Mechanical Properties of Impregnated and Heat Treated Oriental Beech Wood

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The main purpose of this study was to investigate mechanical properties such as the modulus of rupture (MOR) and compression strength parallel to grain (CSPG) of impregnated and heat-treated Oriental beech (*Fagus orientalis* L.) wood. Some copper and boron containing impregnation chemicals such as Wolmanit CX-8 (WCX-8) and Celcure AC-500 (CAC-500) were used. Wood specimens were impregnated 2% aqueous solution of the chemicals according to ASTM D1413-07e1 standard. The wood specimens were heated at 150 and 175 °C for 4 and 8 h, respectively. Results showed that both impregnation and heat treatment decreased the MOR and CSPG of Oriental beech wood. The MOR losses of Oriental beech after both treatments were higher than CSPG losses. The largest reduction of MOR and CSPG were observed with 51.5% and 15.5% for CAC-500 impregnated and heated at 175 °C for 8 h. Except for WCX-8 impregnation and heat treatment at 150 °C for 4 and 8 h, the MOR values of impregnated and heat-treated Oriental beech wood were lower than only heat-treated Oriental beech wood. It was also found that the CSPG values of impregnated and heat-treated Oriental beech wood were higher than only heat-treated Oriental beech wood, except for impregnation and heat treatment at 175 °C for 8 h.

Keywords: Impregnation; Heat treatment; Oriental beech; Modulus of rupture; Compression strength parallel to grain

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INTRODUCTION

Wood is one of the oldest construction materials and is used for a variety of purposes because of its unique properties, including a high strength to weight ratio, resiliency, and toughness (Bultman and Southwell 1976). However, wood is susceptible to environmental degradation because it is a biological material (Williams and Feist 1999; Chang and Chou 2000). Thus, it is necessary to treat wood to provide long service life and improve upon some properties of its intended applications (Srinivas and Pandey 2012). Wood treatments such as impregnation using preservatives and thermal modification techniques can improve wood properties (Kamdem *et al.* 2002). Wood modification is the enhancement of wood properties by chemical, biological, or physical means (Hill 2006; Esteves and Pereira 2008; Thybring 2013). Impregnation, as a wood modification technique, provides dimensional stabilization, protects wood against biological deterioration, and reduces cracking (Kumar 1994). However, chemical treatment presents a serious threat to the environment. Environmental awareness has led to increased interest in developing new methods and chemicals (Percin *et al.* 2015). Thus, new generation wood preservatives such as Celcure AC 500 (CAC-500), micronized copper quat, Tanalith-e, and Adolit KD 5 are less harmful to the environment than previous chemicals containing chromium and arsenic, or they are not harmful at all (Ozgenc *et al.* 2012; Turkoglu *et al.* 2015a). Among the impregnation chemicals, borates have several advantages as wood preservative. In addition to imparting flame retardancy, they provide sufficient protection against wood destroying organisms, and they have a low mammalian toxicity and low volatility. Moreover, they are colorless and odorless (Murphy 1990, Drysdale 1994; Chen *et al.* 1997; Yalinkilic *et al.* 1999). Increased restrictions on the use of conventional heavy-duty wood preservatives have ensured that copper-based formulations gained wide popularity in the wood preservation industry (Freeman and McIntyre 2008). Therefore, they are commonly used in the forest product industry (Turkoglu *et al.* 2015a;b). Wood strength is affected when wood is treated with preservatives or fire retardant chemicals (Winandy 1988). Yildiz *et al.* (2004) investigated modulus of rupture (MOR) of yellow pine (*Pinus sylvestris* L.) wood impregnated with a 2% aqueous solution of Wolmanit CX-8 (WCX-8) which includes copper and borate. They found that MOR of 2% WCX-8 treated yellow pine wood was slightly lower than an untreated control. Toker *et al.* (2008, 2009) reported that compression strength parallel to grain (CSPG) and MOR of borate treated Oriental beech and Scots pine were lower compared to untreated control specimen.

Heat treatment is an alternative wood modification method. While heat-treated wood possesses new properties like improved decay resistance and higher dimensional stability, its strength is considerably decreased (Turkoglu *et al.* 2015c). Heat-treated wood has the advantage in terms of aesthetic properties (uniform and effective change in color) and performance relative to technical guidelines (much reduced swelling and shrinkage, improved resistance to fungi) (Vukas *et al.* 2010; Turkoglu *et al.* 2015c). Heat treatment leads to significant changes in the chemical structure of the wood cell wall components such as cellulose, hemicellulose, and lignin (Sivrikaya *et al.* 2015). As a result of heat treatment, the wood becomes more brittle, and its mechanical strength and technological properties decrease in relation to the level of heat treatment (Gunduz *et al.* 2008). Jämsä and Viitaniemi (2001) reported that at temperatures over 150 °C the strength properties start to weaken and wood becomes more brittle and bending strength decrease by 10 to 30%. In another study, Unsal and Ayrilmis (2005) determined the CSPG of river red gum specimens decreased about 19.0% when heat-treated at 180 °C for 10 h. Therefore, the effects of solely preservative treatment and thermal modification on the mechanical properties of wood are well known; however, combining effects of these treatments has not been widely researched. According to the authors' knowledge, there is limited research and a gap in literature. Baysal *et al.* (2014) researched MOR of Scots pine impregnated with copper and boron containing preservative such as Adolit KD 5 and heat treated. The authors observed a 10.45 to 31.53% decrease in MOR compared with the untreated wood. The present study was conducted on Oriental beech (*Fagus orientalis* L.) wood specimens impregnated with 2% aqueous solution of water-based wood preservatives containing copper and boric acids, such as WCX-8 and CAC-500. After impregnation, heat treatment was applied at 150 °C and 175 °C for 4 and 8 h.

The objective of study was to determine some mechanical properties such as modulus of rupture and compression strength parallel to grain of WCX-8 and CAC-500 impregnated and heat treated Oriental beech wood.

EXPERIMENTAL

Materials

Preparation of test specimens and chemicals

Wood specimens were prepared from Oriental beech wood, which is commonly used in the forest products industry in Turkey. The specimens were selected from air-dried sapwood pieces which were free of knots and with no visible evidence of infection. They were obtained randomly from the wood. Aqueous solution of WCX-8 and CAC-500 were dissolved in distilled water to a concentration of 2%. According to the technical data sheets, WCX-8 contains 2.8% bis-(n-cyclohexyldiazeniumdioxy)-copper, 13.0% copper (II) carbonate hydroxide, and 4.0% boric acid (Wolman 2007). CAC-500 contains an alkaline copper quaternary system, including 16.63% copper (II) carbonate hydroxide, 4.8% benzalkonium chloride, and 5.0% boric acid (Ozgenc and Yildiz 2014).

Methods

Impregnation process

Wood specimens were impregnated with 2% aqueous solution of WCX-8 and CAC-500 according to ASTM D1413-07e1 (2007). While the specimens tested by CSPG were oven dried at 103 ± 2 °C until unchangeable weight before impregnation, the specimens tested by MOR were oven dried at 55 ± 2 °C until unchangeable weight before impregnation to prevent cracking, deflection, and distortion *etc.* at higher temperatures. Retention was calculated from Eq. 1,

$$Retention \left(\frac{kg}{m^3}\right) = \frac{G\ x\ C}{V}\ x\ 10 \tag{1}$$

where $G = (T_2 - T_1)$ is the grams of treatment solution absorbed by the wood specimens (T_1 is the weight of the wood specimens before impregnation, T_2 is the weight of the wood specimens after impregnation), C is grams of preservative in 100 g of the treatment solution, and V is the volume of the wood specimen in cm^3. Impregnated wood specimens were conditioned at 20 °C and 65% relative humidity for two weeks before heat treatment.

Heat treatment

Heat treatment was performed using a temperature-controlled laboratory oven. Two different temperatures (150 and 175 °C) and two treatment durations (4 and 8 h) were applied to wood specimens under atmospheric pressure and in the presence of air. Heat treated wood specimens were conditioned at 20 °C and 65% relative humidity for two weeks before MOR and CSPG tests.

Modulus of rupture (MOR)

The modulus of rupture of wood specimens was examined according to TS 2474 (1976). Dimensions of air-dried sapwood specimens of Oriental beech were 20 (radial) × 20 (tangential) × 360 (longitudinal) mm for MOR test. The MOR of wood specimens impregnated with WCX-8 and CAC-500 was calculated using Eq. 2,

$$MOR\ \left(\frac{kg}{cm^2}\right) = \frac{3\ x\ P\ x\ I}{2\ x\ b\ x\ h^2} \tag{2}$$

where P is the maximum load (kg), I is the span (cm), b is the width of specimen (cm), and h is the thickness of specimen (cm).

Compression strength parallel to grain test (CSPG)

The compression strength parallel to grain test was determined according to the TS 2595 (1977) standard using a 4000-kp capacity universal test machine, and applying 6 mm/min loading time. Dimensions of air-dried sapwood specimens of Oriental beech were 20 (radial) × 20 (tangential) × 30 (longitudinal) mm for CSPG test.

Evaluation of test results

Mechanical test results were evaluated by one-way ANOVA using the SPSS statistical program. The significance ($P<0.05$) between the treatments was compared using Duncan's homogeneity groups. Different letters given with the average values of the tested parameters indicated a significant difference according to Duncan's homogeneity groups.

RESULTS AND DISCUSSION

MOR of Impregnated and Heat-treated Oriental Beech Wood

Table 1 shows WCX-8 and CAC-500 retention levels and MOR of Oriental beech wood. The retention of WCX-8 and CAC-500 were 12.25 and 12.43 kg/m^3, respectively. The highest MOR value was recorded as 1197 kg/cm^2 for untreated Oriental beech, and the lowest MOR value was 580 Kg/cm^2 for CAC-500 impregnated and heat-treated at 175 °C for 8 h. The MOR values of untreated wood specimens were higher than those of WCX-8 and CAC-500 impregnated wood specimens. WCX-8 and CAC-500 treatment decreased the MOR of Oriental beech by approximately 15%. There was a significantly statistical difference in MOR levels between untreated wood and preservative treated wood specimens. However, there was no statistical difference in MOR levels between WCX-8 treated and CAC-500 treated Oriental beech wood.

In this study, the waterborne preservatives WCX-8 and CAC-500 chemicals were used. Different research studies have shown that some preservatives, especially waterborne preservatives, have a negative impact on mechanical properties of the wood (Mourant *et al.* 2008; Toker *et al.* 2008; Simsek *et al.* 2010; Simsek *et al.* 2013). Many of the metallic oxides commonly used in waterborne preservative formulations do react with the cell wall components by undergoing hydrolytic reduction upon contact with wood sugars. This process, known as fixation, oxidizes the wood cell wall components and may reduce wood strength (Winandy 1988). The relative impact of various waterborne preservative systems is directly related to the system's chemistry and the severity of its fixation/precipitation reaction (Winandy 1996). Simsek *et al.* (2013) determined that the MOR of Oriental beech and Scots pine decreased after impregnation with copper- and boron-based impregnation chemicals. Yildiz *et al.* (2004) investigated the effects of a copper-based wood preservative, such as ACQ-2200 treatment, on MOR. They found that there was a significant difference in MOR levels between untreated wood and ACQ-2200 impregnated wood. In another study, Baysal *et al.* (2014) studied the MOR of Scots pine impregnated with a copper and boron contained chemical, such as Adolit KD 5. They found that the MOR values of wood specimens impregnated with Adolit KD 5 were lower than that of the un-impregnated (control) specimen. Toker *et al.* (2009) investigated the MOR of Calabrian pine and Oriental beech impregnated with aqueous solutions of borates. They found that MOR levels of both wood specimens were lower than their corresponding un-impregnated wood specimens. Simsek *et al.* (2010) studied the MOR of Scots pine and

Oriental beech wood specimens impregnated with aqueous solutions (0.25, 0.50, 1.50, and 3.00%) of borates. They found that borate impregnation decreased MOR levels of both wood specimens. Moreover, the higher concentration levels of borates resulted in lower MOR for both wood specimens (Simsek *et al.* 2010). The results reported here are consistent with the findings of the aforementioned studies.

Table 1. MOR of Impregnated and Heat-treated Oriental Beech Wood

Treatment	Retention (kg/m^3)	Heat Treatment (ºC)	Time (h)	MOR* (kg/cm^2)			
				Mean	SD	HG	Change (%)
Control	-	-	-	1197	135	A	-
WCX-8	12.25	-	-	1017	170	CD	-15.0
CAC 500	12.43	-	-	1012	225	CD	-15.5
Solely heat treatment	-	150	4	1105	80	ABC	-7.7
		150	8	1051	75	ABCD	-12.2
		175	4	933	84	DE	-22.1
		175	8	812	90	EF	-32.2
WCX-8	12.25	150	4	1180	133	AB	-1.4
		150	8	1057	196	ABCD	-11.7
		175	4	678	114	FGI	-43.4
		175	8	639	101	GI	-46.6
CAC 500	12.43	150	4	1077	92	ABCD	-10.0
		150	8	1040	254	BCD	-13.1
		175	4	735	173	FG	-38.6
		175	8	580	116	I	-51.5

* Different letters reflect statistical significance at the 95% confidence level. Ten replicates were made for each treatment group. HG: Homogeneity groups; SD: Standard deviations

The results of this study showed that heat treatments decreased the MOR of Oriental beech wood specimens. The findings from previous studies on heat-treated wood MOR are not always compatible with each other because heat treatment temperature, treatment time, size of specimen, treatment method, and chemical composition of wood affect MOR loss in heat-treated wood. In this study, heat treatments decreased the MOR of Oriental beech by 7.7 to 32.2%. Generally, the results of this study on the effect of heat treatment on the MOR of Oriental beech are compatible with the findings of previous research related to the effects of heat treatment on MOR. When heating wood without oxygen, hemicellulose is degraded first, followed by cellulose, and finally lignin. Therefore, heat-treated wood has a higher percentage share of lignin than normal wood (Vukas *et al.* 2010). Hemicelluloses are more affected than other constituents of wood due to their relatively lower thermal stabilities (da Silva *et al.* 2015). Changes in hemicellulose determine the strength properties of woods heated at high temperatures (Hills 1984). The first reason for the loss of strength is the degradation of hemicelluloses, which are not as stable to the heat as cellulose and lignin. The close relationship between hemicellulose content and bending strength was reported by a number of researchers (Winandy and Morrell 1993; Winandy and Lebow 2001; Esteves and Pereira 2008). Kartal *et al.* (2007) found a relationship between strength and the hemicellulose content of specimens. They reported that a lower hemicellulose content in the specimens resulted in lower MOR of wood specimens. The current study showed that the MOR values of Oriental beech wood specimens decreased with increasing treatment temperature and duration. This result is consistent with previous studies (Gunduz and Aydemir 2009; Korkut and Hiziroglu 2009; Aydin *et al.* 2015).

In this study, the MOR decreased 1.4 to 46.6% for WCX-8 treated and heat-treated Oriental beech. The MOR decreased 10.0 to 51.5% for CAC-500 treated and heat-treated Oriental beech. Baysal *et al.* (2014) investigated the MOR of Adolit KD 5 impregnated into Scots pine (*Pinus sylvestris* L.) wood specimens that were subsequently heat-treated. They found that the MOR levels of impregnated and heated Scots pine were lower than that of the un-impregnated and un-heated Scots pine wood specimens. Moreover, they found that the MOR values of Scots pine wood specimens decreased with increasing treatment temperature and duration. The results of the present study are in good agreement with the data provided by Baysal *et al*. (2014). According to these results, except for WCX-8 impregnation and heat treatment at 150 °C for 4 and 8 h, combining impregnation and thermal modification resulted in lower MOR of Oriental beech, compared to only heat treated Oriental beech wood. For example, while the only heat treatments group decreased 7.7 to 32.2% MOR of Oriental beech, combining impregnation and heat treatments decreased the MOR of Oriental beech by 1.4 to 51.5%. The decrease in MOR of Oriental beech wood specimens may be due to the combined effect of chemicals and heat treatment.

CSPG of Impregnated and Heat-treated Oriental Beech

The compression strength parallel to grain (CSPG) and retention values are given in Table 2. The retention of WCX-8 and CAC-500 were 10.26 and 10.87 kg/m^3, respectively. The compression strength parallel to grain value of untreated beech was higher than treated Oriental beech wood. The highest CSPG value was 714 kg/cm^2 for untreated Oriental beech. The lowest CSPG was 603 kg/cm^2 for CAC-500 impregnated and heat-treated at 175 °C for 8 h. These results showed that preservative treatments decreased the CSPG values of Oriental beech wood specimens.

Some preservatives, especially waterborne preservatives, have a negative impact on the mechanical properties of wood (Mourant *et al.* 2008). It can be said that preservative impregnation increased the rate of hydrolysis in the wood, thereby causing loss in strength. Bal (2006) studied the CSPG of Scots pine impregnated with a copper-based wood preservative such as ACQ. He found that ACQ treatments decreased 1 to 3% of CSPG of Scots pine. However, there was no significant difference between untreated and ACQ treated wood specimens in this study. Simsek *et al.* (2013) reported that borate preservative treatments decreased 7.69 to 9.98%, and 7.88 to 10.87% of CSPG for Oriental beech and Scots pine, respectively. This study showed that WCX-8 and CAC-500 treatment decreased the CSPG of Oriental beech by 5.7 and 10.0%, respectively. However, there was no statistical difference between untreated Oriental beech and treated Oriental beech. Furthermore, heat treatments decreased the CSPG of Oriental beech wood specimens. Heat treatments decreased the CSPG of Oriental beech 2.5 to 7.0%. Aydin *et al.* (2015) reported that percentages of compression strength losses for 2, 6, and 10 h were 4.32, 15.92, and 18.85% at 185 °C for Oriental beech wood. In another study, Korkut *et al.* (2009) determined that percentages of compression strength losses for 2, 6, and 10 were 10.36, 11.94, and 16.31% at 180 °C for European hophornbeam (*Ostrya carpinifolia* Scop.) wood. Gunduz *et al.* (2009) reported that the compression strength losses for 170 °C and 4 h was 7%, while for 210 °C and 12 h, it was 34.7%.

Table 2. CSPG of Impregnated and Heated Oriental Beech

Treatment	Retention (kg/m^3)	Heat Treatment (ºC)	Time (h)	CSPG* (kg/cm^2)			
				Mean	SD	HG	Change (%)
Control		-	-	714	83	A	-
WCX-8	10.26	-	-	673	78	AB	-5.7
CAC 500	10.87	-	-	642	63	AB	-10.0
Solely heat treatment	-	150	4	696	66	A	-2.5
		150	8	682	35	A	-4.5
		175	4	676	53	AB	-5.3
		175	8	664	38	AB	-7.0
WCX-8	10.26	150	4	708	64	A	-0.8
		150	8	684	61	A	-4.2
		175	4	691	89	A	-3.2
		175	8	646	77	AB	-9.5
CAC 500	10.87	150	4	698	84	A	-2.2
		150	8	684	71	A	-4.2
		175	4	689	68	A	-3.5
		175	8	603	95	B	-15.5

* Different letters reflect statistical significance at the 95% confidence level. Ten replicates were made for each treatment group. HG: Homogeneity groups; SD: Standard deviations

The effect of thermal modification on mechanical properties of wood is complex, and the magnitude of this effect is a function of parameters such as exposure time, temperature, medium rate of heating, and the moisture content of wood (Yildiz *et al.* 2006; Korkut and Hiziroglu 2009). The decrease in compression strength was due to the holocellulose content degradation, in which the first constituent affected was probably the hemicellulose (da Silva *et al.* 2013). Nuopponen (2005) found that thermally treated wood specimens showed higher lignin contents than unheated wood specimens, which was the result of the degradation of hemicellulose.

This study showed that CSPG values of Oriental beech wood specimens decreased with increasing treatment temperature and duration. This result is consistent with previous studies (Yildiz 2002; Gunduz *et al.* 2009; Aydin *et al.* 2015). In this study, while CSPG decreased 0.8 to 9.5% for WCX-8 treated and heat-treated Oriental beech, it decreased 2.2 to 15.5% for CAC-500 treated and heat-treated Oriental beech. Percin *et al.* (2015) investigated CSPG values of oak (*Quercus petraea* Liebl.) wood impregnated with borates and heat treated. They found that CSPG of oak wood clearly increased after impregnation and heat treatments. In another study, Can *et al.* (2010) studied CSPG of boric acid impregnated and then heated at 212 °C for 2 h. They found that CSPG values of both wood specimens decreased after treatments. According to these results, except for impregnation and heat treatment at 175 °C for 8 h, combining this impregnation and thermal modification caused a higher CSPG of Oriental beech compared to Oriental beech wood that was only heat-treated.

CONCLUSIONS

1. The MOR and CSPG of unheated Oriental beech (control) were higher than heat-treated Oriental beech. Thus, higher treatment duration and temperature resulted in lower MOR and CSPG of Oriental beech.
2. Except for WCX-8 impregnation and heat treatment at 150 °C for 4 and 8 h, combination of impregnation and thermal modification caused lower MOR of Oriental beech compared with only heat-treated Oriental beech wood.
3. Except for impregnation and heat treatment at 175 °C for 8 h, combination of impregnation and thermal modification caused higher CSPG of Oriental beech compared with only heat-treated Oriental beech wood.
4. Impregnation and then heat treatment caused a greater decrease in MOR than in the CSPG of Oriental beech.
5. Preservative treatment and then heat treatment at 150 °C for 4 and 8 h decreased 1.4 to 13.1% of MOR for Oriental beech wood and it decreased 0.8 to 4.2% of CSPG for Oriental beech. As the National Design Specification for Wood Construction (NFPA) requires a 10 to 20% reduction in allowable design stress, these treatments met the NFPA requirements for design purposes. However, MOR of preservative treated and then heat-treated at 175 °C for 4 and 8 h Oriental beech did not met the NFPA requirements for design purposes.
6. In all treatment applications, the lowest MOR and CSPG losses were obtained from Oriental beech wood impregnated with WCX-8 and heat treatment at 150 °C for 4 h.
7. Higher temperature and durations and then preservative treatment of structural members for applications when strength is a dominant factor is usually not recommended.
8. Preservative impregnation before heat treatment is recommended only where a relatively mild heat treatment is involved.

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