EFFECTS OF BORON IMPREGNATION AND HEAT TREATMENT ON SOME PHYSICAL AND MECHANICAL PROPERTIES OF SPRUCE AND PINE WOOD

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Abstract

This study evaluated the effects of boron impregnation and heat treatment on some physical and mechanical properties of wood. Spruce and pine wood samples were treated with 4% aqueous solution of both boric acid (BA) and borax(BX) by using full cell method, and then were exposed to heat treatment at 212 ºC for 120 min in an industrial site system. After the treatments, the physical (water absorption and tangential swelling) and mechanical (bending and compression strengths) properties of the specimens were measured according to the related standards. In addition, the remained boron content in heat treated wood specimens was analyzed by means of Inductively Coupled Plasma (ICP). There were generally negative changes in mechanical properties of heat treated wood due to boron treatment. Boron treatment significantly increased water absorption and decreased tangential swelling values of heat treated wood. It appears that heat treatment provide boron retention of about 18-63 % of initial amount depending on boron compound and wood species.

Keywords: Boron compounds, heat treatment, physical and mechanical properties, pine, spruce.

1. Introduction

Heat treatment which is an alternative method to the treatment with chemical materials is developed to improve physical, chemical, biological and mechanical properties of wood material that has low natural durability. However, decrease in the mechanical properties is observed because of the weight loss at the wood material during the heat treatment (Patzelt et al., 2002; Gunduz and Aydemir, 2008).

After heat treatment, there occurs a decrease on mechanical properties of wood because of the decrease at the specific gravity of wood. Contrary to decrease on mechanical properties, dimensional stabilization is improved (Yıldız, 2002).

Heat treatment causes significant changes at the chemical structure of the wood material. These changes generally result in by degradation of amorphous carbohydrates. Acidity of wood increases, extractive material content decreases and cellulose crystallinity increases because of the decrease at the amorphous zones of cellulose. Acetic acid is generated as a result of degradation of hemicelluloses and this acid causes degradation of carbohydrates (Awoyemi and Westermark, 2005; Kamdem et al., 2002).

These changes at the wood material have important effects on physical properties of the material. During the heat treatment, the number of hydrophilic OH groups is decreased and replaced by hydrophobic O-acetyl groups. The latter creates cross-links between wood fibers and thus it significantly reduces the ability of the water to penetrate into the wood (Poncsak et al., 2006). As a result of physical changes in wood material there would be reduction at the equilibrium moisture content in hygroscopicity and, electrical resistance, and increase in brittleness (Awoyemi, 2009).

Physical and biological properties of wood is improved by heat treatment at high temperatures, but despite the increase at the

strength properties at the initial steps of the heat treatment, there is a decline at the strength properties of wood by the increase at the heat treatment duration and temperature. A negative consequence is that wood becomes more brittle, and bending and pulling strength decrease by 10% to 30% (Jamsa et al., 1996; Awoyemi, 2009; Welzbacher and Rapp, 2002). In a study performed by Awoyemi and Westermark (2005), wood samples impregnated with boron salts are subjected to heat treatment to decrease adversely affect of heat treatment on the strength properties of wood.

At the study reported by Awoyemi (2007), impregnated wood samples with boron salt in different concentrations were subjected to heat treatment. Decrease in the values of MOE (Modulus of Elasticity) and MOR (Modulus of rupture) was minimized until 3% concentration level. However, a significant difference between samples impregnated with boron salt at the 3% and 5% concentration levels was not observed.

Boron compounds are used in wood impregnation against to fungi, insects, and termites for years. The effect of impregnation treatment with boron compounds on the physical, mechanical and biological characteristics of wood material is investigated by many researchers (Jamsa et al., 2001; Awoyemi, 2007; Tomak et al., 2008). According to the results, while a positive change occurs at the biological and physical properties of wood, a negative change occurs at mechanical properties.

This study evaluated the effects of boron impregnation and heat treatment on some physical (water absorption and tangential swelling) and mechanical (bending and compression strengths) properties of wood. The amount of remaining boron after heat treatment of pine and spruce wood samples was also investigated.

2. Materials and Methods

Spruce (*Picea orientalis* Link.) and pine (*Pinus nigra* subsp.) wood samples obtained from an industrial plant in Turkey. Test and control samples were prepared at the dimensions of 5 x 10 x 80 cm (thickness x width x length). Wood samples (80 cm length) were cut into two parts. The first part of each wood samples was impregnated with boron compounds before heat treatment whereas the other part of each wood sample was heat treated without any boron impregnation.

Spruce and pine wood specimens treated with 4% aqueous solution of both boric acid (BA) and borax (BX) at 650mm-Hg vacuum for 30 min and a pressure of 5 bar for 60 min at room temperature. After the treatment, specimens were re-weighed to determine the boric acid (BA) and borax (BX) retention. All treated specimens were then reconditioned at 20±2 ^{O}C and %65±5 RH for 2 weeks.

The retention content for each treatment was calculated following formula.

R (Kg / m^3) = [G X C / V] X10

Where;

G: (T2-T1) is the grams of treating solution absorbed by the block (initial weight of block subtracted from the initial weight plus the treating solution absorbed); C is the grams of chemical solution in 100 g of the treating solution; and V is the volume of block in cubic centimeters.

Basic Thermowood method was applied to the samples as heat treatment method. Heat treatment was carried out in an industrial furnace at 212 ºC and for 2 hours duration.

After heat and boron treatments, samples prepared by large-dimensions were reduced small size to identify physical and mechanical properties of the samples. The wood was sawn into

specimens measuring 2(T) x 2(R) x 30(L) cm for static bending strength test, 3(T) x3(R) x1.5(L) cm for water absorption and tangential swelling tests, 2(T) x2(R) x3(L) cm for compression strength test according to TS 2472 standard method. Preparation of the small-scale samples (2x2x30 cm, 3x3x1.5 cm and 2x2x3 cm) from the large-scale ones (5x10x40cm) is shown in Figure 1.

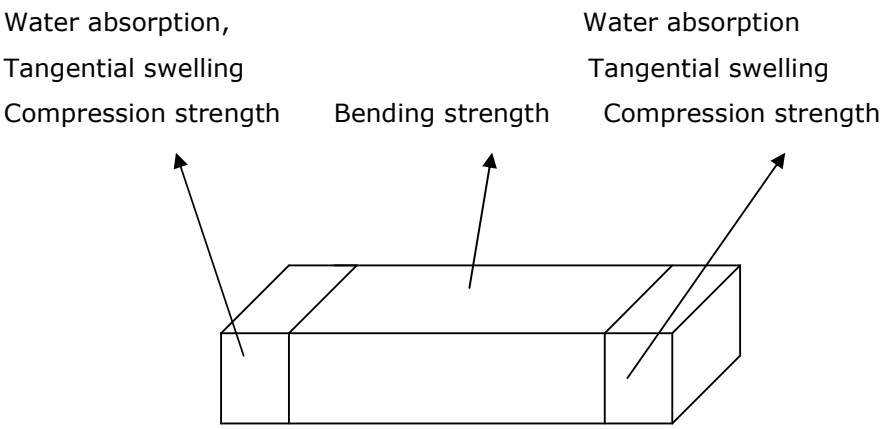

Figure 1: Preparation of the small-scale samples from the large-scale ones.

Boron content of both heated and unheated wood samples was measured using Inductively Coupled Plasma (ICP) analysis, using an ICP spectrometer (ICP-AES, Spectro Genesis). Sample preparation was done according to AWPA A7-93. Wood samples for the ICP analysis were prepared by grinding in a Wiley mill with a mesh size of 0.5 mm (IKA MF10, IKA-Werke, Staufen, Germany) followed by oven drying at 103 ± 2ºC. One gram of ground wood was weighed to the nearest 0.01g and placed in a 100 ml flask. Nitric acid (65%) was then added to the flask, which was placed on a hot plate. After the evolution of brown fumes had ceased, hydrogen peroxide (30%) was added dropwise to clear the solution. After cooling, the contents in the flask were filtered through Whatman #4 filter paper and were

diluted with distilled water for measurement. For each treatment, two replicate samples were ground and analyzed.

3. Results and discussion

3.1. Retention

Retention levels of the compounds in the wood specimens after treatments are shown in Table 1.

Table 1: Retention of the samples (kg/m3)

Wood species		Retention (kg/m^3)	SD
spruce	BA	23.99	0.74
	BX	25.58	0.05
pine	BA	26.93	1.98
	BX	24.3	0.42

On average, nearly 23.9 kg/m^3 retention was obtained for BA and 25.6 kg/m^3 for BX in spruce and, nearly 26.9 kg/m^3 retention was obtained for BA and 24.4 kg/m^3 for BX in pine.

3.2. Boron Analysis of Treated Wood

Table 2 shows the amount of BA and BX remaining in heated samples. As a result of the test, BX significantly remained in wood samples for both spruce and pine, while a significant portion of BA removed from wood during the heat treatment prosess.

Table 2: Amount of BA and BX remaining in heated samples (%)

Wood sampels	Remaning%
Spruce (BX)	63.01
Spruce (BA)	18.20
Pine (BX)	34.61
Pine (BA)	18.64

As expected, heat treatment caused a decrease of the initial amount of the boron compounds. As it is well known, heat treatment includes steaming of the wood at elevated temperatures and, boron compounds can easily be leached out from wood when it is exposed to steam or water. These factors probably interact in complex ways causing boron loss in the pre-impregnated wood samples. Kartal (2006) combined boron and heat treatment as a dual treatment to delay the boron leaching from wood and, stated that heat treatment had no effect on boron release. Boric acid seemed to be more affected from heat treatment compared to borax. This might be due to the different characteristics and leaching properties of the chemicals. These findings need to further investigation.

3.3. Physical properties

Average values of oven-dry density, water absorption and tangential swelling of the test and control samples are given in Table 3.

Table 3: Average values of oven-dry density, water absorption and tangential swelling of the samples.

Heat Treatment Temperature and Time; Wood species		Oven- dry density (g/cm^3)		Water absorption (%) (72 h)		Tangential swelling (%) (72 h)	
212ºC and 120 min		Control *	Test**	Control	Test	Control	Test
Pine	BA	0,505	0,505	85	69	5	4
	BX	0,487	0,468	81	97	5,4	5,2
Spruce	BA	0,331	0,352	97	146	4,1	3,7
	BX	0,340	0,347	114	179	3,2	4,8

*Control samples were subjected to only heat treatment
**Test samples were both boron impregnated and heat treated.

Boron compounds generally increased water absorption and tangential swelling of wood. Increased water absorption of boron treated samples was also demonstrated by Baysal et al. (2006). Water absorption of test samples was higher than those of the controls with an exception of pine samples impregnated with BA. Water absorption of spruce samples was higher than those of pine samples. Similar results were obtained in some studies; spruce samples are more affected than pine samples from heat treatment.
After heat treatment, knots are separated from wood and annual ring structure is relaxed. In this case, after heat treatment of spruce wood is composed of some structural gaps. Hence, water absorption rate of spruce wood increased to some extent (Fojutowski et al., 2009; Yildiz, 2002). The samples treated with boric acid absorbed remarkably less water in comparison with in borax treatments. This

might be due to the different chemical composition of the chemicals. A study by Kartal et al. (2007) reported that in the DOT (disodium ocatborate tetrahydrate;a mixture of borax and boric acid) treatments, the specimens at 180 C for 4 h showed slightly less water absorption than the boric acid treated ones. Toker (2007) also reported that borax treated wood exhibited greater hygroscopicity than boric acid treated material.

Heat treatment significantly reduces the tangential swelling of wood and provides dimensional stabilization to it (Yildiz, 2002; Thermowood Handbook, 2003). However, tangential swelling was increased in impregnated with BX at spruce samples in our study. On the contrary, for pine samples this variation did not differ too much for the control (%5.4) and the test samples (%5.2). This might be due to more hydrophobic nature of borax compared to boric acid. Kartal et al. (2007) reported that water absorption of the wood specimens treated with boron compounds followed by heat treatment increased more than those of the only heat treated ones. Accordingly, boron compounds may affect the water absorption of heat treated wood in spite of hydrophobication provided by heat treatment.

3.4. Mechanical properties

Compression parallel to grain (CS) and static bending (modulus of rupture: MOR) strength values of the test and control samples are given in Table 4.

Table 4: Average values of compression strength parallel to grain and bending strength of the samples.

Heat Treatment Temperature and Time; Woodspecies / 212 C and 120 min		CS (N/mm^2)			MOR (kg/cm^2)		
		Control	Test	Changes (%)	Control	Test	Changes (%)
Spruce	BA	17	12	-29	507	595	+ 17
	BX	15	14	-7	669	663	-5
Pine	BA	23	16	-30	1215	834	-31
	BX	22	24	+ 9	1012	1149	+ 14

As can be seen on Table 4; due to high heat treatment temperature mechanical strength values were quite low for the test samples. While the control samples were only subjected to heat treatment, the test samples have been subjected to heat treatment after impregnation with boron. As known, heat treatment generally decreases the mechanical properties of wood. Degradation of hemicellulose which connects cellulose and lignin in the cell wall causes a decrease in strength of wood. In the present study, borax pre-impregnated wood samples provided more reasonable results than boric acid treated ones for strength losses. On the other hand, the influence of the heat treatment on MOR was rather negligible compared to crushing strength. It is reported that substantial strength loss in pine starts at temperatures over 220°C (Thermowood Handbook, 2007). A study by Yıldız et al. (2002) also reported that compression strength values of beech and spruce wood samples were reduced with increasing exposure durations and temperatures.

In the study the decreased compression strength parallel to grain of treated wood samples might be assumed to be caused by degradation of wood carbohydrates and decreasing hemicelluloses content in the samples subjected to heated and untreated (Talaei et al., 2010).

Borax has more advantage than boric acid in terms of reducing resistance losses. The maximum MOR loss (31%) was obtained for heat treated pine samples that was pre-treated with boric acid. Awoyemi et al. (2005) found that wood specimens treated with sodium borate and subjected to heat treatment at 180 and 200 ◦C for 2 and 4h resulted in some increases in strength loss during heat treatment. They suggest that this is invariably due to the buffering effect of the alkali on the acidity of wood, which could mitigate the degree of degradation. The effect of the boron compound, however, was not clear in comparison with the time and temperature effects. In addition, Kartal (2007) was reported that a direct relationship was found between strength and hemicellulose losses of the specimens. As the hemicellulose content in the specimens decreased, losses in the MOR increased.

3. Conclusions

The combined effects of boric acid and borax solution on some physical and mechanical properties of spruce and pine, together with heat treatments at 212 C for 120 min. were evaluated. In general, compression strength parallel to grain and bending strength decreased in wood samples pre-treated with boric acid and borax. The combination of heat treatment and boron impregnation were negatively affected physical properties of spruce and pine wood. However in some variations tangential swelling rate decreased. Heat treatment process significantly affected remaining boron compounds in wood. The reason might be applied water vapor during heat

treatment. However, chemical structure of boron compound also affects the amount of boron remaining in wood. Therefore there is need to investigate boron remaining in wood after heat treatment with different heat treatment parameters and different boron compounds.

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