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### WOOD-WATER RELATIONSHIPS AND BIOLOGICAL DURABILITY OF HEAT-TREATED TAURUS FIR WOOD

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#### Abstract:

In this study, Taurus Fir (Abies cilicica) wood was treated with hot air at temperatures of 160, 190 and 220°C for 2h durations. After heat treatment, some physical properties and wood-water relationships were evaluated, such as mass loss, density, tangential swelling, radial swelling, volumetric swelling, swelling anisotropy, and fiber saturation point. In addition, the biological durability of Taurus Fir wood was tested in the laboratory with the soil contact test, and determined weight loss. The relationships between mass loss and some of the tested properties were determined using regression analysis.

The results showed that heat treatment at 220°C had significant effects on the physical properties and the biological durability of Taurus Fir wood. Further, it was determined that there was a linear-negative correlation between weight loss and mass loss.

Key words: heat treatment; mass loss; physical properties; Taurus Fir wood.

#### INTRODUCTION

Heat treatment of wood is preferred in many areas because it is an environmentally-friendly method without chemical agents (Garcia et al. 2012; Cademartori et al. 2013). But, after the heat treatment process, wood becomes more fragile due to the loss of amorphous polysaccharides (Esteves and Pereira 2009), and its mechanical properties decrease (Jamsa and Viitaniemi 2001; Esteves and Pereira 2009; Bal and Bektas 2013). Conversely, the dimensional stability and biological durability of heat-treated wood are modified in positive ways. Some previous studies have noted that heat treatment changes the color of wood (Bekhta and Niemz 2003; Welzbacher et al. 2007; Gunduz and Aydemir 2009; Olărescu et al. 2014), reduces the equilibrium moisture content (Esteves et al. 2007; Gunduz and Aydemir 2009; Calonego et al. 2012), reduces shrinking and swelling (Esteves et al. 2007; Gurău et al. 2012; Dubey et al. 2012; Calonego et al. 2012; Câmpean et al. 2011; Bal 2013a, 2013b), and improves the biological durability of the wood (Kamdem et al. 2002; Edlund and Jermer 2004; Welzbacher et al. 2007; Esteves and Pereira 2009; Beldean and Timar 2010; Calonego et al. 2010).

Several factors affect the results of heat treatment, including temperature, duration, the heat transfer medium (Esteves and Pereira 2009), the species of wood, the density of the wood (Bal 2013a), the maturity of the wood (Bal and Bektaş 2012), compression (Gaff and Gasparik 2013), and the extractive content of the wood (Bal 2014).

Hemicelluloses, which are one of the main components of wood, degrade first during the heattreatment process. As the components of the wood degrade, the remaining mass decreases. Mass loss is related to the intensity of the heat treatment. In the literature, there are many studies related to heat-treated wood, but only a few researchers have studied the relationship between mass loss and the properties of treated wood. For example, Esteves et al (2014) compared heat-treated sapwood and heartwood from Pinus pinaster and showed relationships between mass loss and some properties, such as equilibrium moisture content, swelling, modulus of rupture, and modulus of elasticity. Borrega and Karenlampi (2008) studied the heat treatment of spruce wood at various temperatures and reported that the bending properties of the wood decreased as the mass loss increased. Gündüz and Aydemir (2009) investigated heat-treated hornbeam wood and showed the effects of mass loss on oven-dry and air-dry densities. In another study, Pena et al. (2009) conducted experiments using heat-treated beech, Scots pine, and Norway spruce woods, and they noted relationships between mass loss and chemical composition.

#### **OBJECTIVES**

Some species of wood are refractory to preservative treatment. The impregnation processes of such species with chemical wood preservatives are difficult. Taurus Fir (Abies cilicica) wood is very vulnerable biologically. It is nonresistant to decay (Bozkurt and Erdin 1997; Clausen 2010). Without www.proligno.ro

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appropriate treatment, Taurus Fir wood should not be use in a humid environment. In addition, to the best of the author's knowledge, there have been no published studies concerning heat-treated Taurus fir (Abies cilicica) wood. Thus, the main aims of this study are to investigate some physical properties and wood-water relationships of Taurus Fir wood and to determine the relationships between mass loss and some other properties of heat-treated fir wood.

#### MATERIALS AND METHODS **Preparation of test samples**

For this study, Taurus Fir lumber was obtained from a lumberyard in Kahramanmaras City, Turkey. On the cross section of the lumber, the annual ring direction was perpendicular to one surface of the lumber and was parallel to the other side of the lumber. The lumber was cut into planks. Some sticks from these planks were cut with width, height, and length dimensions of 25, 25 and 1000mm, respectively. The sticks were dried for approximately one month in a room at ambient conditions. Then, samples were prepared to test the physical properties of the wood, as can be seen in Fig. 1. Twenty-five samples with width, height, and length dimensions of 20, 20 and 30mm, respectively, were prepared for the physical tests. The test samples that were used to determine the biological durability of the wood had width, height, and length dimensions of 5, 10 and 100mm, respectively. Equal numbers of test samples were prepared from the same sticks to set up homogeneous test groups. Twenty samples were prepared for biological durability test. The test samples were dried in an oven at a temperature of 103±3°C until their moisture content was 0%, and, then, they were weighed.



Fig. 1. Sample preparation method.

#### Heat treatment

Heat treatment was conducted at temperatures of 160, 190 and 220 C for 2 in air and at atmospheric pressure. After heat treatment, the weights and dimensions of the test samples were measured again in order to obtain their oven-dried density. Then, the samples were immersed in distilled water for four weeks. At the end of this period, the samples were removed from the water, and the surfaces of the samples were wiped with a dry cloth. The dimensions and weights of the samples were measured to determine their physical properties.

#### METHOD

Moisture content, density values, linear swelling (tangential and radial), and volumetric swelling were determined according to Turkish standards TS 2471, TS 2472, TS 4084 and TS 4086, respectively. Mass loss (ML) and fiber saturation point (FSP) were calculated using equations (1) and (2), respectively:

$$ML = ((M_1 - M_2) / M_1) \times 100$$
<sup>(1)</sup>

where: ML is the mass loss, M<sub>1</sub> is the mass of the sample after being dried in an oven at 103°C before heat treatment, and  $M_2$  is the mass of the sample after heat treatment.

$$FSP = (VS/D) \times 100$$

where: FSP is the fiber saturation point, VS is volumetric swelling, and D is oven-dried density.

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The biological durability test was conducted according to the procedures defined in TS ENV 807. The test samples after heat treatment were placed in unsterile forest soil in a plastic box. The 80mm section of the test samples was inserted into soil and exposed for 16 weeks. After this period, the samples were removed from the soil, washed with water, dried in an oven, and weighed to determine their weight loss, which was calculated with equation (3):

$$WL = (W_1 - W_2)/W_1 \times 100,$$

(3)

where: WL is the weight loss,  $W_1$  is the oven-dried weight of the sample, and  $W_2$  is the weight of the sample after the biological durability test.

The findings were analyzed using one-way ANOVA (P=0.05) from the SPSS statistical software program, and significant differences were determined by the Tukey HSD (Honestly Significant Difference) comparison test ( $\alpha$ =0.05).

#### **RESULTS AND DISCUSSION**

Table 1 provides the findings of oven-dry density (D), mass loss (ML), equilibrium moisture content (EMC), tangential swelling (TS), radial swelling (RS), swelling anisotropy (TS/RS), volumetric swelling (VS), and fiber saturation point (FSP). The table also provides the ANOVA test results (significance level) and the differences between groups (Tukey HSD classification). The findings indicate that the effects of heat treatment on the physical properties were statistically significant (p < 0.001), with the exception of density. ML values were 0.5, 1.1 and 4.4% for the heat-treatment temperatures 160, 190 and 220°C, respectively. When the temperature increased, mass of the sample decreased. Similar results have been reported by other researchers concerning the relationship between ML and heat-treatment temperature (Esteves *et al.* 2007; Pena *et al.* 2009; Kocaefe *et al.* 2008; Borrega and Karenlampi 2008; Bal and Bektaş 2012; Kamperidou *et al.* 2013; Bal 2013a, Bal 2013b).

As cited before, EMC and FSP are the important dimensional and mechanical properties. Swelling and shrinkage occur between 0% and approximately 30% (FSP) moisture content. The shape and mechanical properties of wood vary in this interval. Table 2 shows that, as the decreases in the EMC, compared to that of the control samples were 10.6, 18.6, and 36% for heat-treatment temperatures of 160, 190 and 220°C, respectively. As the EMC and the FSP decrease, the wood becomes more stable than its original form. This is the most important issue for the use of wood and wood products. Similar results were found for EMC (Gurău *et al.* 2012; Kamperidou *et al.* 2013; Bal 2014) and FSP by other researchers (Esteves *et al.* 2007; Almeida *et al.* 2009; Bal 2014).

Table 1

		Physical p	roperties o	f heat-treat	ted Taurus	Fir wood		
Properties	D	ML	EMC	TS	RS	TS/RS	VS	FSP
Groups	kg/m <sup>3</sup>	%	%	%	%	-	%	%
Control	396	-	10.9a <sup>1</sup>	7.5a	2.8a	2.8a	10.2a	26.8a
	(52)	-	$(0.5)^2$	(1.1)	(0.6)	(0.5)	(1.4)	(2.8)
160°C	396	0.5a	9.7b	7.2a	2.7a	2.7a	9.9a	25.3ab
	(46)	(0.1)	(0.5)	(1.4)	(0.6)	(0.4)	(1.9)	(3.7)
190°C	394	1.1b	8.9c	6.8a	2.6a	2.6ab	9.4a	23.9bc
	(49)	(0.2)	(0.5)	(1.4)	(0.4)	(0.6)	(1.6)	(4.2)
220°C	393	4.4c	7.0d	4.8b	2.1b	2.3b	6.9b	17.3c
	(37)	(0.9)	(0.5)	(1.1)	(0.5)	(0.3)	(1.6)	(3.4)
Significance Level	NS	***	***	***	***	**	***	***

Level 'Means followed by the same letter are not significantly different according to Tukey HSD test.

<sup>2</sup>Standard deviations are shown in parentheses.

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Table 2

The findings of TS, RS, VS, and TS/RS (swelling anisotropy) are given in Table 1, and Table 2 shows their percentage decreases after heat treatment. The effect of heat treatment on the swelling values was statistically significant (p<0.001). But, compared to control samples and according to Tukey HSD comparison test results, there were no significant differences between the wood in the control group, the wood treated at 160°C, and the wood treated at 190°C. However, for wood treated at 220°C, the percentage decreases of the values of TS, RS and VS were 35, 24 and 32%, respectively. It is well known that, when wood is heated, higher temperatures cause greater decreases in the swelling percentage. Similar results were reported by Welzbacher *et al.* (2007), Almeida *et al.* (2009), Kaymakçı and Akyıldız (2011) Gurău *et al.* (2012); Calonego *et al.* (2012).

In general, the TS values of solid wood are higher than the RS values. As a result of this difference, swelling anisotropy occurs. Swelling anisotropy causes some irregularities in the usage areas of solid wood when the moisture content of the environment changes. Table 2 shows that the TS values were higher than the RS values in the present work. Anisotropy values were 2.8, 2.7, 2.6 and 2.3 for the control, 160, 190 and 220°C groups, respectively. Even if the percentage decreases of TS were higher than those of RS, anisotropy still remained. Other researchers have reported similar results concerning this issue (Esteves *et al.* 2007; Câmpean *et al.* 2011; Calonego *et al.* 2012; Dubey *et al.* 2012; Bal 2013a, Bal 2013b).

	EMC	TS <sup>1</sup>	RS¹	TS/RS	VS <sup>1</sup>	FSP
160°C	0.5	10.6	2.3	1.7	3.0	5.5
190°C	9.4	18.6	6.4	4.0	8.4	11.0
220°C	28.8	36.0	24.6	15.7	32.5	35.4

#### Percentage decreases of EMC, TS, RS, TS/RS, VS, and FSP after treatment

Percentage decrease of swelling values has the same meaning as anti-swelling efficiency (ASE)

Fig. 2 shows the relationship between ML and VS. In addition, the regression equation and the coefficient of determination  $(R^2)$  are given. The figure shows that there was a linear-negative relationship between ML and VS. The  $R^2$  value was 0.51, and this shows high relationship. In a similar study, Welzbacher et al. (2007) calculated the relationship between the ML and VS of heat-treated Norway spruce. Also, Esteves et al. (2007) showed the relationship between ML and RS in their study of heat-treated Eucalyptus wood. This issue was explained as follows by Kocaefe et al. (2008), the structure of the wood changes due to the decomposition of hemicelluloses, the crystallization of cellulose, and the ramification of lignin. In another study, Pena et al. (2009) determined the chemical composition of heat-treated beech, Scots pine, and Norway spruce wood and showed that the holocellulose content decreased and the lignin content increased. In addition, Esteves and Pereira (2009) noted that the dimensional stability increased due to the cross linking in lignin and due to the destruction of several hydroxyl groups. Also, the reduction of dimensional stability and hygroscopicity of heat-treated wood was explained by Tjeerdsma et al. (1998) as follows, i.e., the cellulose microfibrils are surrounded by a firm and more inelastic network due to increased cross linking within the lignin complex. The cellulose microfibrils have a decreased possibility of expansion, and, therefore, they have less capacity to adsorb the water between the cellulose chains, thereby impeding the swelling of the cell walls.



Fig. 2. Relationship between mass loss and volumetric swelling.

The relationships between ML and EMC and between ML and FSP are shown, and the regression equation and the coefficient of determination also are given in Fig. 3A and Fig. 3B, respectively. The linear-negative relationship between those values was determined. The  $R^2$  values were 0.84 and 0.53, respectively, indicating significant relationships. EMC and FSP decreased as the mass decreased. Concerning this issue in their study, Mitsui *et al.* (2006) monitored the hydroxyl groups in heat-treated Sitca spruce wood, and they stated that there was an obvious correlation between the decrease in the mass of wood and the decrease of hydroxyl groups in the cellulose during heat treatment.



Fig. 3. Relationships between ML and EMC and FSP A. - ML and EMC; B. - ML and FSP.

The findings of the laboratory soil contact test using heat-treated samples and the control group are listed in Table 3. After 16 weeks of exposure to the soil in the contact test, the WL percentage of the control group was higher than that of the heat-treated groups. The lowest WL value was measured in the 220°C group. The WL percentage decreased as the temperature of treatment and ML increased. In addition, Fig. 4 shows that there was a linear-negative relationship between ML and WL. The  $R^2$  value was guite high (0.70). The biological durability of the heat-treated wood was determined experimentally in previous studies using soil block tests, soil contact tests, and field tests. In general, the researchers concluded that heat treatment did not increase the biological durability of wood in contact with soil. Conversely, Calonego et al. (2010) conducted soil-block tests of heat-treated eucalyptus wood using Picnoporus sanguineus and reported that there was a significant reduction in the WL for temperatures in the range of 180-220°C. Edlund and Jermer (2004) experimentally determined the durability of heat-treated pine wood using the soil-contact test (terrestrial microcosm) in different types of soils, and they noted that heat-treated wood performed very well in all soils and that the mass loss are lower than that of preservative-treated wood. In another study, Esteves et al. (2014) determined the durability of heat-treated sapwood and heartwood of *Pinus pinaster*, and they reported significant increases in the durability of both types of wood at the higher temperature of 200°C. In addition, Sailer et al. (2000) compared some properties and the durability of pine and spruce

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wood heat-treated in hot oil and hot air at various temperatures, and they noted that the wood treated with hot oil had less mass loss than the wood that was treated in hot air.

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Table 3

Results of the son-contact test								
Control		160 °C		190 °C		220 °C		
$ML^1$	WL <sup>2</sup>	ML	WL	ML	WL	ML	WL	
-	7.82 <b>a</b> <sup>3</sup>	0.73	6.44 <b>b</b>	1.02	5.45 <b>b</b>	3.68	1.26 <b>c</b>	
-	(2.34)	(0.16)	(1.37)	(0.26)	(1.62)	(0.58)	(0.48)	

<sup>1</sup><sub>2</sub>ML is the mass loss after heat treatment process

<sup>2</sup>WL is the weight loss after soil contact test

<sup>3</sup>Means followed by the same letter are not significantly different according to Tukey HSD test



Fig. 4. Relationship between ML and WL.

#### CONCLUSIONS

In this study, some physical properties of Taurus Fir wood were investigated, including mass loss, density, tangential swelling, radial swelling, volumetric swelling, swelling anisotropy, and fiber saturation point. These data were used to determine the relationships between mass loss and the other properties of heat-treated fir wood. The following results were obtained:

- The equilibrium moisture content of heat-treated fir wood decreased significantly for all of the treatment temperatures. Tangential and radial swelling decreased significantly only for treatment at 220°C.
- Swelling anisotropy decreased as the treatment temperature increased; this was especially significant for the treatment temperature of 220°C, but anisotropy still existed between tangential swelling and radial swelling.
- There was a linear-negative relationship between mass loss and the other properties of the heattreated wood, such as volumetric swelling, equilibrium moisture content, and fiber saturation point. In addition, the same relationship was determined for biological durability. These are important properties of wood that affect its useful lifetime.

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