower than that of the wood in a nitrogen atmosphere (Araújo *et al.* 2016).

The D values of the test samples treated in air, vacuum, and nitrogen decreased compared to the control group. However, there were no significant differences among the groups. The MOE and MOR values of the three control groups were 8343 N/mm² and 93 N/mm², 8597 N/mm² and 96 N/mm², and 9031 N/mm² and 101 N/mm², respectively. These groups were not treated, but the MOE and MOR values differed from each other. The reason for this is visible *via* reading the table from left to right, which shows that the D values were 492 kg/m³, 505 kg/m³, and 515 kg/m³, respectively. The reason for this was that the test samples were prepared from different slats (Fig. 1). When the table is read

from top to bottom, it is apparent that the D values were as expected. The MOE, MOR, and IB values of the control groups were higher than those of the test groups that were heat-treated in air, vacuum, and nitrogen.

Table 1. ML, D, MOE, MOR, IB, and ANOVA Test Results

	180 °C					200 °C					220 °C					
	ML	D	MOE	MOR	IB	ML	D	MOE	MOR	IB	ML	D	MOE	MOR	IB	
	(%)	($\frac{\text{kg}}{\text{m}^3}$)	($\frac{\text{N}}{\text{mm}^2}$)	($\frac{\text{kgm}}{\text{cm}^2}$)	(%)	($\frac{\text{kg}}{\text{m}^3}$)	($\frac{\text{N}}{\text{mm}^2}$)	($\frac{\text{kgm}}{\text{cm}^2}$)	(%)	($\frac{\text{kg}}{\text{m}^3}$)	($\frac{\text{N}}{\text{mm}^2}$)	($\frac{\text{kgm}}{\text{cm}^2}$)	(%)	($\frac{\text{kg}}{\text{m}^3}$)	($\frac{\text{N}}{\text{mm}^2}$)	($\frac{\text{kgm}}{\text{cm}^2}$)
Control group																
x	-	492	8343	93	0.45	-	505	8597	96A	0.45	-	515	9031A	101A	0.46A	
ss	-	26	880	9	0.09	-	36	948	9	0.1	-	43	1418	12	0.085	
Air Group																
x	1.2A	485	8117	89	0.42	1.6A	484	7719	82B	0.37	3.4A	499	7959B	77C	0.35B	
ss	0.44	32	1378	13	0.14	0.3	36	1152	11	0.12	0.6	41	1027	14	0.11	
pd		1.4	2.7	4.6	7.3		4.0	10.2	14.6	17.6		3.0	11.9	23.8	23.9	
Vacuum group																
x	0.9B	490	8315	92	0.41	1.3B	492	8360	88AB	0.42	2.9B	503	8660AB	88B	0.40AB	
ss	0.3	29	1278	11	0.12	0.2	31	1698	16	0.12	1.2	40	1145	13	0.12	
pd		0.3	0.3	0.8	8.2		2.4	2.8	8.3	8.4		2.3	4.1	12.9	13.0	
Nitrogen group																
x	0.8B	489	8101	91	0.42	1.3B	488	8029	85AB	0.41	2.9B	501	8441AB	81BC	0.38AB	
ss	0.2	28	1235	9	0.12	0.2	37	1676	11	0.11	0.7	41	1054	10	0.09	
pd		0.5	2.9	2.7	6.2		3.2	6.6	11.5	10.4		2.8	6.5	19.8	17.4	
ANOVA test results																
	**	NS	NS	NS	NS	***	NS	NS	*	NS	**	NS	*	***	*	

x: Arithmetic mean, ss: standard deviation, pd: percentage decrease compared to control group

There were no significant differences between the control and test groups in the 180 °C groups. In the 200 °C groups, only the MOR values were different. In the 220 °C groups, the MOE, MOR, and IB values were significantly different from each other. These data indicate that the effects of heat on the treatment media became noticeable as the temperature increased. In addition, in the 200 °C and 220 °C groups, the MOR values were more affected than the MOE values. All of the percentage decreases of the MOR values were greater than those of the MOE values. Concerning the MOE and MOR values of the heat-treated beech wood in vacuum and nitrogen, similar results were reported by Candelier *et al.* (2013b). In previous studies, some researchers have reported that MOR is more affected than MOE by the heat treatment (Esteves and Pereira 2009; Bal 2014; Gao *et al.* 2016). The largest decreases occurred in the IB values. In previous studies, it was reported that IB was the most affected mechanical property (Jimenez *et al.* 2011; Bal 2013d). When the mechanical properties data in Table 1 were analyzed, in general, heat treatment affected the mechanical properties. The most affected groups were the groups treated in the air atmosphere. There were some differences among the groups for the values of ML, MOE, and MOR for the samples treated in vacuum and nitrogen. Contrary to some previous studies, the present study showed that the differences between heat treatments in vacuum and nitrogen atmospheres were insignificant. In addition, the present study verifies the results of Candelier *et al.* (2013b) by showing that there are no significant differences between heat treatments in vacuum and nitrogen atmospheres.

CONCLUSIONS

1. All mechanical properties that were tested were affected by the heat treatment applied in vacuum, air, and nitrogen atmospheres. The most affected mechanical property was impact bending.
2. The mass loss increased as the temperature increased. The higher mass loss (3.44%) was measured at the end of treatment applied in the air atmosphere at temperature of 220 °C. There were no differences in the mass losses between the heat treatment applied in vacuum and nitrogen atmosphere, regardless of the temperature.
3. The D, MOE, MOR, and IB values for the test samples appeared to be less affected by treatment in vacuum than by treatment under air and nitrogen. The differences in these properties in the samples that were heat treated in vacuum and nitrogen were insignificant.
4. Considering the results of previous studies and comparing the data obtained in this study to the control group, it is apparent that the mechanical properties were affected when the samples were treated in a vacuum irrespective of the heat transfer method, *i.e.*, conduction or convection.

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