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SOME PROPERTIES OF TURKISH SWEETGUM BALSAM (*STYRAX LIQUIDUS*) IMPREGNATED ORIENTAL BEECH WOOD
PART I: PHYSICAL PROPERTIES

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ABSTRACT

The purpose of this study was to investigate some physical properties such as water absorption (WA), color, gloss, and surface hardness changes of Oriental beech (*Fagus orientalis* L.) wood impregnated with Turkish sweetgum balsam (TSB). Wood specimens were impregnated with 2, 4, and 6 % (w/V) ethanole solutions of TSB according to ASTM D 1413-76 1976 standard before tests.

Our results showed that WA values of TSB impregnated Oriental beech were higher than that of the untreated control specimen. Moreover, generally higher concentration levels of TSB decreased WA levels of Oriental beech. Untreated and treated Oriental beech had negative Δa^* and Δb^*, and (ΔL^*) values after accelerated weathering. Total color changes (ΔE^*) of TSB treated Oriental beech was lower than that of untreated Oriental beech wood. TSB impregnation decreased gloss and surfaces hardness loss to some extent after accelerated weathering.

KEYWORDS: Turkish sweetgum balsam, Oriental beech, impregnation, physical properties, accelerated weathering.

INTRODUCTION

Wood products are used extensively in both indoor and outdoor applications because of their appealing properties such as aesthetic appearance, low density, low thermal expansion and good mechanical strength (Hsu et al. 2007, Mohan et al. 2008). However, being a natural organic material, wood is susceptible to environmental factors just like other biological materials (Williams and Feist 1999, Chang and Chou 2000). These problems can be partially overcome by modification or impregnation of the wood (Tomak et al. 2011). A major problem of the wood preservatives such as creosote, pentachlorophenol and inorganic arsenicals is that they pose a serious threat to the environment. Because of this, environmentally benign organic preservatives for wood are urgently needed (Hsu et al. 2007).

Liquidambar orientalis Miller (Turkish sweetgum) belongs to the *Hamamelidaceae* family and is an endemic tree species in Turkey. It locally exists in the southwestern coastal area of Turkey (Sagdic et al. 2005). Turkish sweetgum balsam (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 available free and combined with cinnamic acid, which constitutes 30-45 % of the total weight (Hafizoglu 1982). It is not only used in the cosmetics and pharmaceutical industries (Ozturk et al. 2008) but also as a topical parasiticide, expectorant and for the treatment of some skin diseases (Fernandez et al. 2005). Detailed chemical composition of TSB was investigated in several reports and the main constituents was determined as 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); 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. The possibility of using the TSB at wood preservation industry must be investigated. Moreover, some researches were recorded about the protective effect of TSB against decay fungi (Kartal et al. 2012, Degirmentepe 2014). In most of the studies published, some natural compounds extracted from various woods have proven to have antifungal properties. Some examples of this compounds are Eucalyptus oil (Batish 2008), tall oil (Temiz et al. 2008), octyl gallate (Hsu et al. 2007), guayule (Nakayama 2001), heartwood extracts of *Milicia excelsa* and *Erythrophleum suaveolens* (Onuorah 2000) and carvacrol (Kai 1991). Although TSB's protection effect against decay fungi was known, its other effects on wood properties were unknown. In literature, there is no report on the physical properties of TSB impregnated wood. Because TSB is potential candidates for effective and environmentally benign organic preservatives, it is reasonable to examine their effectiveness in preserving wood. From earlier investigations, it is known that the surface properties of wood materials can be enhanced easily by impregnating with various preservatives to provide different performance characteristics for individual applications, such as high hardness, impact resistance, suitable gloss and color (Chang and Lu 2012). Wood color and gloss are important for wood applications in terms of aesthetic considerations, and sometimes may determine its value in the market (Tolvaj et al. 2011, Huang et al. 2012). Therefore, it is important to investigate TSB impregnated wood and its physical characteristics. The present paper was aimed to investigate to evaluate its physical characteristics such as water absorption (WA), color, gloss, and surface hardness changes of TSB impregnated Oriental beech wood.

MATERIAL AND METHODS

Preparation of test specimens and chemicals

TSB was obtained from local supplier in Mugla, an area located in the South West region of Turkey and it was used as it collected from the sources. Air-dried sapwood specimens of Oriental beech (*Fagus orientalis* Lipsky) were prepared for impregnation treatment with dimensions of, 20 (radial) x 20 (tangential) x 20 (longitudinal) mm for water absorption (WA) test, 6 (radial) x 75 (tangential) x 150 (longitudinal) mm for color, gloss, and surface hardness tests. Appropriate amount of TSB was dissolved in ethanole to obtained the solutions with the concentration of 2, 4, and 6 % (% w/V). Water absorption wood specimens were oven dried at 103 ± 2°C before and after treatments. Color, gloss, and surface hardness wood specimens were oven dried at 55 ± 2°C until constant weight before treatment. After treatment, they were conditioned for two weeks at 65 % relative humidty and 20 ± 1°C before tests.

Impregnation method

Wood specimens were impregnated with 2, 4, and 6 % (w/V) ethanole solutions of TSB according to ASTM D 1413 (1976). A vacuum desiccator used for the impregnation process was connected to a vacuum pump through a vacuum trap. Vacuum was applied for 30 min at 760 m Hg^{-1} before supplying the solution into the chamber followed by another 30 min at 760 mm Hg^{-1} diffusion period under vacuum. Retention was calculated from the following equation:

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

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 (cm^3).

Water absorption

Wood specimens measuring 20 (tangential) x 20 (radial) x 20 (longitudinal) mm were prepared from sound sapwood of Oriental beech (*Fagus orientalis* L.). In the applied method, specimens were submerged in distilled water for 2, 4, 8, 24, and 48 hours. Water absorption (WA) levels of the wood specimens were calculated as an index of water repellent efficiency from the initial and final wet weights after water saturation in the following equation:

$$WA = \frac{W_{(wf)} - W_{(oi)}}{W_{(oi)}} \times 100 \qquad (\%) \qquad (2)$$

where: W_{wf} - the wet weight of a wood specimen after saturation with water,
W_{oi} - the initial dry weight.

Color measurement

Specimens measuring 6 x 75 x 150 mm (radial by tangential by longitudinal) were machined from the air-dried sapwood of Oriental-beech (*Fagus orientalis* L.) lumber. All specimens were conditioned at 20°C and 65 % relative humidity for two weeks before test.

The color parameters a^*, b^*, and L^* were determined by the CIELAB method. The L^* axis represents the lightness, whereas a^* and b^* are the chromaticity coordinates. The + a^* and -a^*

parameters represent red and green, respectively. The + b^* parameter represents yellow, whereas -b^* represents blue. L^* can vary from 100 (white) to zero (black) (Zhang 2003). The colors of the specimens were measured by a colorimeter (X-Rite SP Series Spectrophotometer) before and after the accelerated weathering. The measuring spot was adjusted to be equal or not more than one-third of the distance from the center of this area to the receptor field stops. The total color difference, (ΔE^*) was determined for each treatment group as follows (ASTM D 1536–58 1964):

$$\Delta a^* = a_f^* - a_i^* \quad (3)$$
$$\Delta b^* = b_f^* - b_i^* \quad (4)$$
$$\Delta L^* = L_f^* - L_i^* \quad (5)$$
$$(\Delta E^*) = [(\Delta a^*)2 + (\Delta b^*)2 + (\Delta L^*)\ 2]\ ½ \quad (6)$$

where: Δa^*, Δb^*, and ΔL^* - the changes between the initial and final interval values.

Gloss test

The gloss of wood specimens was determined using a glossmeter (BYK Gardner, MicroTRI-Gloss) according to ASTM D523-08 (2008). The chosen geometry was an incidence angle of 60º. Results were based on a specular gloss value of 100, which relates to the perfect condition under identical illuminating and viewing conditions of a highly polished, plane, black glass surface.

Surface hardness test

The surface hardness of test specimens was measured as the König hardness according to ASTM D 4366–95 (1995). Wood specimens were placed on the panel table, and a pendulum was placed on the panel surface. Then, the pendulum was deflected through 6° and released, at the same time, a stopwatch was started. The time for the amplitude to decrease from 6° to 3° was measured as König hardness.

Accelerated weathering

The accelerated weathering experiment was performed in a QUV weathering tester with eight UVA 340 lamps. The weathering schedule involves a continuous light irradiation of 8 h following with a condensation for 4 h. The average irradiance was 0.89 $W.m^{-2}$ at 340 nm wavelengths. The temperature of the light irradiation period and the condensation period was 60 and 50°C, respectively. Wood specimens were mounted on aluminum panels before placing in the QUV. The changes on wood specimens were monitored for a total 500 h.

Evaluations of test results

Water absorption levels of TSB impregnated Oriental beech 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 DISCUSSION

Water absorption levels

Tab.1 shows WA of the Oriental beech specimens impregnated with TSB during the immersion test. TSB retention was calculated as 7.44 to 25.30 $kg.m^{-3}$.

Tab. 1: Water absorption of TSB impregnated Oriental beech.

Impregnation material	Conc. (w/V) %	Retention (kg.m^{-3})	WA* (%)														
			2 hours			4 hours			8 hours			24 hours			48 hours		
			Mean	SD	HG	Mean	SD	HG	Mean	SD	HG	Mean	SD	HG	Mean	SD	HG
Control	-	-	32.25	7.09	A	41.35	11.09	A	46.66	8.88	A	58.16	12.97	A	62.21	11.00	A
TSB	2	7.44	26.55	7.34	A	36.79	10.62	A	44.83	11.24	A	54.39	9.88	A	61.82	9.90	A
	4	17.69	26.91	8.85	A	33.54	11.78	A	40.09	11.53	A	52.85	9.72	A	59.00	7.94	A
	6	25.30	24.82	8.03	A	33.23	13.38	A	38.28	12.76	A	48.47	10.44	A	54.83	8.63	A

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

Results indicated that WA levels were much higher during the first period of WA consistent with the previous data (Hafizoglu et al. 1994, Yildiz 1994, Alma 1991). For example, while WA of the control specimen was 32.25 % in first period (2 h), cumulative WA of control specimen was 62.21 % in last period (48 h). Therefore, more than half of the WA occurred in the first period. These results may be due to WA into available empty pores in wood at the beginning of soaking and the reduction of those wood spaces over time (Yalinkilic et al. 1995). TSB impregnation seems to have positive effect on WA. It is expected because TSB fills the lumen. Therefore, TSB, as a water repellent chemical, is applied to wood for filling the cell lumina or is deposited on the external and to some extent on the internal pore surfaces and impart the hydrophobic properties to the surface (Hyvönen et al. 2005). Hence, the water cannot spontaneously penetrate the wood pores through capillary action and the WA rate is thus limited (Banks and Voulgaridis 1980). However, it was not found significantly affective WA values at all immersing periods. Also, there is not statistical differences in WA levels among all of TSB treated Oriental beech wood at all immersing periods. According to our results, generally, WA levels decreased with increasing TSB concentration. Var and Oktem (1999) investigated that WA of 3 h dipped natural resin treated Oriental beech wood. They found that WA of Oriental beech wood was 28.21 and 49.21 %, for 4 and 24 h, respectively. The WA values obtained in this study were slightly different from those reported by Var and Oktem (1999). These differences may be due to the treatment time, treatment type and usage of different types of chemicals.

Color changes

Tab. 2 shows the overall changes in color (ΔE^*) due to the accelerated weathering of the TSB treated and untreated Oriental beech. In addition, changes in the individual ΔL^*, Δa^*, and Δb^* were also examined.

Tab. 2: Color changes of TSB impregnated Oriental beech after accelerated weathering.

Impregnation material	Conc. % (w/V)	Ret. (kg.m^{-3})	Before accelerated weathering			After 500 hours accelerated weathering			After 500 hours accelerated weathering			
			L*	a*	b*	L*	a*	b*	ΔL*	Δa*	Δb*	ΔE*
Control	-	-	63.10	11.25	19.10	50.51	15.59	28.11	-12.58	-4.35	-9.01	16.07
TSB	2	8.44	62.90	10.83	19.66	50.97	15.71	29.04	-11.93	-4.87	-9.38	15.94
	4	19.47	65.51	10.45	18.73	55.46	14.29	27.08	-10.05	-3.84	-8.35	13.62
	6	25.92	61.16	11.04	19.28	52.67	14.36	27.36	-8.50	-3.31	-8.09	12.19

Note: Ten replicates were made for each treatment group. Ret: Retention; Conc: Concentration.

Retention values were calculated as 8.44 $kg.m^{-3}$, 19.47, and 25.92 $kg.m^{-3}$ for 2, 4, and 6 % (w/V) of TSB impregnated Oriental beech, respectively. The negative lightness stability (ΔL^*) values occurred after the accelerated weathering. All specimens become darker as a result of weathering (Lesar et al. 2011). Depolymerization of the lignin on the exposed surface may also render the surface darker (Temiz et al. 2005). The control specimens became the darkest among the all specimens. The results showed that all the treated Oriental beech caused less changes in the lightness than untreated Oriental beech. Moreover, higher concentration levels of TSB resulted in lower ΔL^* values of Oriental beech after 500 h accelerated weathering period. Negative values of Δa^* indicate a tendency of wood surface to become greenish. Negative values of Δb^* indicate a tendency of wood surface to become blueish. The results demonstrated that Δa^* and Δb^* of untreated and treated Oriental beech had negative values after accelerated weathering. Our results showed that higher concentration levels of TSB resulted in lower Δa^* and Δb^* values of Oriental beech after 500 h accelerated weathering period. However, Δa^* and Δb^* values of untreated Oriental beech were higher than 2 % TSB impregnated Oriental beech wood after 500 h accelerated weathering. The nature of wood very rapidly changed when exposed to accelerated weathering. At this study, TSB impregnated wood specimens showed better color stability as compared to untreated wood specimens after accelerated weathering. The higher color changes resulting from accelerated weathering for untreated Oriental beech are due to the higher contribution from the chromaticity coordinates Δa^* and Δb^*, and ΔL^* (Deka et al. 2008). The greatest total color changes (ΔE^*) occured with untreated Oriental beech after an accelerated weathering period. ΔE^* of TSB impregnated Oriental beech decreased to some extent. The higher concentration levels of TSB resulted in lower ΔE^* of Oriental beech. Hansmann et al. (2006) investigated color charecterictics of spruce and poplar wood samples impregnated with melamine formaldehyde resins after artificial weathering. They found that the treated samples showed advantages in terms of discolouration compared to untreated wood samples. Another study carried out by Lesar et al. (2011) showed that total color changes of wax treated wood was lower than untreated wood's total color changes. Our results are in good agreement with these researchers' findings.

Gloss

Data for the specular gloss of the wood surfaces at a 60° incidence angle measured before and after exposure to accelerated weathering are given in Tab. 3.

Tab. 3: Gloss values of TSB impregnated Oriental beech after accelerated weathering.

Impregnation material	Conc. % (w/V)	Gloss values*				
		Before accelerated weathering		After 500 hours accelerated weathering		
		Mean	SD	Mean	SD	%
Control	-	3.11	0.48	2.44	0.37	-21.54
TSB	2	3.11	0.29	2.54	0.26	-18.32
	4	2.66	0.30	2.34	0.31	-12.03
	6	2.57	0.28	2.41	0.30	-6.22

* Ten replicates were made for each treatment group. Conc: Concentration, SD: Standard deviation.

Glossiness, the property of reflecting light in a mirror is very important for the aesthetic and decorative appearance of surfaces (Cakicier et al. 2011). Our results showed that while the highest gloss value was 3.11 for untreated Oriental beech and 2 % TSB impregnated Oriental beech, the lowest gloss value was 2.57 for the 6 % TSB impregnated Oriental beech before accelerated weathering. Accelerated weathering caused gloss loss of treated and untreated Oriental beech wood specimens. Abrasion on the wood surfaces, along with erosion, causes gloss degradation (Yalinkilic et al. 1999). Especially, untreated Oriental beech and 2 % TSB impregnated Oriental beech showed drastic gloss loss after 500 h accelerated weathering. Gloss loss values were approximately 21.54 % for the control, while it was 18.32, 12.03, and 6.22 % for 2, 4, and 6 %, respectively. As a result, TSB impregnation decreased the gloss loss of Oriental beech after accelerated weathering. Moreover, higher concentration levels of TSB resulted in lower gloss loss of Oriental beech after accelerated weathering.

Surface hardness

Surface hardness is given in Tab. 4. Accelerated weathering conditions softened both treated and untreated Oriental beech wood.

Tab. 4: Surface hardness values of TSB impregnated Oriental beech after accelerated weathering.

Impregnation material	Conc. % (w/V)	Surface hardness values*				
		Before accelerated weathering		After 500 hours accelerated weathering		
		Mean	SD	Mean	SD	%
Control	-	35.40	2.70	21.50	3.11	-39.27
TSB	2	36.40	3.21	26.00	8.93	-28.57
	4	35.50	1.73	26.00	1.63	-26.76
	6	29.17	4.31	21.75	7.14	-25.44

*Ten replicates were made for each treatment group. Conc: Concentration, SD: Standard deviation.

The combined effect of moisture, UV light, and temperature could destroy the lignocellulosic network of the wood. Therefore, the degradation products become water- soluble and are leached out resulting in erosion of the wood surface (Meijer 2001). Our results showed that surface hardness values of untreated and treated Oriental beech wood samples were decreased after accelerated weathering. However, TSB impregnation had a contributory effect on the hardness of Oriental beech specimens after 500 h accelerated weathering. For example, while the surface hardness was decreased approximately by 28.57, 26.76, and 25.43 % for 2, 4, and 6 % (w/V) of TSB impregnated Oriental beech, respectively, it was decreased by 39.27 % for untreated Oriental beech after 500 h accelerated weathering. Therefore, Oriental beech wood impregnation with TSB could lead to the increases in surface hardness after accelerated weathering to some extent. Our results showed that higher concentration levels of TSB resulted in higher surface hardness of Oriental beech after accelerated weathering. Feist et al. (1991) reported that filling wood cell lumens with methyl methacrylate reduced the erosion of by 40 % or more during accelerated weathering. Hansmann et al. (2006) investigated the artificial weathering of wood surfaces modified by melamine formaldehyde resins. They reported that the applied melamine treatment led to significant increases of surface hardness. Our results are in good agreement with aforementioned studies.

CONCLUSIONS

Some physical properties such as WA, color, gloss, and surface hardness changes of TSB impregnated Oriental beech were investigated. Our results showed that TSB treatment improved WA properties of Oriental beech wood. The ΔL^* in TSB impregnated wood after accelerated weathering were found to be lower than that of untreated Oriental beech. The results showed that Δa^* , Δb^*, and ΔL^* of untreated and treated Oriental beech had negative values after accelerated weathering. TSB impregnation enhanced gloss and surface hardness of Oriental beech compared to that of untreated Oriental beech after accelerated weathering. According to our results, generally, higher concentration levels of TSB resulted in better physical properties of Oriental beech wood.

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