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SOME PROPERTIES OF TURKISH SWEETGUM BALSAM (*STYRAX LIQUIDUS*) IMPREGNATED ORIENTAL BEECH WOOD
PART II: DECAY RESISTANCE, MECHANICAL, AND THERMAL PROPERTIES

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ABSTRACT

This study was designed to investigate decay resistance, some mechanical properties such as modulus of rupture (MOR), modulus of eleasticity (MOE), compressison strength parallel to grain (CSPG) and thermal characteristics of Oriental beech wood impregnated with Turkish Sweetgum Balsam (TSB). Wood specimens were impregnated with 2, 4, and 6 percent ethanol solutions of TSB according to ASTM D1413, 2007 standard before tests.

Our results showed that TSB impregnation improved the level of decay resistance of Oriental beech wood considerably. MOR, MOE and CSPG values were lower than that of the untreated control specimen. Moreover, higher concentration levels of TSB resulted in lower MOR, MOE, and CSPG values of Oriental beech wood. The highest residual char yield was obtained with 2 % TSB impregnated Oriental beech wood after thermal test.

KEYWORDS: Oriental beech, Turkish sweetgum balsam, decay resistance, mechanical properties, thermal properties, impregnation.

INTRODUCTION

Wood is a good engineering and structural material because of good mechanical strength, low thermal expansion, and aesthetic appeal (Srinivas and Pandey 2012). It has been popularly and favorably used as a decorative material owing to its aesthetic appearance and characteristics properties (Chang and Chang 2001). However, wood has some weak properties which can be modified with wood preservatives (Lahtela et al. 2014, Turkoglu et al. 2015). A major problem of the wood preservatives such as creosote, pentachlorophenol and inorganic arsenicals is that they pose a serious threat to the environment (Hsu et al. 2007). Thus, an environmentally important task for the future is to use non-toxic preservatives for protection of wood against biodegradation (Ulvcrona et al. 2006). Much research has been carried out on environmentally-friendly and natural wood preservatives to protect wood against biotic and abiotic factors. Some examples of this natural wood preservatives are Eucalyptus oil (Batish et al. 2008), tall oil (Temiz et al. 2008), octyl gallate (Hsu et al. 2007), linseed oil (Megnis et al. 2002), guayule (Nakayama et al. 2001), heartwood extracts of *Milicia excelsa* and *Erythrophleum suaveolens* (Onuorah 2000) and carvacrol (Kai 1991). Turkish sweetgum balsam (TSB) or *Styrax liquidus*, used in cosmetic and pharmaceutical industries (Ozturk et al. 2008), is a natural, organic, hydrophobic and fungi decay prevention substance which can be used as a wood preservative (Kartal et. al. 2012, Degirmentepe 2014).

Liquidambar orientalis Miller is part of the *Hamamelidaceae* family and is endemic to Turkey. It exists in coastal area of the southwestern Turkey (Sagdic et al. 2005, Ozturk et al. 2008). TSB is resinous exudate obtained from the wounded trunk of *Liquidambar orientalis* (Gurbuz et al. 2013). It is known as *Styrax liquidus*, Levant storax, Turkish sweetgum, and Oriental sweetgum (Baytop 1999, Pesmen 1997). TSB consists of resin alcohols avaiblable free and combined with cinnamic acid, which makes up 30–45 % of the total weight (Hafizoglu 1982). Detailed chemical composition of TSB was styrene (1.56); a-pinene (1.02); benzaldehyde (0.47); b-pinene (0.15); benzyl alcohol (1.22); acetophenone (0.19); 1-phenyl-1-ethanol (0.17); hydrocinnamyl alcohol (41.13); trans-cinnamyl aldehyde (0.24); trans-cinnamyl alcohol (45.07) and bcaryophyllene (3.60 %) (Lee et al. 2009). The production of TSB is approximately 2000 tons per year in Turkey and its price per kilogram is between 35 and 40 dollars in domestic market. It is assumed that the TSB will be used 2-6 % concentrations in impregnation process; it has the potential of economical wood preservative (Degirmentepe et al. 2015).

Our previous study on some physical properties of wood impregnated with TSB showed that wood became less hygroscopic and the color of wood tended to darken due to the impregnation with the TSB (Degirmentepe et al. 2015). Another research about biological performance properties of TSB was carried out by Kartal et al. (2012). They found that Scots pine sapwood impregnated with TSB generally displayed moderate resistance to fungal decay by two white-rot fungi. Terzi et al. (2012) explained that the heartwood of the *Liquidambar orientalis* (contains natural balsam) was not resistant against the fungi tested; however, the wood was resistant against termites and furniture beetle larvae in laboratory conditions. Therefore, detailed studies are needed to evaluate TSB as a wood preservative. The purpose of this study was to evaluate its decay resistance, some mechanical properties such as modulus of rupture (MOR), modulus of eleasticity (MOE), compressison strength parallel to grain (CSPG) and thermal characteristics of TSB impregnated Oriental beech wood.

MATERIAL AND METHODS

Preparation of test specimens and chemicals

Wood specimens measuring 20 (radial) x 20 (tangential) x 360 (longitudinal) mm, for MOR and MOE tests, and 20 (radial) x 20 (tangential) x 30 (longitudinal) mm for CSPG test were prepared from air-dried sapwood of Oriental beech (*Fagus orientalis* L.). For decay test, wood specimens measuring 15 (radial) x 25 (tangential) x 50 (longitudinal) mm were prepared from air-dried sapwood of Oriental beech. Fungal decay test was made using a white rot fungus, *Coriolus versicolor*. TSB was dissolved in ethanol to concentrations of 2, 4, and 6 %. Wood specimens were oven dried at 103 ± 2°C before and after treatment. For the thermal test, wood flour was prepared by grinding the small wood pieces in a Wiley mill with a 50 meshes.

Impregnation method

Wood specimens were impregnated with 2, 4, and 6 percent ethanol solutions of TSB according to ASTM D1413-07e1 (2007) for mechanical and decay tests. Retention was calculated with the following equation:

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

where: G - the amount of solution absorbed by wood that is calculated by T_2 - T_1,
T_2 - weight of wood after impregnation,
T_1 - weight of wood before impregnation,
C - solution concentration as percentage,
V - the volume of the specimen as cm^3.

For the thermal test, the wood preservatives having concentrations of 2, 4, and 6 % were prepared using ethanol for the impregnation procedure. Approximately 100 g of wood flour was immersed in the solutions at 60°C for 2 h. The treated samples were subsequently dried at 60°C until they had the unchangeable weight. A similar impregnation procedure of wood flour and wood samples was described in TG and DTA studies on fire retardant treated wood by Jiang et al. (2010) and Yunchu et al. (2000).

Decay test

Test results were expressed as a percentage of mass losses of wood specimens due to fungal attacks after decay test. The mass loss of wood specimens after decay test was calculated from the following formula:

$$\text{Mass loss} = (T_3 - T_4)/T_3 \times 100 \tag{2}$$

where: T_3 - mass of wood specimen before exposure to test fungi (g),
T_4 - mass of wood specimen after exposure to test fungi (g).

Modulus of rupture and modulus of elasticity

The MOR and MOE of wood specimens were determined according to TS 2474 (1976) and TS EN 310 (1999), respectively. Wood specimens had been conditioned at 20°C and 60 % RH for two weeks prior to testing. The MOR and MOE of wood specimens were calculated by using the following formulas;

$$MOR = \frac{3 \times P \times I}{2 \times b \times h^2} \quad \left(\frac{N}{mm^2}\right) \tag{3}$$

$$MOE = \frac{P \times I^3}{4 \times b \times h^3 \times Y} \quad \left(\frac{N}{mm^2}\right) \tag{4}$$

where: P - the maximum load (N),
I - the span (mm),
b - the width of specimen (mm),
h - the thickness of specimen (mm),
P - the load to proportional limit (mm),
Y - the deflection (mm).

Compression strength parallel to grain

The CSPG test was determined according to the TS 2595 (1977) standard by using a 4000-kp capacity universal test machine, and applying 6 $mm.min^{-1}$ loading time. Before tests, wood specimens had been conditioned at 20°C and 60 % RH for two weeks.

Thermal analysis

Differential thermal analysis (DTA) and thermogravimetry (TG) were carried out under argon at a heating rate of 10°$C.min^{-1}$ and a purge rate of 50 $mL.min^{-1}$ using a LABSYS TG-DTA analyzer (France). The temperature was raised from the room temperature up to 600°C. During the heating and pyrolysis of about 10 mg of sample, the mass loss was monitored continuously. Onset and inflection temperatures of the pyrolysis were recorded by the analyzer for each treatment group. The rate of mass loss as a function of time was derived from a TG curve resulting in a derivative TG curve.

Evaluations of test results

Mechanical and decay test results were evaluated by a computerized statistical program composed of analysis of variance and following Duncan tests at the 95 % confidence level. Statistical evaluations were made on homogeneity groups (HG), of which different letters reflected statistical significance.

RESULTS AND DISCUSSIONS

Decay resistance

Results of the decay test of Oriental beech impregnated with TSB after a decay test for 12 weeks are given in Tab. 1.

Tab. 1: The mass loss of Oriental beech wood specimens impregnated with TSB after decay test.

Impregnation material	Conc.%	Oriental beech					
		Retention (kg.m^{-3})		Mass loss* (%)			
		Mean	SD	Mean	SD	HG	Change** (%)
Control	-	-	-	31.66	5.33	A	-
TSB	2	6.50	0.51	15.41	3.26	B	-51.33
	4	18.03	0.98	5.51	0.91	C	-82.60
	6	23.88	3.64	3.30	0.64	C	-89.58

*Ten replicates were made for each treatment group, **Compared to control, SD: Standard deviation, Conc: Concentration, HG: Homogeneity groups obtained by statistical analysis with similar letters reflecting statistical insignificance at the 95 % confidence level.

TSB impregnated wood showed considerable resistance to the decay fungus compared to that of the untreated control specimen, especially 4 and 6 % impregnated wood specimens. While the highest mass loss was obtained as 31.66 % for untreated Oriental beech, the lowest mass losses were recorded as 3.30 with 6 % TSB impregnated Oriental beech. Our results showed that higher concentration levels of TSB resulted in lower mass losses of Oriental beech. There is a statistical difference in mass loss between the control and TSB impregnated Oriental beech. However, there is no statistical difference in mass loss between 4 and 6 % TSB impregnated Oriental beech. Terzi et al. (2012) investigated decay resistance of heartwood specimens of *Liquidambar orientalis* and sapwood specimens of Scots pine according to ASTM D2017 (2014) classification based on mass losses in wood specimen. They found that mass losses of heartwood specimens of *Liquidambar orientalis* and sapwood of Scots pine specimens were 37.08 and 40.95 %, respectively. According to ASTM D2017, 2014 both wood specimens were non-resistant against white rot fungus, *Trametes versicolor*. In our study, mass loss of untreated Oriental beech wood was 31.66 % after the decay test. Although our mass loss was lower than Terzi et al. (2012) findings, it was non-resistant against *C. versicolor* according to ASTM D2017 (2014). Kartal et al. (2012) investigated that biological performance properties of balsam from *Liquidambar orientalis* trees showed that balsam-treated pine sapwood specimens generally displayed moderate resistance to fungal decay by two white-rot fungi, based on the ASTM D2017 (2014) classification. According to our results, while the biological performance of 2 % TSB impregnated Oriental beech showed resistance to *C. versicolor*, 4 and 6 % TSB impregnated Oriental beech showed highly resistance to *C. versicolor* based on the ASTM D2017 (2014) classification. In the literature, there are some studies about required retention levels of borates which are commonly used for protection against decay fungi. For example, Yalinkilic et al. (1996) reported that 2.5 kg.m^{-3} boric acid loading was found sufficient for decay resistance against *C. versicolor*. Tsunoda (2001) reported that that the toxic threshold values for sugi sapwood specimens treated with boric acid were 0.8 kg.m^{-3} against boric acid equivalent against *T. versicolor*. Also, Kartal et al. (2007) reported that European standards usually require disodium octaborat tetrahydrate retention levels of 0.76 kg.m^{-3} for protection against *T. versicolor*. In our study, TSB retentions were calculated as 6.50, 18.03, and 23.88 kg.m^{-3} for 2, 4, and 6 % TSB, respectively. These values loading TSB treated wood at high levels could resist wood-decaying fungi according to the researchers' findings which were mentioned above.

Modulus of rupture, MOE, and CSPG values of TSB impregnated Oriental beech

The MOR and MOE values of TSB impregnated Oriental beech are given in Tab. 2. The highest MOR and MOE values were obtained as 113.8 and 11507 N.mm^{-2}, for untreated Oriental beech, respectively. The lowest MOR and MOE values were recorded as 101.8 and 10038 N.mm^{-2} for 6 % TSB impregnated Oriental beech, respectively.

Tab. 2: MOR and MOE tests results of Oriental beech wood specimens impregnated with TSB.

Impregnation material	Conc. %	Retention (kg.m^{-3})		MOR* (N.mm^{-2})				MOE* (N.mm^{-2})			
		Mean	SD	Mean	SD	HG	Change (%)	Mean	SD	HG	Change** (%)
Control	-	-	-	113.8	13.0	A	-	11507	1152	A	-
TSB	2	6.96	1.20	111.7	8.6	AB	-1.85	11272	864	A	-2.04
	4	16.78	5.88	104.2	9.9	AB	-8.44	10433	1020	A	-9.33
	6	19.11	5.70	101.8	10.3	C	-10.54	10038	1067	A	-12.77

*Ten replicates were made for each treatment group, **Compared to control, MOR: Modulus of rupture, MOE: Modulus of elasticity, SD: Standard deviation, Conc: Concentration, HG: Homogeneity groups obtained by statistical analysis with similar letters reflecting statistical insignificance at the 95 % confidence level.

Researches have shown that some preservatives, especially waterborne preservatives, have a negative impact on mechanical properties of the wood (Mourant et al. 2008) whereas as a water repellent type preservative such as resin impregnation into wood caused an increase in the mechanical properties of wood (Pizzi 2003). Moreover, Deka and Saika (2000) and Pizzi (2003) reported that impregnation of wood with thermosetting resins increased MOR and MOE of wood. Mourant et al. (2008) investigated that mechanical properties of phenol formaldehyde (PF) - pyrolytic oil resin and CCA treated Jack pine and sugar maple wood. They found that treatment with PF-pyrolytic oil resin resulted in similar or slightly better mechanical properties when compared to CCA-treated wood. Our results showed that TSB impregnation decreased the MOR and MOE values of wood specimens compared to untreated control. It may be due to impregnation could affect moisture content of wood speciemens. Higher concentration levels of TSB resulted in lower MOR and MOE values of Oriental beech wood. Furthermore, it is known that mechanical properties of the wood can be altered by retention of wood preservative (Winandy 1995). Yildiz et al. (2004) investigated MOR and MOE of yellow pine impregnated with some copper based chemicals. They found that while MOR was dependent on the retention of the chemical, the retention of chemical had no noticeable effect on MOE. According to our results, no significant differences were found in MOR values between the untreated control and 2 and 4 % TSB impregnated Oriental beech. However, there is a statistical difference in MOR between the control and 6 % TSB impregnated Oriental beech. Also, there are no statistical differences in MOE values between the untreated control and TSB impregnated Oriental beech. MOE measures the stiffness of a material, MOR has been shown as a more reliable measure of strength than stress at the proportional limit. This is generally because the maximum load can be determined more precisely than the proportional limit. Moreover, MOR can be more constant as it is affected less by previous loads applied or by the conditions of testing (Yalinkilic 2000). Thus, the importance of MOR is that it expresses the greatest load the wood will carry (Brown et al. 1952). Therefore, MOR and MOE values have crucial importance for designing wood constructions (Yildiz et al. 2004). The compression strength values of TSB impregnated Oriental beech wood are given in Tab. 3.

Tab. 3: CSPG of Oriental beech wood specimens impregnated with TSB.

Impregnation material	Conc. %	Retention ($kg.m^{-3}$)		CSPG* ($N.mm^{-2}$)			
		Mean	SD	Mean	SD	HG	Change** (%)
Control	-	-	-	59.12	8.52	A	-
TSB	2	7.52	0.52	58.68	7.59	A	-0.74
	4	18.84	2.35	58.11	5.78	A	-1.71
	6	30.72	3.17	56.67	7.31	A	-4.14

*Ten replicates were made for each treatment group, **Compared to control, CSPG: Compression strength parallel to grain test, SD: Standard deviation, Conc: Concentration, HG: Homogeneity groups obtained by statistical analysis with similar letters reflecting statistical insignificance at the 95 % confidence level.

The compression strength parallel to grain value of untreated beech was higher compared to treated Oriental beech. While the highest CSPG values of Oriental beech were recorded as 59.12 $N.mm^{-2}$ for untreated wood, the lowest CSPG values were obtained as 56.67 $N.mm^{-2}$ for 6 % TSB impregnated Oriental beech. Our results showed that TSB impregnation decreased CSPG values of Oriental beech. Also, higher concentration levels resulted in lower CSPG of Oriental beech. However, no significant differences were found between control and TSB impregnated Oriental beech. Also, there are no significant differences among all TSB impregnated Oriental beech wood. The National Design Specification for Wood Construction requires a 10–20 % reduction in allowable design stress, depending on mechanical property under consideration (NFPA 1986). Our results showed that TSB treatment decreased 1.85 - 10.54, 2.04 - 12.77, and 0.74 - 4.14 % of MOR, MOE, and CSPG, respectively. Therefore, our results met the NFPA requirements for design purposes.

Thermal characteristics of TSB impregnated Oriental beech wood

It is well known that the thermal degradation of woods begins with the degradation of hemicelluloses and then the pyrolysis of cellulose and lignin occurs (Tomak et al. 2012, Gao et al. 2006). Thermal degradation of woods consists of three stages. In first stage, physically adsorbed water evaporates between 25–125°C and some volatile compounds eliminated at temperature lower than 200°C. In second stage of the decomposition, primarily cellulose and hemicellulose degrade and it is the main reason of major mass loss in the temperature range of 200–400°C (Pasangulapati et al. 2012). The hemicellulose is basically composed of xylane and xycoglucane and they converted into gaseous products (CO, CO_2, and CH_4) and acetic acid in temperature range of 200-280°C. Also, in temperature range between 250-300°C, lignin and cellulose are converted into gas, tar and char (Pétrissans et al. 2014). This step is also known as wood roasting. The final stages at higher temperature than 400°C, lignin continues to degrade, aromatization begins and more oxygen atoms are eliminated from the system (Brebu et. al. 2013). Thermal analysis (DTA) and thermogravimetry (TG) and first derivative of thermogravimetry (DTG) curves are shown in Fig. 1.

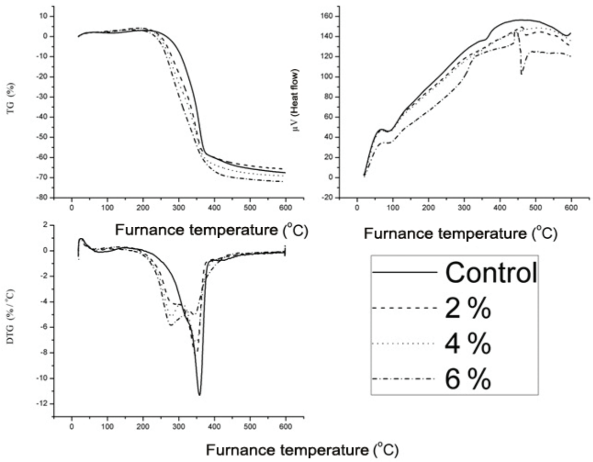

Fig. 1: Thermal analysis (DTA) and thermogravimetry (TG) and first derivative of thermogravimetry (DTG) curves of untreated (control) and TSB impegnated Oriental beech wood specimens.

Pyrolysis experiments were carried out under argon atmosphere with a purge rate of 50 mL.min^{-1} using a LABSYS TG-DTA analyzer (France). The temperature was gradually risen from room temperature to 600˚C with a heating rate of 10°C/min. The TG curves obtained for untreated and 2, 4, and 6 % TSB treated Oriental beech showed similar patterns. For temperature under 200°C, there was no significant changes observed in TG and DTG curves. In this stage which was also known as drying phase, an endothermic peak appeared on the DTA curves corresponding to the evaporation of physically adsorbed water (Yunchu et al. 2000). From the results obtained, it could be said that there were no chemical changes on the main components of wood (Qingwen et al. 2006) and also there was no evidence achieved for desorption of the essential part of TSB in this stage. The residual char (%) after pyrolysis, and maximum and initial temperature (°C) of the pyrolysis were listed in Tab. 4. As illustrated in Fig. 1, main mass loss due to the degradation of cellulose and hemicellulose began at around (Ti) 215 and 240°C, for TSB treated and untreated Oriental beech, respectively. The result obtained from the DTG curves showed that the maximum rate of degradation (T_{max}) was at 359°C for the untreated sample. For the TSB treated samples, two sharp peaks appeared on the DTG curves between 200 and 360°C. The intensity of the peak and also degradation rate of the sample at around 350°C was inversely proportional to increasing percentages of TSB impregnated in Oriental beech. However, the intensity of the other peak appeared at around 275°C proportional to the increasing percentages of TSB. These results showed that the degradation rate of TSB impregnated samples were higher at low temperatures.

Tab. 4: Residual char, initial and maximum temperature of the pyrolysis.

Impregnation material	Conc.%	Residual char (%)	Ti (°C)	Tmax (°C)
Control	-	30.76	240	359
TSB	2	33.03	215	349
	4	30.32	213	347
	6	26.38	211	278

Residual char was calculated as 26.38 to 33.03 % for TSB impregnated Oriental beech after the thermal test (Tab. 4). Only the residual char content of 2 % TSB impregnated samples was higher than that of untreated Oriental beech. Tomak et al. (2012) studied thermal characteristics of boron treated Scots pine wood. They found that residual char was recorded as 49.25 and 55.26 % for 4 borax and 4 % disodium octaborate tetrahydrate treated wood, respectively. These results are considerably higher than our results. Because of this, it is commonly accepted that the fire retardant mechanism of boron compounds is a physical mechanism achieved by the formation of a coating or protective layer on the wood surface at high temperatures (Wang et al. 2004). An increase in the amount of char could provide more surface insulation and can also inhibit volatilization of wood. This can therefore, indicate effective flame retardancy to wood (Baysal 2002, Wang et al. 2004).

CONCLUSIONS

Decay resistance, mechanical, and thermal properties of TSB impregnated Oriental beech were investigated. Our results showed that TSB treatment enhanced decay resistance of Oriental beech wood to significant level. Moreover, higher concentration levels of TSB gave lower mass loss of Oriental beech after the decay test. The mechanical properties of Oriental beech impregnated with TSB decreased from 0.74 to 12.77 %. The National Design Specification for Wood Construction requires a 10-20 % reduction in allowable design stress, depending on the mechanical property under consideration. Thus, our results met the NFPA requirements for design purposes. In our study, higher concentration levels of TSB resulted in lower MOR, MOE, and CSPG of Oriental beech wood. Thermal test results indicates that the degradation rate of TSB impregnated wood specimens were higher at low temperatures with respect to the untreated wood specimens.

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