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Characteristics of heat-treated Turkish pine and fir wood after ThermoWood processing

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Abstract: *The Finnish wood heat treatment technology, ThermoWood, was recently introduced to Turkey. Data about the mechanical and physical properties of Turkish wood species are important for industry and academia. In this study, two industrially important Turkish wood species, pine (Pinus nigra Arnold.) and fir (Abies bornmülleriana Mattf.) were heat-treated using the ThermoWood process. Pine and fir samples were thermally modified for 2 hr at 212 and 190°C, respectively. The modulus of rupture (MOR), modulus of elasticity in bending (MOE), impact bending strength (IBS), and compression strength (CS), in addition to swelling (Sw) and shrinkage (Sh) of thermally-modified wood were examined. The results indicate that the heat treatment method clearly decreased the MOR, MOE and IBS of pine and fir. However, a small increase was observed for CS values of heat treated wood species. The most affected mechanical properties were MOR and IBS for both pine and fir. The reduction in MOE was smaller than that in MOR and IBS. Volumetric shrinkage and swelling of these species were also improved by approximately half. In addition, the changes in the mechanical and physical properties studied in pine were larger than that of fir.*

Key words: *Heat treatment, Mechanical properties, Physical properties, Pine, Fir*
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Introduction

Increasing environmental pressure appeared in the last years in many European countries leading to important changes in the field of wood preservation. Heat treatment of wood has been considered as an effective method to modify wood without the use of any toxic chemicals. Stamm's (1964) is considered a starting point for heat treatment theory and recently heat treatment was systematically researched and industrially applied in some European countries. There are different wood heat treatment processes that are in pilot plant or commercial phases: The Finnish process (ThermoWood) uses steam, the Dutch (Plato Wood) uses a combination of steam and heated air, the French (Rectification) an inert gas and the German (OHT) heated oil (Rapp, 2001). Thermo Wood, used in this study, is an environmentally friendly material. Because the manufacturing process of ThermoWood is based only on the use of high temperature and steam, any harmful substances do not leach to the ground from products and the emissions of it in the surrounding air are very low (Finnish ThermoWood Handbook, 2003).

Heat-treated wood has a growing market in outdoor applications, like exterior cladding, window and door joinery, garden furniture, and decking and indoor applications such as flooring, paneling, kitchen furnishing, and interiors of bathrooms and saunas (Viitaniemi, 2000). The mechanical properties and physical properties of heat treated wood effect the performance of wood products in these applications. The mechanical properties of wood such as modulus of rupture (MOR), modulus of elasticity (MOE), compression strength (CS) and impact bending strength (IBS) are important for load-bearing constructions. Dimensional stability of wood is important factor especially in high humidity condition like saunas, bathrooms and garden furniture. Thus, these properties have been concerned in several studies.

Studies on heat treatment indicated that heat treatment of wood improves wood physical properties by reducing hygroscopicity and improving dimensional stability (Viitaniemi, 1997; Santos, 2000). Studies also showed that the resistance of wood against decay fungal attack can be improved with heat treatment (Kandem *et al.*, 2002). However, Jamsa and Viitaniemi (2001) indicated that heat-treated wood is not suitable material for ground contact applications. Also resistance of heat treated wood to termite attack varies depending on termite types (Finnish Thermo Wood Handbook, 2003). Beyond these desirable changes, heat treatment also causes unfavorable effects such as diminished mechanical properties (Esteves *et al.*, 2007a,b; Shi *et al.*, 2007; Korkut, 2008a).

Chemical modification of wood components occurring during heat treatment is mainly responsible for these new properties. The changes in the properties are related to the depolymerisation reaction of wood polymers, especially hemicelluloses, which are less stable to heat than cellulose and lignin (Hillis, 1984). Kandem *et al.* (2002)

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and Nuopponen (2005) reported that heat treated wood exhibit a higher lignin content and lower acid number compared to the untreated woods indicating to the degradation of some hemicellulose and extractives compounds. According to Hodgin and Lee (2002) wood becomes more brittle with heat treatment and Kubojima *et al.* (2000) noted that the main factors contributing to the reduction of absorbed energy in impact bending are viscosity and plasticity, rather than elasticity.

The degree of the change in the mechanical properties of wood during heat treatment is dependant on the process type, the maximum temperature reached in the process, and the holding time at that temperature (Shi *et al.*, 2007). In addition to, in all cases of thermal treatment, the extent of alterations depends on the chemical composition of the material used (Windeisen *et al.*, 2007).

Pine (*Pinus nigra)* and fir (*Abies bornmülleriana*) are the main wood species naturally grown and intensively used in the forest products industry in Turkey. Thus they are the potential wood species for industrial-scale heat treatment. To our knowledge, there is no information about the influence of the heat treatment on the technological properties of Turkish pine and fir. The objective of this study is to provide some information about the mechanical and physical properties of heat-treated *Pinus nigra* and *Abies bornmülleriana* using the ThermoWood process.

Materials and Methods

Pine (*Pinus nigra* Arnold.) and fir (*Abies bornmülleriana Mattf.*) planks were provided from Nova ThermoWood in Gerede. Prior to heat treatment, the planks were already kiln-dried approximately at a temperature of 70°C to a moisture content of 11-15%. Before heat treatment process, the density and moisture content of the planks were measured and 10 planks with a small variation in density from each species were selected for further experiments. The average densities and initial moisture content of planks were 0.498 g cm^{-3} and 13.7% for pine and 0.416 g cm^{-3} and 12.63% for fir, respectively.

Planks (50×110 mm in cross section and 3 m long) were subject to ThermoWood heat treatment using various schedules. The heat treatment was applied according to the method described in the Finnish ThermoWood Handbook (2003). The Thermo-Wood process involves three distinct process stages: 1. warming up stage, 2. drying stage and 3. cooling and conditioning stage. The warming up stage is to heat and pre-dry lumber. The temperature in the kiln was raised rapidly and a large amount of steam was generated. At the beginning of the drying stage, the temperature was increased steadily and the timber was dried intensively. At a certain point of the drying stage when the moisture content of the lumber reaches nearly 0%, the temperature was raised rapidly to target temperature. The target temperatures for pine and fir were 212 and 190°C, respectively. Wood was kept at this temperature for 2 hr. In cooling and conditioning phase, the temperature of the wood was lowered to 80 to 90°C using water spray system. Conditioning was carried out to moisten the heat-treated wood and bring its moisture content to 4-7%.

After heat treatment, only the planks that were free of defects were selected for further mechanical and physical properties testing. The untreated wood of the same species was used as a control sample.

The specimens that were used for the testing were cut from heat-treated and untreated planks according to the standards. Then, treated and untreated samples were conditioned in a climate controlled room at 65% RH and 20°C for 6 weeks. Before testing, the equilibrium moisture content (EMC) of the heat-treated pine and fir samples and the control samples was measured to be 4.9, 5.7, and 13%, respectively.

The tests were carried out according to Turkish standards (TSE) and 10 replicates were used in each test for treated and untreated pine and fir. The properties were determined based on the specimens with dimension of 2×2×3 cm for compression strength parallel to grain (TS 2595), 2×2×30 cm for bending strength (TS 2474), modulus of elasticity in bending (TS 2478) and impact bending strength (TS 2477), 3×3×1.5 cm for radial, tangential swelling and shrinkage, and 3×3×10 cm for longitudinal swelling and shrinkage (TS 4083, 4084). The changes in properties of heat treated pine and fir were obtained by calculating the property difference between heat-treated and untreated wood of the same species as a percentage of the untreated wood property according to the equations below:

$$D(\%) = \frac{M_{ut} - M_t}{M_{ut}} \times 100 \qquad (1)$$

where, D(%) is the difference of property (MOE, MOR, IBS, CS, Sw and Sh); M_{ut} is the mean value of untreated samples; and M_t is the mean value of the heat-treated samples.

Results and Discussion

The mean values and standard deviations of the obtained properties of untreated and heat-treated wood and the difference between them are presented in Table 1 for pine and Table 2 for fir. The heat treatment process generally reduced the mechanical properties, except for the compression strength in both wood species. Variance analysis results about the effects of heat-treatment on physical and mechanical properties of pine and fir are given in Table 3.

According to the results obtained in this study, the MOE was reduced 13.1% for pine and 9.5% for fir with the heat treatment. Also, heat treatment caused a decrease in MOR by 59.5 and 10.5% for pine and fir, respectively. The decrease in MOE is lower than MOR. According to variance analysis results (Table 3), the reductions in MOE and MOR with heat treatment were statistically significant compared to the untreated control wood for both wood species.

The reduction on the MOR is mainly due to the degradation of hemicelluloses (Esteves *et al.*, 2007a). According to

Table - 1: Mean values of untreated and heat-treated pine and differences in their properties (n=10)

Property	Pine-untreated		Pine-heat-treated		D(%)*
	Mean*	SD	Mean*	SD	
MOE (N mm^{-2})	8528.90	349.7	7411.57	538.4	-13.1
MOR (N mm^{-2})	75.65	4.4	31.03	2.6	-59.0
IBS (N mm^{-2})	0.43	0.13	0.16	0.02	-63.1
CS (N mm^{-2})	42.42	0.2	44.22	0.4	4.2
Sw-T (%)	8.55	0.4	3.22	0.5	-62.4
Sw-R (%)	4.42	0.4	1.50	0.4	-66.1
Sw-L (%)	0.18	0.09	0.07	0.04	-63.4
Sh-T (%)	7.26	0.89	3.62	0.2	-50.1
Sh-R (%)	4.00	0.6	1.79	0.1	-55.3
Sh-L (%)	0.16	0.3	0.08	0.3	-50.0

* = Average value of ten replicates; SD = Standard deviation; D(%) = Difference of property; MOE = Modulus of elasticity in bending; MOR = Modulus of rupture; IBS = Impact bending strength; CS = Compression strength parallel to grain; Sw-T = Swelling in Tangential direction; Sw-R = Swelling in Radial direction; Sw-L = Swelling in Longitudinal direction; Sh-T = Shrinkage in Tangential direction; Sh-R = Shrinkage in Radial direction; Sh-L = Shrinkage in Longitudinal direction

Table - 2: Mean values of untreated and heat-treated fir and differences in their properties (n=10)

Property	Fir-untreated		Fir-heat-treated		D(%)*
	Mean*	SD	Mean*	SD	
MOE (N mm^{-2})	8474.76	664.6	7667.20	447.1	-9.5
MOR (N mm^{-2})	68.29	6.5	61.13	5.0	-10.5
IBS (N mm^{-2})	0.35	0.01	0.32	0.02	-10.5
CS (N mm^{-2})	40.13	0.5	46.96	0.5	17.0
Sw-T (%)	8.60	0.9	4.57	0.6	-46.9
Sw-R (%)	3.44	0.2	2.22	0.3	-35.4
Sw-L (%)	0.17	0.06	0.10	0.02	-39.4
Sh-T (%)	7.73	0.5	4.99	0.1	-35.5
Sh-R (%)	3.17	0.3	2.20	0.1	-30.5
Sh-L (%)	0.17	0.8	0.10	0.1	-38.0

*, Average value of ten replicates; SD = Standard deviation; D(%) = Difference of property; MOE = Modulus of elasticity in bending; MOR = Modulus of rupture; IBS = Impact bending strength; CS = Compression strength parallel to grain; Sw-T = Swelling in Tangential direction; Sw-R = Swelling in Radial direction; Sw-L = Swelling in Longitudinal direction; Sh-T = Shrinkage in Tangential direction; Sh-R = Shrinkage in Radial direction; Sh-L = Shrinkage in Longitudinal direction

Dwianto *et al.* (1996) degradation of hemicelluloses causes the cross-linking reactions in matrix substance and the crystallization of microfibrils as well as the relaxation of stresses stored in microfibrils and matrix substance.

Similar changes in MOE and MOR have been reported in literature. The studies showed that heat treatment caused a decrease in MOR by 1-72% and in MOE by 1-40% (Yildiz, 2002b; Johansson and Moren, 2006; Esteves *et al.*, 2007a,b; Shi *et al.*, 2007; Korkut, 2008a,b). Results reported by Esteves *et al.* (2007b) with steam heat treated pine showed a maximum decrease by 5% in MOE and 40% in MOR at 190-210°C, respectively. Shi *et al.* (2007) observed decreases of 22 and 37% in the MOR and 6 and 4% in the MOE of steam heat treated at 212°C for pine and fir, respectively. Korkut (2008) reported a decrease in MOE of 35% and in MOR of 16% at 180°C for oven heat treated fir for 2 hr. These changes were higher than our results though the same wood species was used. As consistent with this result, Esteves *et al.* (2007a) indicated that the oven heat treatment affects more the mechanical properties of wood than steam heat treatment, possibly due to the oxidation reaction and the higher hemicellulose degradation.

According to the results obtained in this study, the IBS was reduced 63.1% for pine (Table 1) and 10.5% for fir with the heat treatment (Table 2). The reductions were statistically significant compared to the untreated control wood for both wood species (Table 3). It is known that higher the treatment temperature, the higher decrease in strength properties. Thus, the larger change in pine may have resulted from the higher temperature of the heat treatment. We thought that the high decrease in IBS was related to the brittleness of the heat treated wood. According to Hodgin and Lee (2002) wood becomes more brittle with heat treatment and Kubojima *et al.* (2000) noted that the main factors contributing to the reduction of absorbed energy in impact bending are viscosity and plasticity, rather than elasticity.

A little information about the IBS of heat treated wood is available in the literature. Korkut (2008a) reported a decrease in the IBS by 16% for Uludag fir heat treated at 180°C for 2 hr, and another study carried out at the same treatment conditions reported a decrease by 14% for Red-bud maple (Korkut *et al.*, 2008b). These changes were higher than our results. This may explained by differentiation of the methods of heat-treatment used.

The CS increased by 4.2% for pine and 17% for fir with the heat treatment (Table 1,2). According to variance analysis results, the increase of CS with heat treatment was significant for fir, but not for pine. Similar results have been reported by the Finnish ThermoWood Association (2003), where the CS of heat treated timber at 195°C for 3 hr was approximately 30% higher than that of normal untreated timber. However, these observations contradict some of the reports in the literature. Heat treatment is reported to have caused a decrease in the CS by 2 to 32% (Schneider, 1973; Korkut, 2008a; Yildiz, 2002b; Unsal and Ayrilmis, 2005; Korkut *et al.*, 2008b). Using oven-dried heat treatment method, Korkut (2008) reported a decrease in CS by 10% for Uludag fir and Yildiz *et al.* (2006) reported a decrease in CS by 6% for spruce. Yildiz *et al.* (2006) suggested that the decrease in the CS properties can be reduced using a closed system with an inert gas like nitrogen or water vapor as the shielding gas instead of air. These changes in the CS, although this is not explicitly stated, appear to correspond to the heat treatment method and, accordingly, a modified chemical structure. Kandem *et al.* (2002) and Nuopponen (2005) reported that heat treated samples exhibit a higher lignin content and lower acid number compared to the untreated controls indicating to the degradation of some hemicellulose and extractive compounds.

Table - 3: Variance analysis results with regard to the effect of heat treatment on mechanical and physical properties of pine and fir wood species

Wood species	Dependent variable	Sum of squares	Degree of freedom	F ratio	Sig.
Pine	MOE	6.242E+06	1	30.546	0.000
	MOR	9954.722	1	768.902	0.000
	IBS	0.370	1	44.821	0.000
	CS	16.200	1	0.107	0.747
	Sw-T	141.831	1	618.691	0.000
	Sw-R	42.574	1	246.661	0.000
	Sw-L	0.065	1	14.153	0.001
	Sh-T	66.321	1	161.356	0.000
	Sh-R	24.376	1	111.134	0.000
	ShL	0.032	1	26.667	0.000
Fir	MOE	3.261E+06	1	10.164	0.005
	MOR	257.762	1	7.607	0.013
	IBS	0.007	1	24.992	0.000
	CS	232.971	1	17.588	0.001
	Sw-T	81.285	1	130.423	0.000
	Sw-R	7.491	1	132.723	0.000
	Sw-L	0.025	1	12.629	0.002
	Sh-T	37.538	1	245.625	0.000
	Sh-R	4.685	1	99.261	0.000
	Sh-L	0.020	1	6.024	0.025

Swelling and shrinkage in all directions were improved for both pine (Table 1) and fir (Table 2) with the ThermoWood process. The improvement of swelling and shrinkage with heat treatment were statistically significant compared to the untreated control wood for both wood species (Table 3). Volumetric swelling and shrinkage of heat treated pine were decreased by 63.9 and 51.8% with the heat treatment. For heat treated fir, the reductions were determined as 40.6% in volumetric swelling and 34.6% in volumetric shrinkage. The difference between pine and fir can be explained by a higher treatment temperature for the pine than the fir. The higher treatment temperature caused higher depolymerization of hemicellulose; consequently, there was less water absorption in the cell wall.

Shrinkage and swelling are phenomena that occur under the fibre saturation point (FSP) due to moisture absorption. It sequences with dimensional changes in wood. The availability and/or accessibility of the free hydroxyl groups of the wood carbohydrates play an important role in the process of water adsorption and desorption. The heat treatment changes the chemical structure of the wood, especially the hydroxyl groups. Hence, the reduction of water absorption after heat treatment is most probably due to the combination of the following effects: Depolymerisation of the carbohydrates and especially hemicelluloses, resulting in a reduction of the total amount of hydroxyl groups, including the free hydroxyl groups; an increase in the relative proportion of the crystalline cellulose, in which the hydroxyl groups are not easily accessible to water molecules (Kartal *et al.*, 2007) and further cross-linking of the lignin network (Tjeerdsma *et al.*, 1998), which hinders accessibility of free hydroxyl groups to water (Pizzi *et al.*, 1994). Thus, the wood absorbs less moisture and becomes much more water repellent.

The decreases in volumetric swelling and shrinkage have been indicated by several authors. According to Viitaniemi (1997), the effects of thermal treatment of wood at temperatures between 185 and 250°C were shrinkage and swelling reduction of 30-80% for spruce, pine, and birch. Mohebby and Sanaei (2005) reported that the volumetric swelling in treated wood was 7.86% with an ASE of 45.5% (180°C for 4 hr). In another study, the swelling-reduction efficiency was found to be 24% in Eucalyptus (Eucalyptus globulus) wood treated at 180°C (Santos, 2000). Yildiz (2002a) reported an Anti Swelling Efficiency (ASE) of heat treated beech about 47.64%.

Taking into account the results of these tests, it can be said that the mechanical properties were negatively affected by the ThermoWood process, except for the compression strength in both wood species. The volumetric shrinkage and swelling of these species were improved by approximately half without using any water repellent.

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