

**SURFACE CHARACTERISTICS OF SCOTS PINE TREATED
WITH CHEMICALS CONTAINING SOME COPPER
COMPOUNDS AFTER WEATHERING**

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(RECEIVED MARCH 2015)

ABSTRACT

In this study, it was aimed to investigate surface hardness, gloss, and color changes of Scots pine treated with chemicals containing some copper compounds after six months weathering. Adolit KD-5 (AD KD-5), celcure AC-500 (CAC-500), and wolmanit CX-8 (WCX-8) were used as impregnation chemicals containing copper compounds. Scots pine wood specimens were treated with 2 % aqueous solution of chemicals according to ASTM D1413-07e1 (2007) standard.

Results showed that while surface hardness and gloss values of untreated Scots pine wood specimens were decreased after weathering, they increased treated Scots pine wood specimens after weathering. The decrease in L* of untreated and treated wood indicates that the specimens became darker after weathering. While weathering caused less green and less yellow for untreated control specimen, it caused less red and less yellow for treated wood. Treated Scots pine wood specimens showed better color stability compared to untreated Scots pine after weathering. In terms of surface hardness, gloss, and color stability values CAC-500 treated Scots pine gave the best results after weathering.

KEYWORDS: Weathering, surface hardness, gloss, color, Scots pine, impregnation.

INTRODUCTION

Wood has been popularly and favorably used as a decorative material owing to its aesthetic appearance and characteristic properties (Chang and Chang 2001). However, wood surfaces exposed outdoors are rapidly degraded because lignin strongly absorbs UV light, which leads to radical-induced depolymerization of lignin and cellulose, the major structural constituents of wood (Evans et al. 2002). The main factor that causes the greatest changes in the surface properties of wood during outdoor exposure is sunlight (Tolvaj et al. 2011). Because lignin is the main chemical component of wood, the aromatic polymer, strongly absorbs sunlight (Jebrane et al. 2009), which causes depolymerization of lignin. The yellowing, browning, and/or graying of wood surfaces indicate the modification of lignin when wood is exposed outdoors (Grelier et al. 2000). The surface properties of wood materials can be enhanced easily by impregnating and finishing with various preservatives to provide different performance characteristics for individual applications, such as high hardness, impact resistance, suitable gloss, and chemical resistance (Chang and Lu 2012; Degirmentepe et al. 2015a, b). Treatment with wood preservatives especially formulated with chromium and/or copper compounds improve the durability of wood surfaces against UV irradiation and weathering factors (Temiz et al. 2005). The most effective method of preventing the photodegradation of wood involves treatment with dilute aqueous solutions of inorganic salts, particularly hexavalent chromium compounds. Application of chromium trioxide to wood surfaces prevents lignin degradation during natural weathering (Evans et al. 1992, Kiguchi and Evans 1998). Chromated copper arsenate (CCA) has provided long-term protection against weathering and erosion (Feist and Ross 1995; Jirouš-Rajković et al. 2004) but it is no longer being produced for use in most residential settings, because it contains chromium and arsenic. Nowadays, several new copper-based wood preservatives such as tanalith-E (TN-E) and adolit-KD5 (AD-KD5) are being used in the forest products industry instead of CCA (Turkoglu et al. 2015a, b). Pizzi (1980) reported that the beneficial effects of chromium were attributed to the formation of complexes between chromium and guaiacyl units of lignin. Sell and Feist (1985) studied weathering durability of obeche, red beech, spruce, and fir woods impregnated with CCB. They found that CCB-impregnated wood specimens have high resistance and protective effect against weathering. Sell et al. (1974) reported that high resistance of the CCB-impregnated wood against weathering has been attributed to the protective effect of Cr-Cu salt solutions of the wood surface. In another study, Williams et al. (1996) found that the chromium oxides in copper-chromate-arsenic (CCA) which bond to the wood after treatment, decrease photodegradation of the wood surface. The focus on copper-based preservatives has increased following concerns about environmental effects of chromium and arsenic (Freeman

and McIntyre 2008). Therefore, weathering aspects of treated wood with new wood preservatives become of a practical importance (Temiz et al. 2007). Copper forms certain complexes with wood components, such as copper-cellulose complexes, copper-lignin complexes, and crystalline or amorphous inorganic/organic copper compounds, and reduces the degradation of the wood surface from weathering factors (Temiz et al. 2005, Grelier et al. 2000). Weathering of wood impregnated with alkylammonium compounds (AACs), ammoniacal copper quat (ACQ 1900 and ACQ 2200), tanalith-E 3491 and wolmanit CX-8 was investigated by Jin et al. (1991) and Temiz et al. (2005). Cornfield et al. (1994) and Zhang and Kamdem (2000a) determined that wood treatment with azole and copper ethanolamine decreases photodegradation of wood and increases the hydrophobicity. Zhang et al. (2009) studied accelerated weathering performance of copper-amine treated Sothern pine wood. They found that copper amine treatment can retard photodegradation of wood. Another study, Zhang and Kamdem (2000b) determined that wood impregnated with copper monoethanolamine showed much less lignin degradation compared to untreated wood.

In this study, surface hardness, gloss, color changes of Scots pine treated with chemicals containing some copper compounds such as adolit KD- 5 (AD KD-5), celcure AC-500 (CAC-500), and wolmanit CX-8 (WCX-8) after 6 months weathering were studied. Weathering was performed in Mugla, which is in Southern Aegean Region of Turkey.

MATERIAL AND METHODS

Preparation of test specimens and chemicals

Ten wood specimens were used for each treatment group. Wood specimens measuring 10 x 100 x 150 mm were prepared from air-dried sapwood of Scots pine (*Pinus sylvestris* L.). According to technical data sheets of products, AD KD-5 comprises 20.53 % copper (II) hydroxide carbonate, 10 % didecylpolyoxethylammoniumborate, and 8 % boric acid (Özgenc et al. 2012; Baysal et al. 2014). CAC-500 consists of 10-30 % copper carbonate, 30-50 % 2-aminoethanol, and 5 % > benzylammonium chloride (Ozgenc and Yildiz 2014). The constitutes of WCX-8 are 2.8 % bis-(n-cyclohexyldiaziniumdioxy)-copper, 13.0 % copper (II) carbonate hydroxide, and 4 % boric acid (Technical Leaflet 2007). Aqueous solutions of AD KD-5, CAC-500, and WCX-8 were dissolved in distilled water to concentration 2 %.

Impregnation method

Wood specimens were treated with 2 % aqueous solution of chemicals according to ASTM D1413-07e1 (2007). Retentions of chemicals were calculated from the following equation:

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

where: G - amount of solution absorbed by wood that is calculated by $T_2 - T_1$,

T_2 - masses of wood after impregnation (g),

T_1 - masses of wood before impregnation (g),

C - solution concentration as percentage,

V - volume of the specimen as cm^3 .

Surface hardness

The surface hardness of wood specimens was measured as the König hardness according to ASTM D4366-14 2013. Surface hardness measurements were made in the direction parallel to

the fiber. Wood specimens were placed on a 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.

Gloss test

The gloss test of wood specimens was determined using a gloss meter (BYK Gardner, Micro-TRI-Gloss) according to ASTM D523-14 2013. The chosen geometry was an incidence angle of 60°. 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. Gloss measurements were made in the direction parallel to the fiber.

Color test

The color parameters L^* , a^* and b^* were determined by the CIEL*a*b* 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 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 color difference, (ΔE^*) was determined for each wood as follows ASTM D1536-58 T (1964):

$$\Delta a^* = a^*f - a^*I \quad (2)$$

$$\Delta b^* = b^*f - b^*I \quad (3)$$

$$\Delta L^* = L^*f - L^*I \quad (4)$$

$$(\Delta E^*) = \sqrt{[(\Delta a^*)^2 + (\Delta b^*)^2 + (\Delta L^*)^2]/2} \quad (5)$$

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

Color measurements were made in the direction parallel to the fiber.

Weathering exposure

Wood specimens were prepared for weathering exposure according to ASTM D7787 (ASTM D7787/D7787M-13 2013). They were exposed to weathering conditions during from February to July in 2015. The site is situated at Mugla Sitki Kocman University (37° 09' N and 28° 22' E, 670 m above sea level) in Mugla, Southern Aegean Region of Turkey. Weather conditions of Mugla during weathering were given Tab. 1 (Meteorological data 2015).

Tab. 1: Weather conditions of Mugla from February to July in 2015.

Months	Feb	Mar	Apr	May	Jun	Jul
Average temperature (°C)	5.9	8.8	11.5	18.4	20.7	27.1
Sunbathing time (hour)	2.0	3.5	7.5	7.5	8.2	9.6
The number of the rainy day	14	14	6	6	8	4
Rainfall per month (kg·m ⁻²)	238.6	197.1	25.2	89.1	59.0	24.2
Humidity (%)	77	77	60	61	62	44

The exposure rack was positioned so that the exposed specimens were at an angle of 45° facing south. Wood specimens were set outside for weathering exposure according to ASTM G7/G7M-13 (ASTM G7/G7M-13 2013). Exposure covered a period of 6 months. Surface hardness,

gloss, and color measurements were made on the exposed surfaces of the wood specimens before and after weathering.

RESULTS AND DISCUSSION

Surface hardness changes

Surface hardness values of Scots pine wood treated with chemical preservatives containing copper compounds before and after weathering are given in Tab. 2 along with the retention values of Scots pine wood specimens.

Tab. 2: Surface hardness of specimens before and after natural weathering.

Treatment	(%)	Retention (kg·m ⁻³)	Before natural weathering		After 6 months natural weathering		
			Mean	SD	Mean	SD	Change (%)
Control	-		17.00	4.76	12.56	3.78	-26.12
AD KD-5	2	10.61	11.38	2.33	13.00	5.61	14.24
CAC-500	2	11.47	8.87	1.89	13.25	3.24	49.38
WCX-8	2	9.59	11.13	2.70	11.75	1.58	5.57

Note: Results reflect observations of ten specimens

SD: Standard deviation.

Retention values were found to be as 11.47, 9.59, and 10.61 kg·m⁻³ for CAC-500, WCX-8, and AD KD-5, respectively for treated Scots pine wood specimens. The surface hardness value measured before weathering test was 17.00 for untreated control specimen. The lowest surface hardness value was calculated for the Scots pine wood before weathering with 8.87 for the wood specimens treated with CAC-500. Our results showed that impregnation resulted in a decrease in the surface hardness of the Scots pine wood before weathering. However, weathering conditions softened untreated Scots pine wood in some extent. 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). However, increases in surface hardness were recorded for treated Scots pine wood surfaces after weathering, especially CAC-500 treated Scots pine wood. According to our results, impregnation had a contributory effect on the hardness of the wood after weathering. Because, while surface hardness of untreated Scots pine wood decreased 26.12 % after weathering, surface hardness of treated Scots pine increased from 5.57 to 49.38 % after weathering exposure. 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. Yalinkilic et al. (1999), Baysal (2008), and Turkoglu et al. (2015a) studied surface hardness values of some weathered wood species. They found that weathering softened wood surfaces and caused decrease in surface hardness values of wood specimens. Our results are in good agreement with these researchers' findings.

Gloss changes

Glossiness, the property of reflecting light in a mirror is very important for the aesthetic and decorative appearance of surfaces (Cakicier et al. 2011). Gloss of Scots pine wood specimens was measured at a 60° angle of incidence using a gloss meter. Gloss values of Scots pine wood specimens before and after weathering are given in Tab. 3.

Tab. 3: Gloss values of specimens before and after natural weathering.

Treatment	(%)	Before natural weathering		After 6 months natural weathering		
		Mean (60°)	SD	Mean (60°)	SD	Change (%)
Control		5.04	1.44	4.51	0.67	-10.52
AD KD-5	2	1.83	0.14	2.74	0.32	49.73
CAC-500	2	2.03	0.18	3.11	0.41	53.20
WCX-8	2	1.90	0.16	2.89	0.30	52.11

Note: Results reflect observations of ten specimens

SD: Standard deviation.

Before weathering, the gloss value of untreated (control) wood specimen was much higher than wood specimen treated with chemicals containing copper compounds. While the highest gloss value was 5.04 for untreated Scots pine, the gloss values of all other treated Scots pine were found to be between 1.83 and 2.03 before weathering. Simsek and Baysal (2012) investigated gloss values of borate treated some wood species. They found that borate treatments remarkably decreased gloss values of wood species. It can be explained that impregnation process with the solutions might limit the glossiness to a point in Scots pine before weathering, probably owing to the absorption and dispersion of the reflected rays by salt crystals prominent in the large lumens of the vessel in the wide early wood sections of the grains. Photoactive ion on the wood surface was assumed to cause same losses in glossiness before weathering. This result is consistent with the outdoor performance of CCB (chromated copper boron) impregnated Scots pine before varnish coating (Yalinkilic et al. 1999). According to our results, impregnation had a contributory effect on the gloss of the wood after weathering. Our results showed that gloss of untreated Scots pine wood decreased 10.52 % after weathering. Thus, abrasion on the wood surfaces along with the erosion causes gloss loss. However, gloss of treated Scots pine increased from 49.73 to 53.20 % after weathering exposure. Baysal (2012) studied some surface characteristics of CCA treated Scots pine after accelerated weathering exposure. They found that CCA treatment caused increases in gloss of Scots pine after accelerated weathering. Our results are in good agreement with data from Baysal (2012).

Color changes

Tab. 4 presents L^* , a^* , and b^* values of untreated (control), and treated Scots pine specimens before and after weathering, and also shows the values of change for all three color parameters (ΔL^* , Δa^* , and Δb^*), as well as the total color changes (ΔE^*) of the Scots pine wood specimens after 6 months of weathering. Before natural weathering, while L^* value of untreated Scots pine wood specimen was 77.03, L^* values of treated Scots pine wood specimens changed from 58.75 to 60.10.

Tab. 4: Total color changes of specimens before and after natural weathering.

Treatment	(%)	Before natural weathering						After 6 months natural weathering						Total color change			
		L^*		a^*		b^*		L^*		a^*		b^*		ΔL^*	Δa^*	Δb^*	ΔE^*
		Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD				
Control		77.03	1.44	6.93	0.69	29.17	2.35	62.67	1.50	4.71	0.44	13.97	0.69	-14.36	-2.22	-15.20	21.03
AD KD-5	2	58.75	1.08	0.75	0.13	21.27	1.56	55.04	1.40	7.79	0.43	21.07	0.69	-3.71	7.04	-0.20	7.96
CAC-500	2	59.07	1.10	1.33	0.23	22.25	0.93	56.19	1.37	7.74	0.44	22.21	0.75	-2.88	6.41	-0.04	7.03
WCX-8	2	60.10	1.38	1.50	0.51	20.70	1.51	55.58	0.99	7.57	0.53	20.46	1.01	-4.52	6.07	-0.24	7.57

Note: Results reflect observations of ten specimens, SD: Standard deviation.

The decrease in L^* value of wood specimens indicates that the specimens become darker after treatment. Our results showed that impregnation treatment caused a decrease a^* and b^* values of Scots pine wood in some extent before weathering. Baysal (2012) investigated color changes of CCA treated Scots pine before weathering. He found that CCA treatment decreased L^* , a^* , and b^* values of Scots pine compared to untreated Scots pine. Our results are consistent with this researcher finding. The negative lightness stability (ΔL^*) values for untreated and treated Scots pine occurred after weathering. Therefore, the wood surface got rougher and darker after weathering. Previous researches show that weathering causes darkness in wood surface (Turkoglu et al. 2015a, b, c). Depolymerization of the lignin on the exposed surface may also render the surface darker (Temiz et al. 2005). The results showed that treated Scots pine caused less change in the lightness than untreated Scots pine. While ΔL^* of untreated Scots pine was (-14.36), it changed from (-2.88) to (-4.52) for treated Scots pine after weathering. Grelier et al. (2000) found that ΔL^* values of copper-amine treated wood was lower than the values of controls with a possible explanation that copper - amine treatment enhanced the color stabilization of wood and reduced the lignin degradation resulting from UV light irradiation.

The positive values of Δa^* indicate a tendency of wood surface to become redder and negative values represent less red wood surface after weathering. Our results showed that while untreated Scots pine showed a tendency of less red, treated Scots pine showed a tendency of less green after natural weathering. After weathering, Δa^* value was found to be -2.22 for untreated Scots pine. Δa^* values changed from +6.07 to +7.04 for treated Scots pine after weathering. Positive values of Δb^* show a tendency of wood surface to become yellower, and negative values represent less yellow of wood surface after weathering. According to our results, untreated and treated Scots pine showed a tendency of more blue after weathering. But, this tendency was very small for treated Scots pine. For example, after weathering, while Δb^* value was found to be -15.20 for untreated Scots pine, Δb^* values changed from -0.04 to -0.24 for treated Scots pine after weathering. Negative Δb^* values represent less yellow for untreated and treated Scots pine wood surface to become more blue after weathering. Total color changes (ΔE^*) values of all treated Scots pine wood specimens were less than that of untreated Scots pine wood specimens. While ΔE^* of untreated Scots pine was 21.03, it changed from 7.03 to 7.96 for treated Scots pine after weathering. These findings compatible agree with the literature reporting that copper containing treatments provided better protection for color changes than untreated wood specimens (Ozgenc and Yildiz 2014, Grelier et al. 2000, Temiz et al. 2005). Our results showed that the best color stability was obtained with CAC-500 treated Scots pine after weathering. Ozgenc and Yildiz (2014) studied surface characteristics of wood treated with new generation preservatives such as celcure AC500, micronized copperquat(MCQ), and traditional preservatives(didecyldimethylammonium chloride (DDAC), and copper (II) sulfate pentahydrate ($Cu(II)SO_4 \cdot 5H_2O$)) after accelerated weathering. They found that the least color change (ΔE^*) for all species was observed on MCQ and celcure AC 500 treated wood samples. Celcure AC 500 and MCQ treatments for all species slowed down photodegradation by retarding the formation of carbonyl groups. Temiz et al.

(2005) investigated the color characteristics of Scots pine treated with ammonium copper quat (ACQ₂ and ACQ₂₂₀₀), chromate copper arsenate (CCA), tanalith – E 3491 and wolmanit CX-8 after accelerated weathering test. They found that the most effective treatment for stabilizing wood color was treatment with CCA and ACQ₁₉₀₀. The photostabilization of wood by copper treatments may be explained by the retarding the formation of carbonyl groups and reduced delignification after weathering (Temiz et al. 2005).

CONCLUSIONS

Some surface characteristics such as surface hardness, gloss and color changes of Scots pine (*Pinus sylvestris* L.) treated with chemicals containing some copper compounds were studied after natural weathering. AD KD-5, CAC-500, and WCX-8 were used as impregnation chemicals containing copper compounds. Wood specimens were treated with 2 % aqueous solution of chemicals. Natural weathering was performed in Mugla, which is in Southern Aegean Region of Turkey. Scots pine wood specimens were exposed to weathering conditions during from February to July in 2015.

Surface hardness and gloss values of Scots pine wood specimens decreased in some extent during the impregnation. However, the weathering caused significant increases of surface hardness and gloss of impregnated Scots pine wood specimens. In terms of surface hardness, gloss, and color stability values CAC-500 treated Scots pine gave the best results after weathering. Weathered all Scots pine wood specimens became darker tonality, especially untreated Scots pine. After weathering, untreated Scots pine wood tended to become less green and less yellow treated Scots pine wood tended to become less red and less yellow. Total colors changes of untreated Scots pine wood were higher than treated Scots pine after weathering.

In conclusion, chemical impregnation containing copper compounds improved weathering durability of Scots pine wood. In impregnation chemicals, CAC-500 treated Scots pine gave the best results in terms of surface characteristics.

ACKNOWLEDGMENT

This study was made use of M.Sc. Thesis by Suleyman Ustun in the Graduate School of Natural and Applied Sciences, Mugla Sitki Kocman University. The design and development of study was proposed and supervised by Ergun Baysal. Turkyay Turkoglu, Hilmi Toker, Cevdet Sacli and Huseyin Peker contributed to the writing, revising and proofing of the paper. Extensive advice and critique of the research was provided by Ergun Baysal.

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