Colorability of Wood Material with *Punica granatum* and *Morus nigra* Extracts

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The aim of this study was the development of an eco-friendly dye that may be used in furniture, woodworking, and upper surface treatment, with no ill effects to human health. The plant dyestuff was extracted from pomegranate skin (*Punica granatum*) and black mulberry (*Morus nigra*) using an ultrasonic method at different rates. The extraction percentage ratios were, by weight in distilled water, 25%, 30%, 35%, 40%, 45%, and 50% and were applied to Scotch pine (*Pinus sylvestris* L.) and Oriental beech (*Fagus orientalis* L.) through an immersion method. After that, the determination of color change under the accelerated weathering conditions and the feasibility as the upper surface stain of this dyestuff were studied. The values of total color change of the natural dye samples that were applied to the test material were determined after accelerated weathering processes lasting 50, 100, and 150 h, according to ISO 2470. The results showed the best color stability in Oriental beech wood that was treated with 30% pomegranate skin or black mulberry extract as well as Scotch pine wood that was treated with 25% pomegranate skin extract or 50% black mulberry extract. Exactly the same ΔE^* values were obtained with the same dyestuff and same wood type, indicating close color stability behaviors. According to the results, both pomegranate skin (*Punica granatum*) and black mulberry (*Morus nigra*) can be used to color wood materials.

Keywords: Morus nigra; Punica granatum; Natural dye; UV weathering

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INTRODUCTION

Wooden construction materials are indispensable for both interior and exterior decoration material. Due to the structural characteristics of wood material, it is still preferred in various production areas (Peker 1997). Decreasing forest area has made mandatory the efficient use of wood material. Wood material is often valued for its aesthetic qualities. While it can be used to make beautiful images, they must be protected from both indoor and outdoor environmental factors. Wood, especially in outdoor environments, may be subject to considerable damage. Seasonal changes, sun damage, and rain may cause images to look worn and old (Peker 1997). Paint, varnish, and wood preservatives are often used to prevent these negative effects on wood material. However, during the preservation of wood by chemical means and in terms of environmental health, drawbacks have emerged in recent years (Kurtoglu 1988). Typically, protection for wood must have toxic effects against pests. Exposure to contamination indoors causes adverse effects on human health, and so this has become a subject of careful scrutiny for society, particularly customers of these products, administrative sciences, industrial employees, and researchers (Salthammer *et al.* 2002).

For years, it has been known that many synthetic dyes are dangerous to human health, and the industry has been searching for environmentally friendly alternatives. The majority of textile dyestuffs are derivatives of carcinogenic, aromatic compounds. It is known that benzene and some aromatic hydrocarbons derived from benzene are carcinogenic. If used too much, these compounds pose a threat to human health and the environment (Kizil 2000). As human and environmental health became a concern, this industry began to develop new protective standards; thus, natural dyes are demanded by the society as an alternative to the synthetic and harmful dyes (Kamel *et al*. 2005; Calogero and Marco 2008). There has been a recent increase in the use of natural dyes in cloth, wool, cotton, food, and cosmetic products, and various studies have been made on dyeing.

Goktas *et al.* investigated the development of environmentally friendly wood stains derived from laurel (*Laurus nobilis* L.) (2008a), oleander (*Nerium oleander* L.) (2009a), and madder root (*Rubia tinctorium* L.) (2009b) and determined the color stability of the stains when exposed to ultra-violet (UV) light irradiation. Their results showed that the wood stains derived from laurel, oleander, and madder root extract provided some color stability after UV irradiation. Goktas *et al.* (2008b) investigated the antifungal properties and color stability under UV exposure of wood treated with aqueous solutions of *Juglans regia* extract. Results showed that the lightness values of the wood specimens did change slightly.

From early antiquity, pomegranate has been cultivated throughout the Mediterranean and North African regions, including Central Saharan oases, for its valuable fruit. Considering the locations and context of pomegranate representations and archaeobotanical evidence, this fruit has held a long-time position as a luxury food (Bosi *et al.* 2009). At the Villa Rustica in Oplontis, over a ton of carbonized pomegranates, dated around the time of the Roman empire, were discovered (Jashemski and Meyer 2002). Several parts of the plant were used as both a tanning agent and dye. The dried fruit rind yields a yellow dye which can be used for dyeing clothes, making a hair dye, or even as a mordant (Dastur 1964).

Black mulberry, which originates from Iran, is cultivated for its fruit in southern Europe, southwest Asia, and in the Mediterranean (Tutin 1996). Black mulberry is widely grown in Turkey (Yaltirik 1982). Northeastern Turkey, the Coruh valley in particular, has notable populations of black mulberry, which have been cultivated in gardens for their delicious fruits. Recently, wild edible plants have received much attention as sources of biologically active substances including antioxidants, antimutagens, and anticarcinogens (Dillard and German 2000). Numerous studies carried out on plants have resulted in the development of natural antioxidant formulations for food, cosmetics, and other applications.

Wood-based products and decoration elements are potential sources for a number of volatile organic compounds (VOCs) that may be released indoors (Goktas 2009). Many chemical components are used in wood finishes and the coatings industry. Salthammer *et al.* (1998) identified about 150 different VOCs. These pollutants are emitted from different sources such as floor coverings, wood-based panel, furniture, solid woods, wood stains, and paints (Cheng and Brown 2003). Billions of people in the world suffer from diseases resulting from low air quality, and trillions of dollars are spent to compensate for such problems (Mo *et al.* 2009).

For many years, it has been known that many synthetic dyes are dangerous to human health, and the industry has been seeking environmentally friendly products (Peker *et al.* 2012). In this study colorants extracted from pomegranate (*Punica granatum*) and black mulberry were used as colorants for wood. These substances have been used for textiles but have never been used as natural colorants in wood industry.

The objectives of this study were to develop an environmentally friendly, natural wood dye derived from pomegranate (*Punica granatum*) and black mulberry (*Morus nigra*) instead of synthetic dyes and to determine its color stability. The CIELab system (CIE 1976 L*a*b*) was used to measure the color change of the colored wood materials after periods 50, 100, 150 h of UV light exposure.

EXPERIMENTAL

Wood Materials

Scots pine (*Pinus sylvestris* L.) and Oriental beech (*Fagus orientalis* L.), woods commonly used in the furniture and decoration industries in Turkey, were chosen for this experiment. The samples were prepared from first-class wooden materials, which are smooth fiber, knotless, crack-free, without color and density difference, with annual rings perpendicular to the surfaces, and from sapwood, in accordance with TS 2470 standards (TS 2470 1976).

The samples prepared with dimensions of 150 × 75 × 0.5 mm and were kept under suitable temperature (20 ± 2 °C) and suitable moisture conditions (moisture of ± 12% and relative humidity of ± 65%) until they became air-dried to achieve the moisture value in furniture used in interior areas, in accordance with TS 2471 1976 standard.

Plant Materials

In this study, the peels of pomegranate and black mulberry were gathered from the Karaman area in Turkey.

Preparing Dye Extracts

A weighed amount of dry plant material was extracted with distilled water in an ultrasonic bath (Elmasonic X-tra 150 H). The extraction percentage ratios were, by weight in distilled water, 25%, 30%, 35%, 40%, 45%, and 50%. Extraction was performed for 180 min at 45 °C and 180 W in a stainless, ultrasonic bath. Due to the rather high solution ratio, some manual stirring was sufficient to diffuse the plant material in the liquid during the extraction period. Volume loss due to evaporation was compensated for by the addition of water at the end of the extraction period to obtain the initial volume.

Dyeing Test Samples

The air-dried wood specimens were placed into an ultrasonic bath container and were treated by immersion method. Dyeing was performed for 60 min and at 45 °C. Any extra solution that remained on the specimens was removed with a clean cloth. Specimens were then left to dry at 20 ± 3 °C in a vertical position.

Accelerated Weathering Test

The accelerated weathering device operates in two stages. The first is the condensation stage. This stage induces expansion in the samples by changing the temperature and moisture of the environment at regular intervals to imitate outdoor environment conditions and by spraying hot steam thereon. In the second stage, the test pieces are subjected to UV radiation through the use of UV lamps. The weathering process was performed by operating the device for 4 h in the condensation stage and for 8 h in the UV stage. The average irradiance was about 330 nm at 50% relative humidity and 20 °C. Specimens were directly exposed to UV light at a distance of 20 cm and an angle of 90° (Kamdem and Grelier 2002). Eight replicate samples treated with each stain solution and untreated controls were irradiated for the randomly selected times of 0 (*i.e.*, no irradiation), 50, 100, and 150 h. The color change of the samples was measured after each irradiation period.

Color Measurements

To determine the color change values of the accelerated weathering tests, the colors of the coated parts were identified prior to weathering using a Konica Minolta CR–10, a portable color reader device. Because of the non-homogeneous color structure of the wooden material, color measurements were performed on each sample in two measures (cross-corners). The identified color values were classified according to the coordinates set by the *Commission International de I'Eclaireage-CIELAB* 1976 in ISO 2470 standards (Fig. 1). The obtained colors were represented with numerical values in the directions of “L”, “a”, and “b”. Here, “L” indicates light, “a” indicates red color values, and “b” indicates yellow color values. Coated sample pieces were subjected to color measurement prior to being exposed to the accelerated weathering environment and were labeled “color values prior to accelerated weathering”. Afterwards, the coated samples were placed on the weathering device, exposed to UV and condensation for 50 h, and color measurement was carried out again from the same cross-corners. This process was repeated after 100 and 150 h.

Determination of Color Change Values

Color changes due to accelerated weathering were calculated with the following formulas, in accordance with ISO 2470 standards.

$$\Delta L^* = L^*_{f} - L^*_{i} \tag{1}$$

$$\Delta a^* = a^*_{f} - a^*_{i} \tag{2}$$

$$\Delta b^* = b^*_{f} - b^*_{i} \tag{3}$$

$$\Delta E^* = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}} \tag{4}$$

Here ΔL^*, Δa^*, and Δb^* are the changes occurring between the initial (i) and final (f) states of the colors. ΔE^* indicates the total color changes occurring in the directions of *L*, *a*, and *b*. Here, the highest value shows the highest color change.

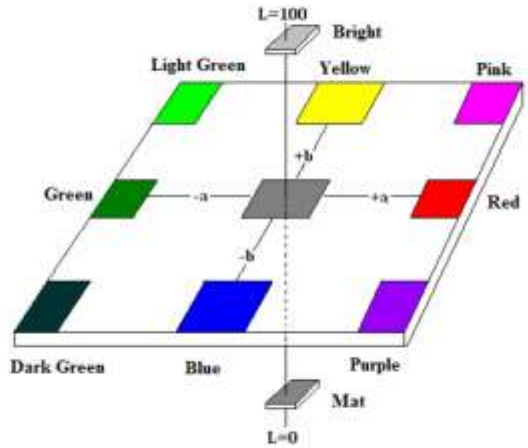

Fig. 1. CIELAB–76 color system

RESULTS AND DISCUSSION

Color changes values occurring on Scots pine and Oriental beech wood test samples that were stained with *Punica granatum* and *Morus nigra* extracts and exposed to UV application for 50, 100, and 150 h are numerically represented in Table 1. A schematic representation is given for pomegranate in Figs. 2 and 3 and for black mulberry in Figs. 4 and 5. Positive values of ΔL^* show whitening, and negative values of ΔL^* indicate the color turning grey. Positive values of Δa^* indicate reddening of the colors, and negative values of Δa^* show a shift towards green. Positive values of Δb^* represent yellowing in color, and negative values of Δb^* represent the color turning blue.

According to the results, both wood specimens exposed to UV radiation of 350 nm for all the exposure periods generally showed negative values of ΔL^*. This was attributed to chemical changes which occurred, especially in lignin, due to photodegradation resulting from UV exposure and, consequently, to a darkening of color on the wood (Peker *et al.* 2012). Feist and Hon (1984) reported that higher color changes were found in samples having higher lignin content. Lignin is the compound absorbing UV of 80 to 95% among the three main components of wood (Peker *et al.* 2012). The high negative values of ΔL^* indicate the sensitivity of that wood type against UV radiation and the surface quality thereof (Feist and Hon 1984).

The lightness values (ΔL^*) of both wood specimens treated with a pomegranate skin extract concentration of 50% were, after 150 h, comparatively higher than those treated with the other solution of pomegranate skin. The resulting lightness values (ΔL^*) of specimens treated with black mulberry for 150 h were determined to be 45% for Scots pine and 50% for Oriental beech wood. Generally, the Δa^* and Δb^* values of Scots pine specimens had increased much more than the Oriental beech wood specimens. Goktas *et al.* (2008b) reported Δa^* and Δb^* values similar to the results of this experiment. The general increase in the chromaticity coordinates of Δa^* and Δb^* for both wood species confirms that the chromophoric groups of the wood had been modified. Lignin is the wood component that has chromophoric groups capable of absorbing UV light in the range of 300 to 550 nm (Davidson 1996).

The best color stability (ΔE^* 20.23) of Oriental beech wood was attained with a 30% pomegranate skin extract after 150 h of exposure. For Scots pine wood, treatment with 25% pomegranate skin extract resulted in a higher color change (ΔE^* 31.74) after 150 h of UV radiation exposure compared to treatment with other pomegranate skin solutions. Color stability tests with black mulberry on Oriental beech wood showed that a 30% black mulberry extract provided the best color value (ΔE^* 14.92) after 150 h of exposure. Specimens of Scots pine wood treated with 50% black mulberry extract showed better color stability (ΔE^* 27.46) after 150 h of UV radiation exposure compared to other treatments.

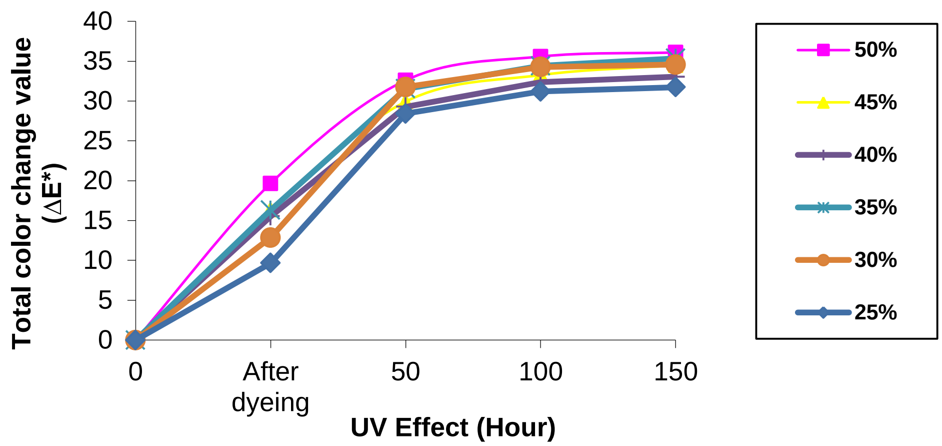

Fig. 2. Total color change in Scots pine wood dyed with pomegranate

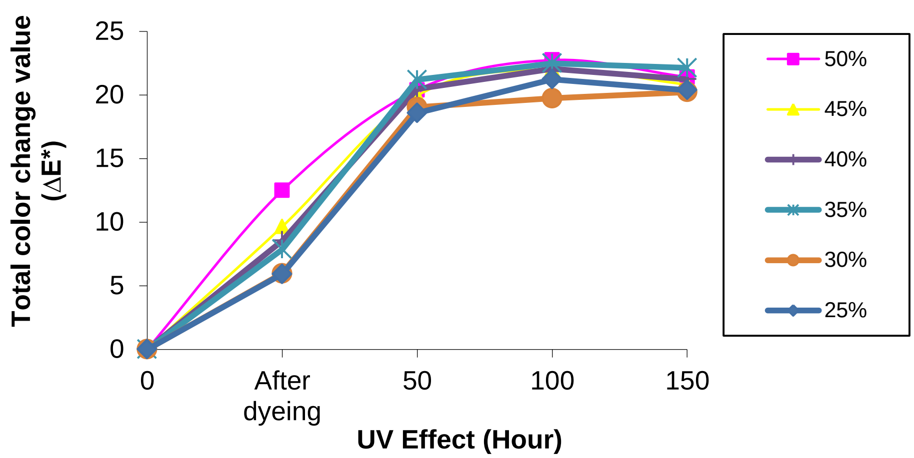

Fig. 3. Total color change in Oriental beech wood dyed with pomegranate

In general, it can be seen that most of the change in color had already occurred during the first 50-h period of exposure. Nevertheless, lesser changes occurred in subsequent periods. The first part of the color change process is because of the formation of chromophoric groups as carbonyl and carboxyl groups resulting mainly from degradation of α-carbonyl, biphenyl and ring-conjugated double bond structures in lignin and moving the extractives towards to wood surface from inside of wood (Lin and Kringstad 1970; Hon and Feist 1992). Relatively more rapid change is reported even if the wood material is exposed to UV light in external environment for short period or under accelerated weathering (Feist and Hon 1984; Peker 2012).

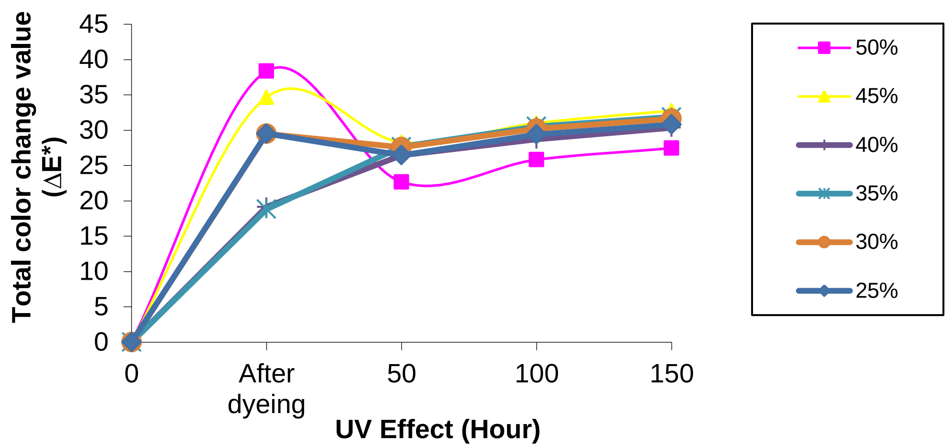

Fig. 4. Total color change in Scots pine wood dyed with black mulberry

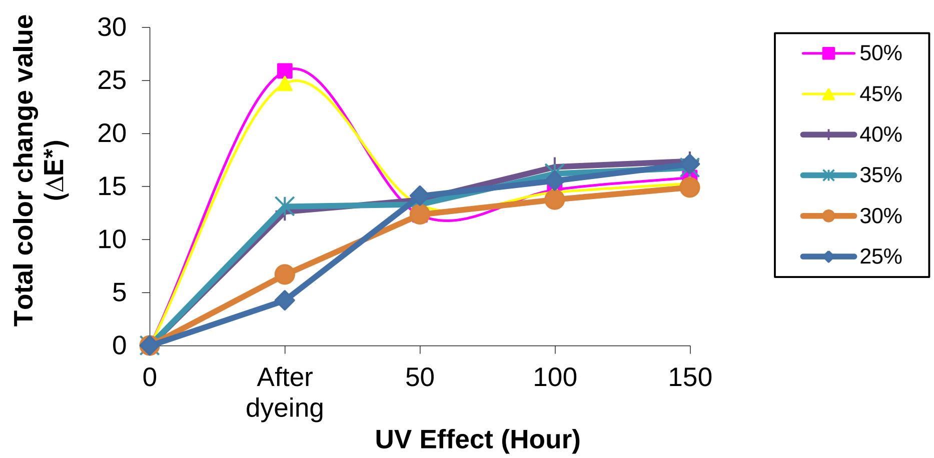

Fig. 5. Total color change in Oriental beech wood dyed with black mulberry

It was observed that, for both dye types, total color differences (ΔE^*) of Scots pine wood were higher than those of Oriental beech wood after 50, 100, and 150 h of exposure. The roughness of color stability in wood is a complex phenomenon affected by wood being an anisotropic and heterogeneous material along with various factors such as anatomical differences, growing characteristics, machining properties of the wood, and pre-treatments (*e.g.*, steaming, drying) (Temiz *et al.* 2005). Exactly the same ΔE^* values were found with the same dyestuff and same wood type, which indicated close agreement in color changes values.

Table 1. Color Change Values in UV Application for 150 h for Dyed Test Samples

Wood type	Dye extract	Concentration (%)	After dyeing				After 50 h				After 100 h				After 150 h			
			ΔL*	Δa*	Δb*	ΔE*	ΔL*	Δa*	Δb*	ΔE*	ΔL*	Δa*	Δb*	ΔE*	ΔL*	Δa*	Δb*	ΔE*
Scots pine	Pomegranate	50	-7.96	4.05	17.51	19.66	-25.54	15.46	13.07	32.59	-27.55	17.89	13.67	35.58	-28.79	17.78	12.56	36.09
		45	-5.86	2.72	15.01	16.34	-22.55	14.05	13.76	29.92	-24.41	16.72	15.19	33.26	-26.33	17.03	14.35	34.48
		40	-6.58	2.05	13.92	15.53	-22.28	12.61	14.23	29.29	-23.12	15.58	16.42	32.36	-25.07	15.42	15.02	33.04
		35	-7.91	2.78	13.99	16.31	-23.63	13.38	16.03	31.53	-24.39	16.12	18.15	34.41	-26.48	16.23	16.87	35.34
		30	-5.71	1.86	11.37	12.86	-23.99	14.47	14.95	31.76	-24.91	16.80	16.46	34.26	-26.33	16.58	15.05	34.56
		25	-4.56	1.55	8.38	9.67	-20.33	13.00	15.00	28.41	-21.60	15.44	16.37	31.19	-23.02	15.60	15.30	31.74
Oriental beech		50	-4.42	1.49	11.60	12.50	-15.25	7.04	11.59	20.40	-16.63	9.68	12.13	22.75	-15.23	8.28	12.57	21.41
		45	-2.75	0.91	9.21	9.65	-13.35	7.26	12.94	19.96	-14.44	9.41	13.71	22.02	-13.28	8.39	13.66	20.82
		40	-2.66	-0.42	8.11	8.54	-13.80	6.92	13.46	20.48	-12.80	8.51	15.82	22.05	-13.74	7.74	14.24	21.24
		35	-2.44	-0.76	7.45	7.87	-15.29	7.32	12.73	21.20	-14.71	9.31	14.26	22.50	-15.41	8.50	13.42	22.13
		30	-2.07	-0.63	5.57	5.97	-13.37	7.18	11.50	19.04	-13.84	8.81	10.99	19.74	-11.37	9.18	13.99	20.23
		25	-0.53	0.94	5.83	5.92	-10.69	7.42	13.27	18.58	-11.44	9.97	14.87	21.24	-12.34	8.86	13.60	20.39
Scots pine	Black mulberry	50	-24.42	27.78	-10.18	38.36	-15.69	8.68	13.88	22.68	-17.23	11.18	15.66	25.83	-19.75	11.79	15.00	27.46
		45	-21.68	25.20	-9.88	34.68	-21.55	13.28	12.65	28.30	-23.64	15.44	12.82	31.01	-25.96	15.83	12.22	32.77
		40	-14.74	10.98	-5.44	19.17	-20.31	11.37	12.53	26.43	-21.98	13.33	12.80	28.72	-24.23	13.79	12.01	30.36
		35	-14.72	11.34	-3.00	18.82	-21.01	11.59	13.77	27.67	-23.33	13.94	13.91	30.53	-25.23	14.43	13.10	31.88
		30	-24.95	12.18	-9.97	29.50	-19.70	11.79	15.31	27.60	-21.50	13.98	15.99	30.22	-23.65	14.47	15.21	31.62
		25	-25.51	12.41	-8.20	29.53	-18.58	10.45	15.68	26.46	-20.31	12.82	16.98	29.41	-22.20	13.61	16.48	30.81
Oriental beech		50	-16.09	17.34	-10.55	25.89	-8.72	3.33	8.02	12.31	-9.90	5.07	9.61	14.70	-11.45	5.40	9.54	15.85
		45	-15.58	16.38	-9.96	24.70	-8.72	4.68	8.80	13.25	-8.89	5.91	9.76	14.46	-10.12	6.03	9.75	15.29
		40	-9.37	7.49	-3.96	12.63	-8.53	3.65	10.14	13.75	-9.92	5.83	12.33	16.86	-11.01	5.85	12.14	17.40
		35	-9.48	7.77	-4.70	13.12	-8.50	3.68	9.59	13.33	-9.45	5.71	11.89	16.22	-10.82	6.01	11.30	16.76
		30	5.46	2.42	3.04	6.70	-7.24	2.98	9.57	12.36	-7.29	4.22	10.90	13.77	-8.44	4.64	11.39	14.92
		25	0.83	4.18	0.38	4.27	-9.15	4.24	9.88	14.12	-9.48	5.54	11.01	15.55	-10.93	6.28	11.59	17.12

CONCLUSIONS

1. The total color difference of treated Oriental beech wood was lower than that of treated Scots pine after 150 h of exposure. The ΔE^* values of all concentrations were somewhat similar within the same dye source and wood specimens. Oriental beech wood treated with 30% pomegranate skin extract or black mulberry and Scots pine wood treated with 25% pomegranate skin extract or 50% black mulberry extract showed the best color stability.
2. The average color change values were found to be different between each wood types. This is caused by the chemical formation difference between the tree groups (soft/hard) (Temiz *et al.* 2005; Sogutlu and Sonmez 2006; Peker *et al.* 2012). However, color change in the wood is known to result from many factors, and such factors include such complicated processes as heterogonous anatomical structure of the wood, the conditions of the place where they are grown, handling conditions, and whether pre-treatment is applied (Temiz *et al.* 2005; Peker *et al.* 2012).
3. These studies have shown potential sources for natural, plant-based dyes that pose no threat to human or environmental health. These dyes may be used as colorants and protective materials in the surface treatments of furniture and, through their proliferation, may open new avenues of business. Hence, naturally sourced and more economical surface treatment substances that have suitable coloring properties and that are harmless to the environment and human health may be developed.

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