

Effects of Boron Impregnation and Heat Treatment on Some Mechanical Properties of Oak (*Quercus petraea* Liebl.) Wood

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Heat treatment changes some physical, mechanical, and chemical properties of wood. Inorganic borates have been used as wood preservatives for many years. The aim of this study was to investigate the effects of impregnation chemicals on some mechanical properties (bending strength (MOR), modulus of elasticity (MOE), tensile strength parallel to the grain (TS), compression strength parallel to the grain (CS), and shear strength parallel to the grain (SS)) of heat-treated oak (*Quercus petraea* Liebl.). For this purpose, the oak wood specimens were impregnated with 5% aqueous solution of boric acid (BA) and borax (BX). Then specimens were heat-treated at 160, 190, and 220 °C for 2 and 4 h. According to the results of the study, borax retention value was higher than boric acid. The bending strength, modulus of elasticity in bending, tensile strength parallel to the grain, and shear strength parallel to the grain decreased due to heat treatment. The highest mechanical strength losses were determined in samples heat treated at 220 °C for 4 h. Generally the mechanical strength losses of samples impregnated with borax were lower than non-impregnated controls and specimens impregnated with boric acid.

Keywords: Borax; Boric acid; Heat treatment; Oak wood; Mechanical strength

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INTRODUCTION

Environmental awareness has led to increased interest in developing new, alternative wood modification methods. Heat treatment is one of the processes used to modify the properties of wood (Mazela *et al.* 2004) and can be considered an environmentally friendly technique (Ates *et al.* 2009). Heat treatment, as a wood modification method, serves to improve the natural properties of the wood, such as dimensional stability (Esteves *et al.* 2008b; Todorovic *et al.* 2012) and resistance to bio-deterioration, as well as bestowing the wood material with new properties (Altinok *et al.* 2010). The modification treatment is always performed in the temperature range of 180 to 240 °C (Niemz *et al.* 2010). Heat treatment, especially at temperatures over 150 °C permanently changes the physical, mechanical, and chemical properties of wood (Mitchell 1988; Yildiz *et al.* 2006; Gunduz *et al.* 2008). Cellulose and lignin degrade more slowly and at higher temperatures than hemicelluloses. Thermal degradation starts by deacetylation of hemicelluloses, and the released acetic acid acts as a depolymerization catalyst, which accelerates the decomposition of polysaccharides (Tjeerdsma *et al.* 1998; Nuopponen *et al.* 2004).

It is known that organic acids, such as formic and acetic acid, are formed during the heat treatment process and that these may affect the properties of wood (Garrote *et al.* 2001; Feng *et al.* 2002; Manninen *et al.* 2002). In addition, heat treatment also reduces wood's pH (Boonstra *et al.* 2007a). According to Salim *et al.* (2008), depending on the acid concentration and applied temperature, hemicellulose, the most reactive wood component, will be hydrolysed into oligomeric and monomeric structures (Carrasco and Roy 1992).

The strong correlation between strength reduction and acidity in wood has been reported in previous studies. Treatments between 200 and 260 °C can cause significant degradation in hemicellulose content of wood, which releases a large amount of acetic acid (Weiland *et al.* 1998). The loss of strength is associated with changes in wood acidity (Hodgin and Lee 2002). Sundqvist *et al.* (2006) found that when birch was heated to 180 °C for 1 to 2.5 h, it lost considerable strength and hardness. Losses in mechanical properties can be linked to the loss of mass and increase in concentrations of formic and acetic acid. Cellulose degradation can contribute to the loss of mechanical strength in wood materials under high temperature treatment (Sundqvist 2004). Boron compounds are used as wood preservatives as they are both fungicides and insecticides, relatively inexpensive, and environmentally acceptable (Thévenon *et al.* 2010). According to Tjeerdsma *et al.* (1998), the degradation of the hemicelluloses results in the formation of acetic acid, which serves as the catalyst in further carbohydrate decomposition (Awoyemi and Westermarck 2005).

Wang *et al.* (2012) investigated that the impact of pH on chemical and mechanical properties of thermally modified Cathay poplar (*Populus cathayana* Rehd.) wood. As a result, disodium octoborate tetrahydrate (DOT) and buffering solutions decreased mass loss of heat-treated wood and increased MOR and MOE. Winandy (1997) determined that adding boron-based buffers to the fire retardant treatment chemicals appeared to significantly reduce subsequent thermal degradation. Awoyemi (2008) reported that pre-impregnation of borate as an alkali-buffering medium decreased the severity of strength loss during heat treatment. This was invariably due to the buffering effect of alkali on wood acidity. Awoyemi and Westermarck (2005) suggested that a preliminary borate impregnation of wood before heat treatment may reduce the severity of mechanical strength loss during heat treatment. Kartal *et al.* (2008) determined that effects of boron impregnation and heat treatment on chemical and strength properties of wood and discovered that boric acid (BA) and di-sodium octoborate tetrahydrate (DOT) clearly changed the pH value of wood, which made the decrease in MOE in the untreated wood slightly higher than that in the treated specimens. The objective of this study was to determine the effect of the impregnation with different boron compounds (borax and boric acid) that have different pH value before heat treatment on mechanical strength of oak (*Quercus petraea* Liebl.) wood.

EXPERIMENTAL

The oak (*Quercus petraea* Liebl.) wood samples used in this study were selected randomly. The wood was parallel to the grain and sawn into specimens measuring 50×80×800 (tangential, radial, longitudinal) mm in dimensions. Afterwards, the lumber was air dried for two months until it reached approximately 12% moisture content.

In the literature wood samples were impregnated with different concentrations (1 to 13%) of boron compounds (Peker *et al.* 1999; Toker 2007; Awoyemi 2008;

Özçifçi 2008). In conclusion, mechanical strength *losses increased* with increasing concentration of boron compounds. Therefore, 5% concentration was preferred in this study, and the oak wood specimens were impregnated with 5% aqueous solution of boric acid (BA) (H_3BO_3 is 56.30% $\frac{1}{2} \text{B}_2\text{O}_3$, 43.70% H_2O with a molecular weight 61.84, density 1.435 g.cm^{-3} and melting point $171 \text{ }^\circ\text{C}$) and borax (BX) ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$ content is 21.28 Na_2O , 47.80% B_2O_3 and 30.92% H_2O with a molecular weight of 291.3, density 1.815 g.cm^{-3} , and melting point $741 \text{ }^\circ\text{C}$) (Örs *et al.* 2006). The samples were dipped in the impregnation pool for 16 h. Prior to the impregnation, the dimensions respectively were measured by a digital caliper and their weights were recorded on a digital scale. After the impregnation, specimens were weighted again. All treated and control specimens were then reconditioned at $20 \pm 2 \text{ }^\circ\text{C}$ and $65 \pm 5\%$ relative humidity for three weeks.

The impregnation was carried out at $20 \pm 2 \text{ }^\circ\text{C}$. The retention content for each treatment was calculated by the following formula (Eq. 1),

$$R = \frac{G \cdot C}{V} 10^3 \text{ (kg.m}^{-3}\text{)}, \quad G = T_2 - T_1 \quad (1)$$

where G is the amount of impregnation solution absorbed by the sample, T_2 is the sample weight after the impregnation, T_1 is the sample weight before the impregnation, C is the concentration (%) of the impregnation solution, and V is the dry volume of the samples.

Afterward, impregnated specimens of oak wood were cut into smaller specimens that were free of defects for determination of air-dry density according to (ISO3131, 1975), compression strength parallel to the grain (CS) according to (ISO3787, 1976), bending strength (MOR) according to (ISO3133, 1975), modulus of elasticity in bending (MOE) according to (ISO3349, 1975), shear strength parallel to the grain (SS) according to (TS3459, 2012), and tension strength parallel to the grain (TS) according to (ISO3345, 1975).

The samples were heat-treated at 160, 190, and $220 \text{ }^\circ\text{C}$ for 2 and 4 h. Heat treatment was carried out under atmospheric pressure, with water vapour as a shielding gas. The total duration of the heat treatment (pre-treatment period + actual heat treatment period + cooling and moisture conditioning period) was 30 h and actual times at the high temperatures were 2 and 4 h. After the heat treatment, specimen dimensions were re-measured with the digital caliper, and their weights were recorded on a digital weight scale. All treated (impregnation and heat treated) and control specimens were then reconditioned at $20 \pm 2 \text{ }^\circ\text{C}$ and $65 \pm 5\%$ relative humidity for 3 weeks.

Multiple variance analysis was used to determine the difference between the physical and mechanical properties of impregnated and heat-treated woods. When there was a significant difference between the groups, the $P \leq 0.05$ confidence level was compared with Duncan's test.

RESULTS AND DISCUSSION

Properties of the chemicals ($23 \pm 2 \text{ }^\circ\text{C}$, 5% solution concentration) used in the impregnation process are given in Table 1.

Table 1. Properties of Impregnation Chemicals

Impregnation material	Solution Concentration (%)	Solvent	Retention (kg.m ⁻³)	SD	pH		Density	
					BI	AI	BI	AI
Borax	5	DW	8.27	0.426	11.2	10.90	1.24	1.20
Boric acid	5	DW	6.73	0.366	6.2	6.20	1.22	1.22

DW: Distilled water, SD: Standard deviation, BI: Before impregnation, AI: After impregnation

According to Table 1, there was no important change in the acidity and density of the solutions before and after the impregnation.

Averages of the mechanical properties of impregnation chemical, heat temperature, and heat treatment duration are given in Table 2.

Table 2. Average of Mechanical Properties of Impregnation Chemical, Heat Temperature, and Heat Treatment Duration

		MOR	MOE	TS	CS	SS
Impregnation chemical	Non-impregnated HG	101.80 A	11000 B	84.02 B	75.15 B	14.66 B
	Borax HG	103.20 A	11270 A	85.75 A	78.11 A	15.30 A
	Boric acid HG	98,84 B	10830 C	82.53 C	75.50 B	14.45 B
Heat temperature	Untreated HG	108.80 A	11860 A	94.25 A	73.29 D	17.07 A
	160 °C HG	108.60 A	11370 B	90.76 B	79.66 A	15.87 B
	190 °C HG	97.89 B	10840 C	82.17 C	76.98 B	13.78 C
	220 °C HG	89.96 C	10060 D	69.22 D	75.07 C	12.49 D
Heat treatment duration	2 hours HG	104.40 A	11360 A	86.51 A	77.09 A	15.43 A
	4 hours HG	98.16 B	10710 B	81.69 B	75.41 B	14.17 B

MOR: Bending strength, MOE: Modulus of elasticity, TS: Tensile strength parallel to the grain, CS: Compression strength parallel to the grain, SS: Shear strength parallel to the grain, HG: Homogeneity groups

The retention value of boric acid (8.27) was higher than borax (6.73). Results of variance analyses are given in Table 3. According to Table 3, the effect of the impregnation chemical, heat treatment temperature, and heat treatment duration on bending strength (MOR), modulus of elasticity (MOE), tensile strength parallel to the grain (TS), compression strength parallel to the grain (CS), and shear strength parallel to the grain (SS) were found to be statistically significant ($P < 0.05$). The Duncan test was used to determine the differences between means at a prescribed level of $\alpha = 0.05$.

The results of the Duncan test are given in Table 4. Table 5 shows the percentage decrease or increase of values in relation to the control for each impregnation and heat treatment and each measured parameter.

Table 3. Results of Variance Analyses

Tests	Source	Degrees of Freedom	Sum of Squares	Mean Square	F Value	Prob P ≤ 0,05
MOR	Factor A	2	803.464	401.732	16.4753	0.0000
	Factor B	3	15126.406	5042.135	206.7808	0.0000
	A*B	6	369.570	61.595	2.5260	0.0220
	Factor C	1	2353.178	2353.178	96.5052	0.0000
	A*C	2	3.032	1.516	0.0622	
	B*C	3	822.224	274.075	11.2400	0.0000
	A*B*C	6	114.962	19.160	0.7858	
	Error	216	5266.936	24.384		
	Total	239	24859.772			
MOE	Factor A	2	7766913.308	3883456.654	18.0046	0.0000
	Factor B	3	106552736.083	35517578.694	164.6676	0.0000
	A*B	6	10446755.792	1741125.965	8.0723	0.0000
	Factor C	1	24797938.817	24797938.817	114.9689	0.0000
	A*C	2	726168.658	363084.329	1.6833	0.1882
	B*C	3	8869670.617	2956556.872	13.7073	0.0000
	A*B*C	6	627334.908	104555.818	0.4847	
	Error	216	46589605.000	215692.616		
	Total	239	206377123.183			
TS	Factor A	2	415.332	207.666	15.1596	0.0000
	Factor B	3	22356.812	7452.271	544.0167	0.0000
	A*B	6	121.732	20.289	1.4811	0.1858
	Factor C	1	1397.899	1397.899	102.0468	0.0000
	A*C	2	1.638	0.819	0.0598	
	B*C	3	816.224	272.075	19.8615	0.0000
	A*B*C	6	7.074	1.179	0.0861	
	Error	216	2958.899	13.699		
	Total	239	28075.611			
CS	Factor A	2	417.865	208.933	16.2011	0.0000
	Factor B	3	1338.989	446.330	34.6093	0.0000
	A*B	6	188.071	31.345	2.4306	0.0270
	Factor C	1	169.411	169.411	13.1365	0.0004
	A*C	2	74.662	37.331	2.8947	0.0575
	B*C	3	201.453	67.151	5.2070	0.0017
	A*B*C	6	241.085	40.181	3.1157	0.0060

	Error	216	2785.584	12.896		
	Total	239				
SS	Factor A	2	31.551	15.776	5.9049	0.0032
	Factor B	3	759.328	253.109	94.7402	0.0000
	A*B	6	24.122	4.020	1.5048	0.1777
	Factor C	1	95.330	95.330	35.6827	0.0000
	A*C	2	4.743	2.371	0.8876	
	B*C	3	36.951	12.317	4.6103	0.0038
	A*B*C	6	13.598	2.266	0.8483	
	Error	216	577.069	2.672		
	Total	239				

Factor A: Borax, Boric acid; Factor B: Heat treatment temperature; Factor C: Heat treatment duration, MOR: Bending strength, MOE: Modulus of elasticity, TS: Tensile strength parallel to grain, CS: Compression strength parallel to grain, SS: Shear strength parallel to grain

Table 4. Results of the Duncan Test

Impregnation Chemical	Heat treatment	Hour	SV	Density (g/cm ³)	MOR (N/mm ²)	MOE (N/mm ²)	TS (N/mm ²)	CS (N/mm ²)	SS (N/mm ²)
Non-impregnated	Un-treated		Mean	0.7320	111.43	11848	94.45	71.81	17.16
			HG		B	AB	AB	M	A
			SD	0.0573	4.59043	498.86	3.2940	3.5851	2.1361
			Mean	0.7141	110.82	11541	91.14	74.28	16.27
	160 °C	2	HG		BC	BC	CDE	IJKLM	AB
			SD	0.0182	4.8675	553.32	2.6080	3.1456	1.8578
		4	Mean	0.6981	103.60	10785	88.27	80.65	14.75
			HG		D	E	EFG	BC	CD
			SD	0.0150	3.3999	508.17	2.6298	4.1887	1.4755
			Mean	0.7020	102.80	10985	86.44	78.84	14.36
	190 °C	2	HG		DE	E	GH	CDEF	CDE
			SD	0.0222	4.9296	364.90	2.6844	3.6248	1.5508
	4	Mean	0.6840	93.36	10363	79.28	75.25	12.95	
		HG		HI	F	JK	GHIJKL	EFG	
		SD	0.0167	2.8002	310.69	4.1911	3.4761	1.4081	
		Mean	0.6720	94.14	10778	73.25	75.87	12.64	
220 °C	2	HG		GHI	E	LM	FGHIJK	FG	
		SD	0.0124	3.9902	429.64	3.1754	3.6136	1.3428	
	4	Mean	0.6463	86.56	9873	64.87	72.65	11.98	
		HG		K	G	NO	LM	G	
		SD	0.0078	3.2961	361.68	3.2533	3.3721	1.1209	
		Mean	0.7430	108.20	11820	95.25	74.76	16.99	
Borax	Un-treated		HG		BC	AB	A	HIJKLM	A
			SD	0.0245	7.7744	612.90	5.8435	2.1234	1.3249
		2	Mean	0.7210	116.60	12027	93.45	84.78	16.86
			HG		A	A	ABC	A	A
		4	Mean	0.7120	106.60	11180	90.75	82.12	15.75
			HG		CD	CDE	CDEF	AB	ABC
			SD	0.0221	5.1147	360.72	2.6839	2.9083	1.6380
			Mean	0.7170	103.28	11517	87.65	79.32	15.01
	190 °C	2	HG		D	BCD	FG	BCDE	BCD
			SD	0.0189	3.7032	412.99	3.0053	5.5376	1.3923
		4	Mean	0.7010	97.89	10936	80.16	76.15	13.75
			HG		FG	E	IJ	FGHIJ	DEF
		SD	0.0132	3.9201	364.22	3.3399	4.0461	1.6354	

	220 °C	2	Mean HG SD	0.6881 0.0133	97.19 FGH 3.4060	10825 E 325.48	76.65 K 3.4969	78.27 CDEFG 2.7474	14.95 BCD 1.1626
		4	Mean HG SD	0.6570 0.0142	87.93 JK 3.3014	10017 FG 213.02	66.84 N 2.8352	74.69 HIJKLM 4.0315	12.11 G 1.4250
Boric acid	Un-treated		Mean HG SD	0.7370 0.0238	106.89 CD 8.0217	11910 AB 493.91	93.06 ABC 4.4790	72.95 KLM 3.3846	17.05 A 2.1241
		160 °C	2	Mean HG SD	0.7190 0.0226	110.25 BC 5.2538	11780 AB 367.02	91.74 BCD 4.2748	16.55 A 2.2252
	160 °C	4	Mean HG SD	0.7080 0.0176	103.37 D 4.6622	10925 E 479.85	89.21 DEFG 3.9144	76.25 EFGHIJ 2.6952	15.05 BCD 1.2535
		190 °C	2	Mean HG SD	0.7140 0.0183	98.75 EF 3.7364	11123 DE 303.53	83.24 HI 3.6911	77.45 DEFGH 3.7963
	4		Mean HG SD	0.6986 0.0191	91.28 IJ 2.8464	10113 FG 309.56	76.24 KL 3.4677	74.89 HIJKLM 3.2710	12.38 FG 1.2248
	220 °C	2	Mean HG SD	0.6820 0.0089	92.64 I 3.2079	10107 FG 292.56	71.85 M 3.1899	76.89 DEFGHI 3.1238	13.11 EFG 1.5037
			4	Mean HG SD	0.6520 0.0103	80.69 L 3.8151	8779 H 334.69	61.85 O 3.1462	72.06 M 4.3009

HG: Homogeneity groups, SD: Standard deviation, SV: Statistical values, MOR: Bending strength, MOE: Modulus of elasticity, TS: Tensile strength parallel to the grain, CS: Compression strength parallel to the grain, SS: Shear strength parallel to the grain

The mechanical strengths of impregnated and heat treated oak wood were shown Figs. 1, 2, 3, 4, and 5.

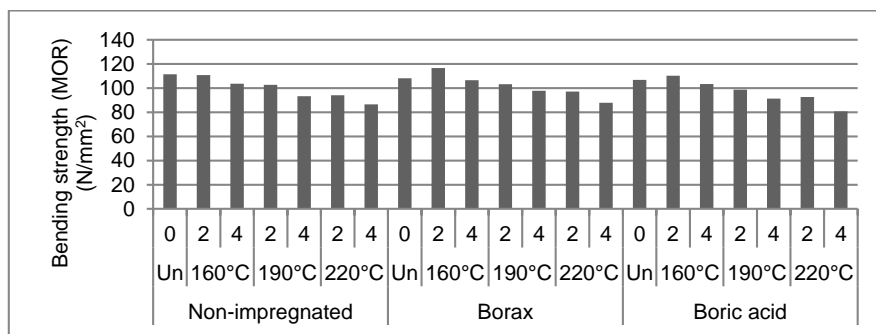


Fig. 1. Bending strength of impregnated and heat-treated oak wood

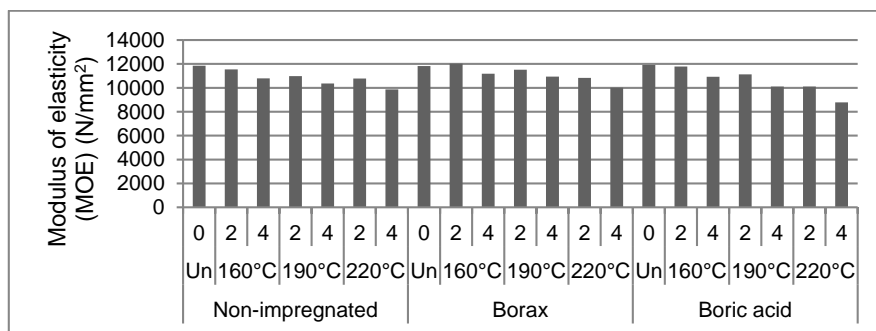


Fig. 2. Modulus of elasticity of impregnated and heat-treated oak wood

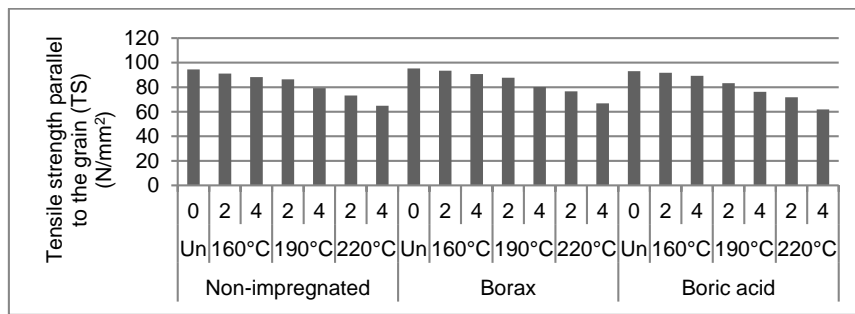


Fig. 3. Tensile strength parallel to the grain of impregnated and heat-treated oak wood

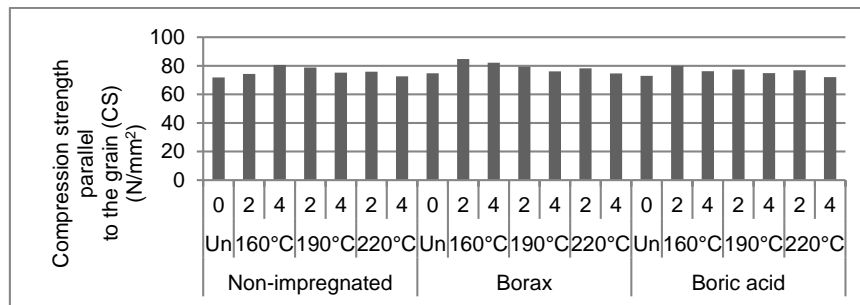


Fig. 4. Compression strength parallel to the grain of impregnated and heat-treated oak wood

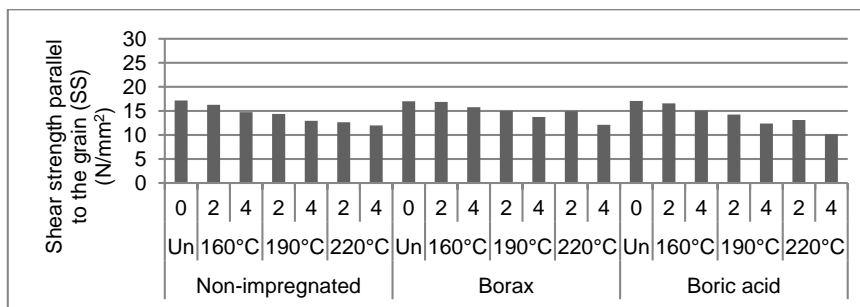


Fig. 5. Shear strength parallel to the grain of impregnated and heat-treated oak wood

Density

According to Table 4, the highest density was obtained in specimens impregnated with borax and untreated samples (0.7430 g/cm^3), and the lowest density was obtained in non-impregnated and untreated samples (0.6463 g/cm^3). The density of the heat-treated wood specimens decreased significantly more than that of the the unheat treated samples. Densities of all the specimens decreased depending on the temperature and duration of the heat treatment. Boonstra *et al.* (2007b) reported a 10% and 8.5% decrease in density for heat-treated Scots pine and Norway spruce, respectively. The results are parallel to previously published reports for different species (Yildiz *et al.* 2006; Korkut *et al.* 2008; Gunduz *et al.* 2009).

Bending strength (MOR)

According to Table 4, the highest MOR was obtained in samples impregnated with borax and heat-treated at 160°C for 2 h (116.60 N/mm^2) and the lowest in samples impregnated with boric acid and heat-treated at 220°C for 4 h (80.69 N/mm^2).

Table 5. Percentage Decrease or Increase of Mechanical Properties in Oak Wood Following Impregnation and Heat Treatment for Different Durations

Impregnation	Heat Treatment (°C)	Time	MOR (%)	MOE (%)	TS (%)	CS (%)	SS (%)
Non-impregnated	160	2	-0.54	-2.59	-3.50	3.43	-5.18
		4	-7.02	-8.97	-6.54	12.31	-14.04
	190	2	-7.74	-7.28	-8.48	9.78	-16.31
		4	-16.21	-12.53	-16.06	4.79	-24.53
	220	2	-15.51	-8.94	-22.44	5.65	-26.34
		4	-22.31	-16.66	-31.31	1.16	-30.18
Borax	Untreated	-	-2.89	-0.23	0.84	4.11	-0.99
	160	2	4.63	1.51	-1.05	18.06	-1.74
		4	-4.33	-5.63	-3.91	14.35	-8.21
	190	2	-7.31	-2.79	-7.19	10.45	-12.52
		4	-12.15	-7.69	-15.12	6.04	-19.87
	220	2	-12.77	-8.63	-18.84	8.99	-12.87
4		-21.08	-15.45	-29.23	4.01	-29.42	
Boric acid	Untreated	-	-4.07	0.52	-1.47	1.58	-0.64
	160	2	-1.05	-0.57	-2.86	11.23	-3.55
		4	-7.23	-7.79	-5.54	6.18	-12.30
	190	2	-11.37	-6.11	-11.86	7.85	-16.95
		4	-18.08	-14.64	-19.28	4.28	-27.85
	220	2	-16.86	-14.69	-23.92	7.07	-23.60
4		-27.58	-25.90	-34.51	0.34	-40.85	

The bending strength (MOR) values showed a small increase in the samples impregnated with borax and heat-treated at 160 °C for 2 h. The highest bending strength (MOR) loss compared to the control was 27.58% (Table 5). Results showed that boron treatments before heat treatment increased the MOR values of wood specimens compared to non-impregnated and heat-treated specimens, except for those impregnated with Boric acid and heat-treated at 220 °C for 2 and 4 h (Table 4). The effects of impregnation chemicals, heat treatment, and heat treatment duration on MOR are given in Table 3. According to Table 3, no statistical difference was found in MOR values between untreated and borax-treated wood. However, there was a statistical difference in MOR values between borax-treated and boric-acid treated wood. The highest MOR was obtained in untreated samples. MOR of all the specimens decreased depending on the temperature and duration of the heat treatment. Toker *et al.* (2009) determined the effects of some boron compounds on modulus of rupture and modulus of elasticity of wood. They reported that MOR values of wood specimens treated with borates were lower than those of untreated control specimens. Yıldız *et al.* (2004) determined the effects of the wood preservatives on mechanical properties of yellow pine. They reported that 2.8% of Wolmanit CX-8 and 7% of ACQ-1900 increased the MOR compared to control specimens, while the other chemicals either decreased or did not affect the MOR. Colakoglu *et al.* (2003) determined that MOR levels of laminated beech veneer lumber impregnated with 5% boric acid were reduced 5.12% compared to untreated control specimens.

Modulus of elasticity in bending (MOE)

According to Table 4, the highest MOE was obtained in samples impregnated with borax and heat-treated specimens at 160 °C for 2 h (12027 N/mm²) and the lowest in those impregnated with boric acid and heat-treated specimens at 220 °C for 4 h (8779 N/mm²). The modulus of elasticity (MOE) had a small increase in impregnated with borax and heat-treated specimens at 160 °C for 2 h, in addition to samples impregnated with borax and untreated specimens. The highest modulus of elasticity in bending loss compared to the control was 25% (Table 5). The modulus of elasticity (MOE) of the wood samples was reduced after the heat treatment. However, losses of MOR and MOE in the impregnated samples with boron compounds, especially borax, were less than the non-impregnated samples. The effects of impregnation chemicals, heat treatment, and heat treatment duration on MOE are given in Table 3. According to Table 3, there was a statistical difference in MOE values between non-impregnated, borax-treated, and boric-acid treated samples. Yıldız *et al.* 2004 reported that 2.8% of Wolmanit CX-8 and 2% of Tanalith E-3491 increased the MOE compared to the control samples. In this study, the MOE of all specimens decreased depending on the temperature and duration of the heat treatment. Can *et al.* (2010) determined the effects of boron impregnation and heat treatment on some physical and mechanical properties of spruce and pine wood. They reported that borax pre-impregnated wood samples provided more reasonable results than boric acid treated ones for strength losses. Colakoglu *et al.* (2003) determined that MOE levels of laminated beech veneer lumber impregnated with 5% boric acid were reduced 3.75% compared to untreated control specimens. Wahab *et al.* (2012) reported that the increase in the hemicellulose and cellulose contents or holocellulose contents causes an increase in the strength (MOR and MOE) of acacia hybrid wood. On the other hand, the reduction of the hemicellulose and lignin contents causes a drop in strength of the hot oil treated acacia hybrid.

Tensile strength parallel to the grain (TS)

According to Table 4, the highest TS was obtained in impregnated with borax and untreated samples (95.25 N/mm²) and the lowest in impregnated with boric acid and heat-treated samples at 220 °C for 4 h (61.85 N/mm²). The highest tensile strength parallel to the grain (TS) loss compared to the control was 34.51% (Table 5). The effects of impregnation chemicals, heat treatment, and heat treatment duration on TS are given in Table 3. According to Table 3, there was a statistical difference in TS values between non-impregnated samples, borax-treated samples, and boric acid-treated samples. The TS of all specimens decreased depending on the temperature and duration of the heat treatment. The highest decrease in TS was observed in samples treated at 220 °C (26.56%). The TS of the heat-treated wood specimens decreased significantly compared to the untreated samples. Boonstra *et al.* (2007b) reported that two-stage heat treatment clearly affects the tensile strength of Scots pine specimens, which is strongly reduced (39%). When tensile stresses occur in wood, the cellulose microfibrils and/or fibrils slide and pull away from one another which requires the breaking of covalent bonds. Degradation of the cellulose polymer, decreasing the DP, was suggested as the main cause for losses in tensile strength (Kass *et al.* 1970; Esteves and Pereira 2009). Korkut *et al.* (2008) reported that the lowest tensile strengths perpendicular to the grain were at 180 °C for 10 h (2.131 N/mm²) in Red-bud maple (*Acer trautvetteri* Medw.) wood. The loss of tensile strength perpendicular to the grain was 46.563%.

Compression strength parallel to the grain (CS)

According to Table 4, the highest CS was obtained in samples impregnated with borax and heat-treated specimens at 160 °C for 2 h (84.78 N/mm²) and the lowest in those impregnated with boric acid and heat-treated at 220 °C for 4 h (72.06 N/mm²). The effects of impregnation chemicals, heat treatment, and heat treatment duration on CS are given in Table 3. According to Table 3, there was a statistical difference in CS values between non-impregnated wood, borax-treated wood, and boric acid-treated wood. The highest CS was obtained in samples impregnated with borax. The CS of all the specimens increased depending on the temperature of heat treatment compared to untreated specimens. The compressive strength parallel to the grain clearly increased after impregnation and heat treatments. Sahin Kol (2010) reported that the compressive strength increased by 4.2% for pine and 17% for fir with heat treatment. The compressive strength parallel to the grain clearly increased after heat treatment (28%), the radial compressive strength decreased (43%), and the tangential compressive strength slightly increased (8%) after heat treatment (Boonstra *et al.* 2007b; Kol 2010). Can *et al.* (2010) determined effects of boron impregnation and heat treatment on some mechanical properties of spruce and pine wood. For this purpose, spruce and pine wood specimens were treated with 4% aqueous solution of both boric acid (BA) and borax (BX), and then the basic thermowood method was applied to the samples as the heat treatment method. Heat treatment was carried out in an industrial furnace at 212 °C for 2 h. As a result, compression strength decreased depending on heat treatment except for the pine specimens impregnated with borax. Colakoglu *et al.* (2003) determined that compression strength in the longitudinal direction levels of laminated beech veneer lumber impregnated with 5% boric acid increased 1.38% compared to untreated control specimens. Vital *et al.* (1983) reported that the compression strength values generally deteriorated with increasing temperature or exposure time.

Shear strength parallel to the grain (SS)

According to Table 4, the highest SS was obtained in non-impregnated and untreated samples (17.16 N/mm²) and the lowest in samples impregnated with boric acid and heat-treated at 220 °C for 4 h (10.15 N/mm²). According to Table 3, the greatest decrease in SS was observed in samples treated at 220 °C. The highest shear strength parallel to the grain (SS) loss compared to the control was 40.85% (Table 5). The SS of the heat-treated wood specimens decreased significantly compared to the un-heat treated samples. According to Stamm (1964), degradation of the hemicelluloses reducing the load-sharing capacity between cellulose microfibrils/fibrils most probably has a negative impact on shear strength (Boonstra *et al.* 2007b). When wood is heated at a high temperature, it becomes more brittle and its mechanical strength decreases, depending on the level and duration of the treatment (Bekhta and Niemz 2003; Korkut 2008; Esteves and Pereira 2009).

In this study, bending strength (MOR), modulus of elasticity in bending (MOE), tensile strength parallel to the grain (TS), and shear strength parallel to the grain (SS) decreased due to heat treatment. The decreases in the mechanical strength can be explained by the rate of thermal degradation and losses of substance after heat treatments. Also, the decreases in mechanical strength may be due to the decomposition of wood components, especially hemicellulose. In accordance with Esteves *et al.* (2008), hemicelluloses are affected first, followed by cellulose and lignin. The decrease in mechanical strength is mainly due to the depolymerization reactions of wood polymers (Kotilainen *et al.* 2000). The primary reason for the mechanical 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). There are chemical changes in the the wood during heating. Chemical changes start by deacetylation of hemicelluloses followed by depolymerization catalysed by the released acetic acid (Tjeerdsma *et al.* 1998; Sivonen *et al.* 2002; Nuopponen *et al.* 2004).

CONCLUSIONS

1. The effects of impregnation chemicals (borax and boric acid) on density and some mechanical properties (bending strength (MOR), modulus of elasticity in bending (MOE), tensile strength parallel to the grain (TS), compression strength parallel to the grain (CS), and shear strength parallel to the grain (SS) of heat treated oak wood were studied.
2. The values of density decreased with increasing heat treatment temperature and time.
3. Compression strength increased with the impregnation and increasing temperatures and durations of the heat treatment. However, as the temperature increased, compression strength showed a declining trend.
4. The bending strength (MOR), modulus of elasticity in bending (MOE), tensile strength parallel to the grain (TS), and shear strength parallel to the grain (SS) decreased due to heat treatment. Shear strength decreased more than tensile strength.
5. The heat treatment temperature was more effective than the heat treatment time on mechanical strength, and borax impregnation had relatively more positive values.

REFERENCES

- Altinok, M., Ozalp, M., and Korkut, S. (2010). "The effects of heat treatment on some mechanical properties of laminated beech (*Fagus orientalis* L.) wood," *Wood Research* 55(3), 131-141.
- Ates, S., Akyildiz, M. H., and Ozdemir, H. (2009). "Effects of heat treatment on calabrian pine (*Pinus brutia* Ten.) wood," *BioResources* 4(3), 1032-1043.
- Awoyemi, L. (2008). "Determination of optimum borate concentration for alleviating strength loss during heat treatment of wood," *Wood Science and Technology* 42(1), 39-45. DOI: 10.1007/s00226-007-0139-4
- Awoyemi, L., and Westermarck, U. (2005). "Effects of borate impregnation on the response of wood strength to heat treatment," *Wood Science and Technology* 39(6), 484-491. DOI: 10.1007/s00226-005-0001-5
- Bekhta, P., and Niemz, P. (2003). "Effect of high temperature on the change in color, dimensional stability and mechanical properties of spruce wood," *Holzforchung* 57(5), 539-546. DOI: 10.1515/hf.2003.080
- Boonstra, M. J., van Acker, J., Kegel, E., and Stevens, M. (2007a). "Optimisation of a two-stage heat treatment process: Durability aspects," *Wood Science and Technology* 41(1), 31-57. DOI: 10.1007/s00226-006-0087-4

- Boonstra, M. J., Van Acker, J., Tjeerdsma, B. F., and Kegel, E. V. (2007b). "Strength properties of thermally modified softwoods and its relation to polymeric structural wood constituents," *Annals of Forest Science* 64(7), 679-690. DOI: 10.1051/forest:2007048
- Can, A., Yildiz, S., Yildiz, U. C., and Tomak, E. D. (2010). "Effects of boron impregnation and heat treatment on some physical and mechanical properties of spruce and pine wood," in: *The 1st International Symposium on Turkish Japanese Environment and Forestry*, Trabzon, Turkey.
- Carrasco, F., and Roy, C. (1992). "Kinetic-study of dilute-acid prehydrolysis of xylan-containing biomass," *Wood Science and Technology* 26(3), 189-208. DOI: 10.1007/BF00224292
- Colakoglu, G., Colak, S., Aydın, I., Yıldız, U., and Yıldız, S. (2003). "Effect of boric acid treatment on mechanical properties of laminated veneer lumber," *Silva Fennica* 37(4), 505-510. DOI: 10.14214/sf.488
- Esteves, B., Graca, J., and Pereira, H. (2008a). "Extractive composition and summative chemical analysis of thermally treated eucalypt wood," *Holzforschung* 62(3), 344-351. DOI: 10.1515/hf.2008.057
- Esteves, B. M., Domingos, I. J., and Pereira, H. M. (2008b). "Pine wood modification by heat treatment in air," *BioResources* 3(1), 142-154.
- Esteves, B. M., and Pereira, H. M. (2009). "Wood modification by heat treatment: A review," *BioResources* 4(1), 370-404.
- Feng, Z., Alén, R., and Niemelä, K. (2002). "Formation of aliphaticcarboxylic acids during soda AQ pulping of kenaf bark," *Holzforschung* 56, 388-394.
- Garrote, G., Dominguez, H., and Parajo, J. C. (2001). "Study on the deacetylation of hemicelluloses during the hydrothermal processing of *Eucalyptus* wood," *Holz als Roh- und Werkstoff* 59(1-2), 53-59. DOI: 10.1007/s001070050473
- Gunduz, G., Korkut, S., Aydemir, D., and Bekar, I. (2009). "The density, compression strength and surface hardness of heat-treated hornbeam (*Carpinus betulus*) wood," *Maderas-Ciencia Y Tecnologia* 11(1), 61-70. DOI: 10.4067/s0718-221x2009000100005
- Gunduz, G., Korkut, S., and Korkut, D. S. (2008). "The effects of heat treatment on physical and technological properties and surface roughness of Camiyani black pine (*Pinus nigra* Arn. subsp pallasiana var. pallasiana) wood," *Bioresource Technology* 99(7), 2275-2280. DOI: 10.1016/j.biortech.2007.05.015
- Hillis, W. E. (1984). "High temperature and chemical effects on wood stability," *Wood Science and Technology* 18(4), 281-293. DOI: 10.1007/BF00353364
- Hodgin, D. A., and Lee, A. W. C. (2002). "Comparison of strength properties and failure characteristics between fire-retardant-treated and untreated roof framing lumber after long-term exposure: A South Carolina case study," *Forest Products Journal* 52(6), 91-94.
- ISO3131. 1975. "Wood -- Determination of density for physical and mechanical tests," International Organization for Standardization. Switzerland.
- ISO3133. 1975. "Wood -- Determination of ultimate strength in static bending," International Organization for Standardization. Switzerland.
- ISO3345. 1975. "Wood -- Determination of ultimate tensile stress parallel to grain," International Organization for Standardization. Switzerland.
- ISO3349. 1975. "Wood -- Determination of modulus of elasticity in static bending," International Organization for Standardization. Switzerland.

- ISO3787. 1976. Wood -- Test methods -- Determination of ultimate stress in compression parallel to grain," International Organization for Standardization. Switzerland.
- Kartal, S. N., Hwang, W.-J., and Imamura, Y. (2008). "Combined effect of boron compounds and heat treatments on wood properties: Chemical and strength properties of wood," *Journal of Materials Processing Technology* 198(1-3), 234-240. DOI: 10.1016/j.jmatprotec.2007.07.001
- Kass, A., Wangaard, F., and Schroeder, H. (1970). "Chemical degradation of wood: the relationship between strength retention and pentosan content," *Wood and Fiber Science* 2(1), 31-39.
- Kol, H. S. (2010). "Characteristics of heat-treated Turkish pine and fir wood after ThermoWood processing," *Journal of Environmental Biology* 31(6), 1007-1011.
- Korkut, D. S., Korkut, S., Bekar, I., Budakci, M., Dilik, T., and Cakicier, N. (2008). "The effects of heat treatment on the physical properties and surface roughness of Turkish Hazel (*Corylus colurna* L.) wood," *International Journal of Molecular Sciences* 9(9), 1772-1783. DOI: 10.3390/ijms9091772
- Korkut, S. (2008). "The effects of heat treatment on some technological properties in Uludag fir (*Abies bornmuelleriana* Mattf.) wood," *Building and Environment* 43(4), 422-428. DOI: 10.1016/j.buildenv.2007.01.004
- Kotilainen, R. A., Toivanen, T. J., Alen, R. J. (2000). FTIR monitoring of chemical changes in softwood during heating. *Journal of Wood Chemistry and Technology*, 20(3), 307-320. DOI: 10.1080/02773810009349638
- Manninen, A. M., Pasanen, P., and Holopainen, J. K. (2002). "Comparing the VOC emissions between air-dried and heat-treated Scots pine wood," *Atmospheric Environment* 36(11), 1763-1768. DOI: 10.1016/s1352-2310(02)00152-8
- Mazela, B., Zakrzewski, R., Grześkowiak, W., and Cofta, G. (2004). "Resistance of thermally modified wood to basidiomycetes," *Wood Technology* 7(1), 253-262.
- Mitchell, P. H. (1988). "Irreversible property changes of small loblolly-pine specimens heated in air, nitrogen, or oxygen," *Wood and Fiber Science* 20(3), 320-335.
- Niemz, P., Hofmann, T., and Retfalvi, T. (2010). "Investigation of chemical changes in the structure of thermally modified wood," *Maderas-Ciencia Y Tecnologia* 12(2), 69-78. DOI: 10.4067/so718-221x2010000200002
- Nuopponen, M., Vuorinen, T., Jamsa, S., and Viitaniemi, P. (2004). "Thermal modifications in softwood studied by FT-IR and UV resonance Raman spectroscopies," *Journal of Wood Chemistry and Technology* 24(1), 13-26. DOI: 10.1081/wct-120035941
- Örs, Y., Atar, M., Keskin, H., and Çolakoğlu, M. H. (2006). "Impacts of impregnation with boron compounds on surface roughness of woods and varnished surfaces," *Journal of Applied Polymer Science* 102(5), 4952-4957. 10.1002/app.24099
- Özçifçi, A. (2008). "Impacts of impregnation with boron compounds on the bonding strength of wood materials," *Construction and Building Materials* 22(4), 541-545. <http://dx.doi.org/10.1016/j.conbuildmat.2006.11.006>
- Peker, H., Sivrikaya, H., Baysal, E., and Yalınkılıç, M. K. (1999). "Static bending strength of wood treated with fire retardant and water repellent preservation chemicals," *Pamukkale University, Journal of Engineering Sciences* 5(1), 975-982.

- Salim, R., Wahab, R., and Ashaari, Z. (2008). "Effect of oil heat treatment on chemical constituents of Semantan bamboo (*Gigantochloa scortechinii* Gamble)," *Journal of Sustainable Development* 1(2), 91-98.
- Sivonen, H., Maunu, S. L., Sundholm, F., Jamsa, S., and Viitaniemi, P. (2002). "Magnetic resonance studies of thermally modified wood," *Holzforschung* 56(6), 648-654. DOI: 10.1515/hf.2002.098
- Sundqvist, B. (2004). "Colour changes and acid formation in wood during heating," Vol. Ph. D., Lulea University of Technology. Sweden.
- Sundqvist, B., Karlsson, O., and Westermark, U. (2006). "Determination of formic acid and acetic acid concentrations formed during hydrothermal treatment of birch wood and its relation to colour, strength and hardness," *Wood Science and Technology* 40(7), 549-561. DOI: 10.1007/s00226-006-0071-z
- Thévenon, M.-F., Tondi, G., and Pizzi, A. (2010). "Friendly wood preservative system based on polymerized tannin resin-boric acid for outdoor applications," *Maderas. Ciencia y Tecnología* 12, 253-257.
- Tjeerdsma, B. F., Boonstra, M., Pizzi, A., Tekely, P., and Militz, H. (1998). "Characterisation of thermally modified wood: Molecular reasons for wood performance improvement," *Holz als Roh- und Werkstoff* 56(3), 149-153. DOI: 10.1007/s001070050287
- Todorovic, N., Popovic, Z., Milic, G., and Popadic, R. (2012). "Estimation of heat-treated beechwood properties by color change," *BioResources* 7(1), 799-815.
- Toker, H. (2007). "Determination of effects of boron compounds on some physical mechanical and biological properties of wood," Vol. PhD, Gazi University, Institute of Science and Technology. Ankara, pp. 213.
- Toker, H., Baysal, E., Simsek, H., Senel, A., Sonmez, A., Altinok, M., Ozcifci, A., and Yapici, F. (2009). "Effects of some environmentally-friendly fire-retardant boron compounds on modulus of rupture and modulus of elasticity of wood," *Wood Research* 54(1), 77-88.
- TS3459. (2012). "Wood - Determination of ultimate shearing stress parallel to grain," Turkish Standards Institution (TSE). Ankara.
- Vital, B., Lucia, R., and Euclides, R. (1983). "Effect of heating on some properties of *Eucalyptus saligna* wood," *Revista-Arvore* 7(2), 136-146.
- Wahab, R., Khalid, I., Sudin, M., Rasat, M., Sulaiman, O., and Tabert, T. A. (2012). "Changes in strength and chemical contents of oil heat treated 15-year-old cultivated acacia hybrid," *International Journal of Chemistry* 4(2), 90-100. DOI: 10.5539/ijc.v4n2p90
- Wang, W., Cao, J., Cui, F., and Wang, X. (2012). "Effect of pH on chemical components and mechanical properties of thermally modified wood," *Wood and Fiber Science* 44(1), 46-53.
- Weiland, J. J., Guyonnet, R., and Gibert, R. (1998). "Analysis of controlled wood burning by combination of thermogravimetric analysis, differential scanning calorimetry and Fourier transform infrared spectroscopy," *Journal of Thermal Analysis and Calorimetry* 51(1), 265-274. DOI: 10.1007/bf02719028
- Winandy, J. E. (1997). "Effects of fire retardant retention, borate buffers, and redrying temperature after treatment on thermal-induced degradation," *Forest Products Journal* 47(6), 79-86.

Yildiz, S., Gezer, E. D., and Yildiz, U. C. (2006). "Mechanical and chemical behavior of spruce wood modified by heat," *Building and Environment* 41(12), 1762-1766. DOI: 10.1016/j.buildenv.2005.07.017

Yildiz, U. C., Temiz, A., Gezer, E. D., and Yildiz, S. (2004). "Effects of the wood preservatives on mechanical properties of yellow pine (*Pinus sylvestris* L.) wood," *Building and Environment* 39(9), 1071-1075. DOI: 10.1016/j.buildenv.2004.01.032

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