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Full Length Research Paper

Usage opportunities of the natural dye extracted from acorn (*Quercus ithaburensis Decaisne*) in the furniture industry upper surface treatment

Huseyin PEKER[1]*, Abdi ATILGAN[1], Hatice ULUSOY[1] and Osman GOKTAS[2]

[1]Department of Forestry Industrial Engineering, Faculty of Forestry, Artvin Coruh University, 08000, Seyitler Campus, Artvin, Turkey.
[2]Department of Wood Works and Industrial Engineering, Faculty of Technology, Mugla University, 48000, Kotekli Campus, Mugla, Turkey.

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This study is aimed to develop a natural dye to be used in the furniture industry upper surface treatment and to be sensitive to the environment and human health. For this reason, the natural dye extracted from acorn *(Quercus ithaburensis Decaisne)* is applied to different types of wood. After that, the determination of color change under the accelerated weathering conditions and applicability as the upper surface material of this dye was researched. The mordanting process of this extract obtained from acorn was provided with 3% ferrous sulphate ($FeSO_4.7H_2O$), 5% potassium aluminum sulphate (alum) [$KAl(SO_4)_2.12H_2O$] and vinegar of grape 10%. The values of total color change of the natural dye samples applied to Scots pine *(Pinus sylvestris* L.*)*, Oriental beech *(Fagus orientalis* L.*)* and oak *(Quercus petraea* L.*)* chosen as wood test material were determined after accelerated weathering processes throughout 100, 200 and 300 h according to ISO 2470. The experiment results showed that while the least color change (ΔE = 2.27) was determined in the application of oak with vinegar mordant, the most color change (ΔE = 26.08) was determined in the application of Scots pine without mordant. According to these results, the dye obtained from acorn has an aesthetic appearance as a coloring and preservative upper surface material for indoor furniture and wooden toys.

Key words: Natural dyes, accelerated weathering, acorn, mordant, vegetable dyes, furniture industry upper surface treatments.

INTRODUCTION

Environmental pressures and prohibitions on commonly used wood preservatives due to their toxic components have necessitated usage and development of eco-friendly wood protection and surface treatment agents. Contamination especially exposed indoors in consequence of chemical protection and coloring of wooden material leads to negative impacts upon human health. Therefore, this has become a subject carefully followed by the society, particularly customers of this product, administrative sciences, industrial employees, and the researchers (Salthammer et al., 2002).

Recently, various researches have been conducted on the reasons of pollution of air breathed indoors, and it has been determined that such pollution is considerably resulted from "volatile organic compounds" (VOC). Such compounds are composed of typical solvents, which are aliphatic and aromatic hydrocarbons, alcohols, ketones and esters (Salthammer et al., 2002).

In a research conducted by Salthammer et al. (1998), it is stated that there are approximately 150 sources in emission of volatile components into modern closed areas and the majority thereof results from textile, furniture, and wooden products. If we detail these volatiles, we can confront paints, lacquers, paint cleaners, cleaning supplies, pesticides, glues, and adhesives (http://www.epa.gov/iaq/voc.html, 2009). Some

*Corresponding author. E-mail: peker100@hotmail.com.

of these chemicals result in spread of bad odors, irritation in eyes, airways, mucous membranes, pathogenic properties, and some in cancer formation. It has been determined that even the resulting gases react with other substances in time and they may form secondary harmful substances (Salthammer et al., 1998).

For many years, it has been known that many synthetic dyes are dangerous to the human health and the industry seeks for environmentally friendly products. 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 environment (Kizil, 2000).

Apart from the harmful effect of the synthetic dyestuffs to the environment, since it is determined that they cause allergy in people, interest in natural dyes has been increased in recent years. Usage of plants as dye by the humankind dates back to centuries ago. Dye plants have become the dyestuffs of such industries as textile, food, leather, and the like (Korur, 1937; Mert et al., 1992).

Dye plants have many fields of usage. These include preparations of food colorants, textile, cosmetics, and pharmaceutical preparations (Piccaglia and Venturi, 1998).

Today, synthetic dyestuffs are manufactured through nanotechnological methods. The manufactured dyestuffs are also known to exhibit antibacterial properties. Therefore, billions of people in the world suffer from diseases resulting from low air quality and trillions of dollars are spent to compensate thereof (Mo et al., 2009).

Studies are on progress in every field in order to develop harmless products as an alternative to all the products harmful to human and environmental health. As human and environmental health come into prominence, the states develop new protective standards; thus, natural dyes are demanded by the society as a significant alternative to the synthetic based and harmful dyes (Kamel et al., 2005; Calogero and Marco, 2008; Tsatsaroni et al., 2003).

Painting by using plants dates back to many years ago in Anatolia. In Ottoman period, dyeing was made and dye plants were grown where people were engaged in handicrafts such as carpet and rug weaving. These centers included especially Bursa, Istanbul, Tokat, Kayseri, Ankara and Konya. An important dye plant in dyeing, acorn has been used as human and animal food since the Paleolithic Age (before 50,000 BC ago). It was used in leather dyeing and tanning in the past. It was frequently used in obtaining black color with iron mordant in Turkish rugs and carpets. However, since the dyestuff in its content is tannin, there is wear in sections painted black in the long run. Turkish oak (*Quercus cerris*) is a species of broad-crested deciduous oak that is generally grown in the hills with low altitude, can grow taller up to 15 to 20 m, and can grow in diameter of more than one meter. Its fruit generally matures in two years. It grows in Turkey and Greece, especially in the Aegean Region. Oak cupule is half-sphere with a diameter of 4 to 6 cm, is covered by cups, and these cups are covered by scales. It has the largest cupule among the oak species. The fruit of Turkish oak is up to 3 cm, has a cylinder shape, a light brown color, and a bitter taste. About 20 oak (*Quercus*) species are present in Turkey. Fruit of these trees are gathered under the name of “acorn” without differentiation (Karadag, 1997).

Natural-sourced dyes have been used since the ancient ages in dyeing; however, many vegetable dyes are extracted today and various scientific studies are carried thereon. To exemplify: In the study carried out by Atilgan et al. (2011), pinar plant extract with painted wood species, under UV color changes were determined. The end of this study, while the least color change (ΔE = 9, 03) was determined in the application of Oriental beech with ferric sulphate mordant, the most color change (ΔE = 26, 08) was determined in the application of Scots pine without mordant. Recent studies have been conducted on usage of natural dyes extracted from plants in coloring furniture and wooden products. Many plant-derived dyes used in textile painting works made with natural products have antibacterial properties and these can be exemplified by walnut fruit peel extracts (Goktas et al., 2008).

In this study, the purpose is to extract natural wood dye from Valonia oak *(Quercus ithaburensis subsp. Macrolepis/Quercus ithaburensis Decaisne)* used in dyeing carpets and rugs in Anatolia, and the values of color change for the extracted dyes were determined under rapid weathering conditions.

MATERIALS AND METHODS

Wood materials

As wood material, Scots pine (*Pinus sylvestris* L.), Oriental beech (*Fagus orientalis* L.), and oak (*Quercus petraea* L.) woods commonly used in furniture and decoration industries in Turkey were chosen. 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 parts of sapwood, in accordance with TS 2470 standards (TS 2470, 1976). The samples prepared in dimensions of 50 × 75 × 10 mm were first polished with sandpapers No. 80 and 100 and the polished surfaces were thoroughly cleaned from its dust with a soft-bristled brush before dyeing. The pieces were kept under suitable temperature (20 ± 2°C) and suitable moisture (moisture of ±12% and relative humidity of ±65%) conditions until they became air-dried, in order to achieve the moisture value in furniture used under interior area conditions in accordance with TS 2471, 1976 standard.

With respect to the parts list of wood types, 60 sample parts (4×5×3) in total were prepared 5 sample parts, 4 types of dyes, and 3 types of woods, for each solution.

Plant material

In this study, oak powder obtained by drying and grinding a woody

Table 1. Dye extract + mordant mixture ratios.

Mordant	Mixture ratio [water (ml)/dye plant/(g) mordant (g)]
Without mordant	150/25/none
Alum	150/25/7.5
Ferrous sulphate	150/25/4.5
Vinegar	150/25/15

Prepared dye mixtures; Mixture 1: Dye extract prepared without mordant; Mixture 2: Dye extract mixed with alum mordant; Mixture 3: Dye extract mixed with ferrous sulphate mordant; Mixture 4: Dye extract mixed with vinegar mordant.

plant species Valonia oak (*Quercus ithaburensis* Decaisne) frequently used in dyeing wools, hand-woven carpets and rugs in Anatolia as a dyestuff. The acorns were gathered from the Afyonkarahisar area (Turkey).

Preparing dye extracts

Dye extracts were prepared by boiling water, in which 25 g of the granulated Walloon oak (*Quercus ithaburensis* Decaisne) plant were transferred into glass containers. In order to ensure that the dye disperses in the water homogenously and water temperature is kept at equal level, a magnetic stirrer was placed into a glass container. Dye extract was obtained by percolating through a filter cloth after boiling the aqueous mixture at 100°C for 1 h. It was brought to the water level indicated initially by adding water in the amount of vaporized water and dye extract was divided into 4 equal parts for one mordant and three mordant-free mixtures. It was divided into (150 ml) for the dye mixtures without or with mordant. The mixtures added with mordant were stirred in a glass container using a shaker (magnetic stirrer) and mordant were ensured to mix with the dye homogenously. Aqueous solutions were mordanted by adding ferrous sulphate ($FeSO_4.7H_2O$) of 3%, Potassium aluminum sulphate (alum) [$KAl(SO_4)_2.12H_2O$] of 5%, and grape vinegar of 10% in order to stabilize the color of dyes extracted from oak powder, to ensure it to hang on the applied material (to increase retention amount), and to create color options. Dye extracts and mordant mixture ratios are given in Table 1.

Dyeing test samples

After the extracts were cooled at room temperature (25°C), they were applied on the sample parts in one layer using a fine wire brush and kept to dry in shade for 2 h. Then, the second coat of dye was applied to the dried samples.

Color measurements

In order to determine the color change values in the accelerated weathering tests, the colors of the coated parts were identified prior to weathering by using Konica Minolta CR–10, a portative color reader device.

Color measurements were performed on each sample due to the non-homogenous color structure of the wooden material in two measures (cross-corners). The identified color values were classified according to the coordinates *Commission International de l'Eclaireage-CIELAB_1976)* set in ISO 2470 standards (Figure 1). The obtained colors were indicated with numerical values in the directions of L, a, and b. Here, L indicates the light, a red color, and b yellow color values. Coated sample pieces were subjected to color measure prior to be exposed to accelerated weathering environment and they were stated as "color values prior to accelerated weathering". Afterwards, the coated samples were placed on the weathering device, were exposed to UV+ condensation environment for 100 h, and color measurement was carried out again from the same cross-corners. This process was repeated at the 200th and 300th h.

Accelerated weathering test

Operation of accelerated weathering device is composed of two periods. The first is condensation stage. This stage ensures the sample pieces to expand by changing the temperature, coolness, and moisture amount of the environment at regular intervals in order to imitate the external environment conditions and by spraying hot steam thereon. In the second stage, the test pieces are subjected to UV beams by using UV lamps. Weathering process was performed by operating the device for 4 h in the condensation stage, and for 8 h in UV period.

Determination of color change values

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

$$\Delta L^* = L^*_f - L^*_i$$

$$\Delta a^* = a^*_f - a^*_i$$

$$\Delta b^* = b^*_f - b^*_i$$

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

Here; ΔL^*, Δa^* ve Δb^* are the changes occurring between the initial state (i) and final state (f) of the colors.

ΔE^*, indicates total changes of the colors occurring in the directions of L, a, and b. Here, the highest value shows the highest color change.

RESULTS

Color change values occurring on Scots pine, Oriental beech, and Valonia oak wood test samples coated with oak (acorn) extract and exposed to UV application for 100, 200, and 300 h are shown in Table 2 numerically, and in Figures 2 to 4 schematically.

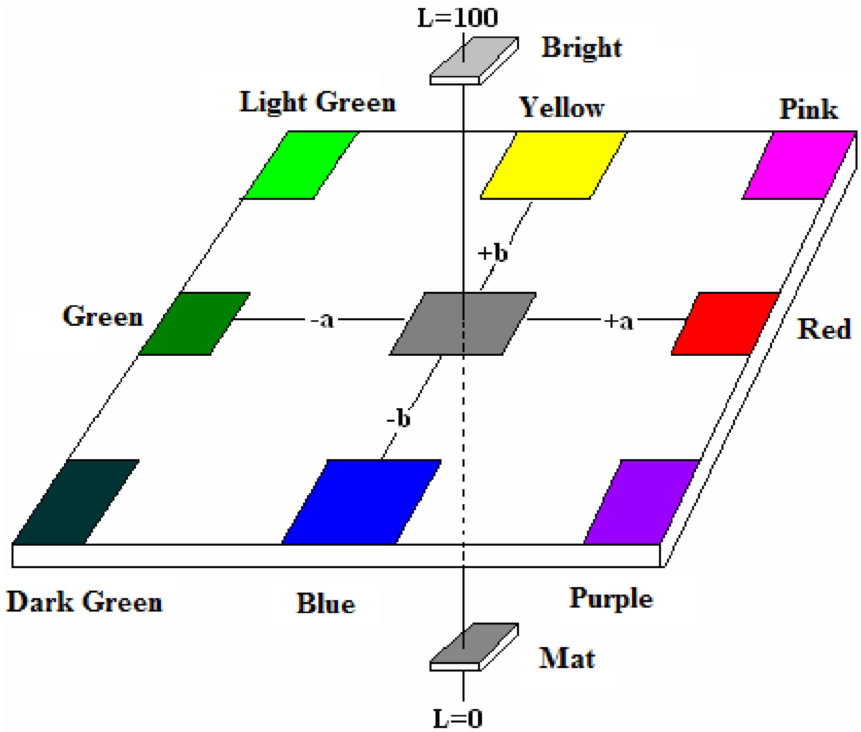

Figure 1. CIELAB–76 color system.

In the application of natural dye extracted from acorn powder on Scots pine wood samples, the least color change was observed in the ferrous sulphate mordant application (ΔE: 8.91) after accelerated weathering for 300 h. After application of the first 100 h, not much color change was observed in all the colors.

Change values of the colors extracted from acorn for accelerated weathering on Scots pine wood test samples are shown in Table 2, and graphics thereof is provided in Figure 3.

In the application of natural dye extracted from acorn powder on Oriental beech wood samples, the least color change was observed in the ferrous sulphate application (ΔE: 9.03) after accelerated weathering for 300 h.

Change values of the colors extracted from acorn for accelerated weathering on Oriental beech wood test samples are shown in Table 2, and graphics thereof is provided in Figure 4. In the application of natural dye extracted from acorn powder on Valonia oak wood samples, the least color change was observed in the ferrous sulphate mordant application vinegar (ΔE: 11.53) and ferrous sulphate (ΔE: 11.97) mordant after accelerated weathering for 300 h.

DISCUSSION

When we look at the color change in general (Table 2), positive values of Δa* indicate reddening of the colors, and negative values of Δa* show a shift to the green color. Positive values of Δb* represent yellowing in the color, and negative values of Δb* represent the color turning blue. Positive values of ΔL* show whitening, and negative values of ΔL* indicate the color turning grey. After the UV application of 300 h, the highest change in ΔL* values is in the negative direction (ΔL = -24) and from the mordant-free application carried on Scots pine. According to Table 2, it is seen that all the ΔL* values are negative. This was attributed to chemical change occurring especially in lignin as a result of photodegradation resulting from UV process, and consequently, to the darkening of color on wood. This darkening of color on wood occurs because of the free radicals formed on the wood after transformation of hydrogen peroxide generated from the quick interaction between the light and the oxygen into carbonyl, carboxyl, peroxide, hydro peroxide, and double-bonded pigment-containing groups by dissolving. The high negative values of ΔL* indicate the sensitivity of that wood type against UV and surface quality thereof (Feist and Hon, 1984).

In general, it is observed that color changes occur quickly in 100-h periods; however, a less change occurs in the subsequent periods. Relatively more quick change is reported even if the wood material is exposed to UV light in external environment for short periods or under accelerated weathering (Feist et al., 1984). Such change style is observed in the studies conducted by Kandem et al. (2002). The lowest color change value (ΔE* = 8.91) is

Table 2. Color change values in UV application for 300 h for dyed and undyed test samples.

Wood type	Dye extracts	Prior to weathering			After 100 h				After 200 h				After 300 h			
		L*	a*	b*	ΔL*	Δa*	Δb*	ΔE*	ΔL*	Δa*	Δb*	ΔE*	ΔL*	Δa*	Δb*	ΔE*
Scots pine	Mordant-free	62	6.42	25.98	17.34	10.38	4.14	20.62	-20.91	9.98	-2.08	23.26	-24	9.86	-2.72	26.08
Scots pine	Acorn + aluminum sulphate	57.59	10.17	26.24	-6.65	7.59	8.1	12.93	-8.27	8.09	5.21	12.68	-9.97	7.79	5.26	13.70
Scots pine	Acorn + ferrous sulphate	26.82	-0.51	-5.4	-8.52	0.66	5.06	9.93	-7.6	0.71	6.76	10.19	-5.96	0.91	6.57	8.91
Scots pine	Acorn + vinegar	58.66	10.99	26.88	-9.31	5.04	4.63	11.55	-9.96	5.95	4.5	12.44	-10.56	7.66	6.52	14.58
Oriental beech	Mordant-free	50	13.21	24.56	-7.94	3.13	0.8	8.57	-8.79	5.89	3	10.99	-11.21	4.71	0.14	12.16
Oriental beech	Acorn + aluminum sulphate	58.59	8.15	23.38	-12.41	7.81	2.84	14.93	-15.75	10.65	3.47	19.32	-17.48	8.73	1.2	19.57
Oriental beech	Acorn + ferrous sulphate	26.42	-0.38	-5.39	-6.34	0.61	5.96	8.72	-5.81	0.76	6.97	9.10	-4.79	1.14	7.58	9.03
Oriental beech	Acorn + vinegar	56.72	12.84	26.5	-11.42	4.55	1.37	12.36	-11.95	6.94	3.41	14.23	-14.01	5.14	-0.36	14.92
Oak	Mordant-free	53	11.15	26.01	-0.48	1.59	2.4	2.90	-8.18	6.14	4.07	11.00	-12.48	6.42	-1.31	14.09
Oak	Acorn + aluminum sulphate	57.96	4.65	20.33	-7.88	0.01	-3.24	8.52	-18.43	10.27	5	21.68	-20.64	10.73	2.62	23.40
Oak	Acorn + ferrous sulphate	28.37	-0.66	-5.27	-10.28	1.21	9.44	14.00	-10.21	0.86	5.47	11.61	-10.36	0.75	5.95	11.97
Oak	Acorn + vinegar	52.6	11.96	26.09	-1.18	0.75	1.79	2.27	-7.49	5.08	5.49	10.58	-9.84	6.02	0.05	11.53

acquired from the application of acorn dye on Scots pine wood with ferrous sulphate mordant, and the highest color change value (ΔE* = 26.08) from the application of acorn dye on Scots pine without mordant. When we look at the color change values of control samples without mordant, it is observed that the application performed with ferrous sulphate mordant increases the color stability; in other words, the colors undergo less change. This is attributed to the fact that metal ions contribute to the formation of complexes of free radicals and wood components even when the wood material is exposed to UV beam, and it is emphasized that formation of complex between iron and lignin enables such color stability (Kandem et al., 2002). In similar studies, it is indicated that mordanting ensures color stability in the wools coated with natural dyes as well (Olmez, 2004).

When we look at the average color change values in the wood types, the highest change value (ΔE* = 26.08) is obtained from the application of Scots pine without mordant, and the lowest value (ΔE* = 9.03) from the application of ferrous sulphate mordant on beech wood. In the similar studies in the literature, it is observed that the color change in the material obtained from coniferous trees is higher than those obtained from the broad-leaved trees (Sahin, 2002). It is stated that this is caused by the chemical formation difference between the tree groups (soft/hard) (Temiz et al., 2005; Sogutlu and Sonmez, 2006). In other words, coniferous trees have more lignin than the broad-leaved trees at 2 to 10% in general. Lignin is the compound absorbing UV of 80 to 95% among the tree components. Feist and Hon (1984) reported that higher color change in soft trees than that of the hard trees results from higher lignin rate. However, it is indicated that color change in the wood results from many factors, and such factors include such complicated processes as inhomogeneous 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).

This study is carried out in order to develop natural surface dyestuffs extracted from acorn as an alternative to synthetic based dyestuffs, and to determine color change performances of application of such dyes on wood types under conditions exposed to UV for 100, 200, and 300 h. According to the study results, it is found that color change performances of ferrous sulphate mordant applications are higher. Vinegar mordant applications provide resistance as much as other mordant applications; thus, a 100% natural dyestuff for wood surfaces is developed by using natural mordant in natural dye application. Aluminum sulphate is observed to be the mordant type with the highest color change among all the wood types in general.

All of the dyes extracted from acorn powder have the aesthetic appearance, which can be used on furniture and decoration surfaces. Important contributions will be able to be obtained

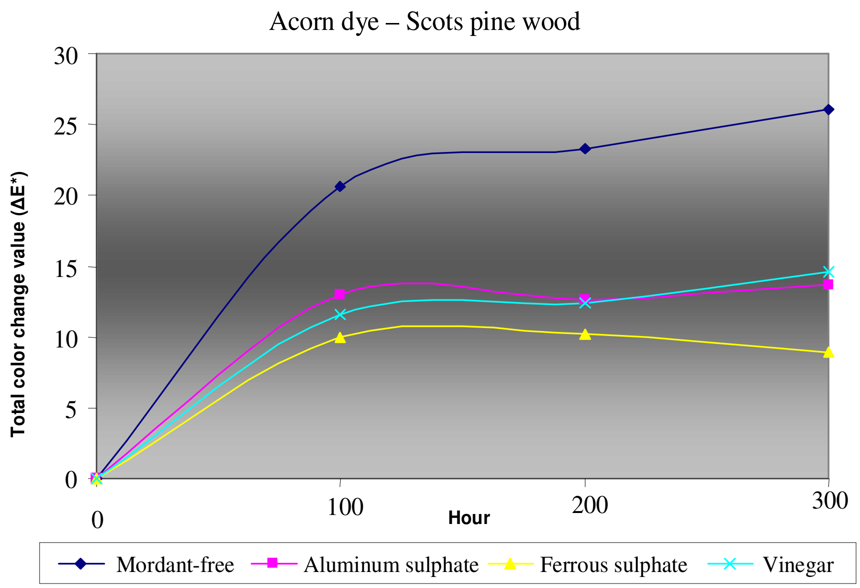

Figure 2. Graphics of total color change in Scots pine.

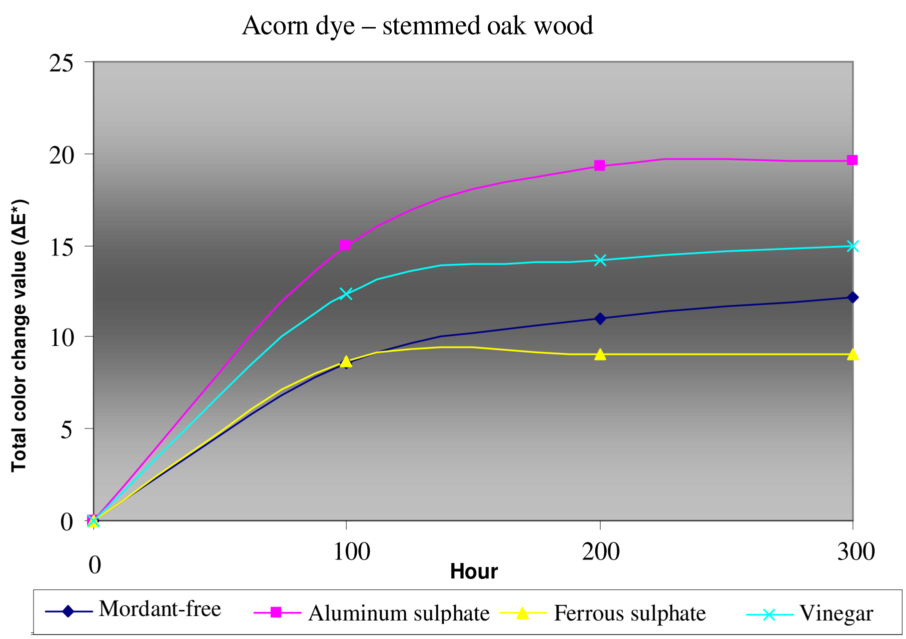

Figure 3. Graphics of total color change in oriental beech.

to environmental and human health owing to the usage of such type of natural dyes in particular in interior furniture, children's toys and stuff, and interior decoration of wood structures. Turkey has the potential of approximately 150 dye plants, and does not benefit there sufficiently (www.turkelhalilari.gov.tr/doğalboyamacilik, 2009). In consequence of these studies, natural plant sources that have a great potential, but are not benefited will be able to be activated through ensuring and disseminating usage of natural dye plant extracts harmless to environmental and human health as colorant and protective materials in surface treatments for furniture units, and by means of proliferation thereof, new line of business will emerge (bioenergy). Hence, naturally sourced and more economical surface treatment substances that bear coloring properties and are harmless to environmental and human health will be able to be developed. As implemented in other industrial

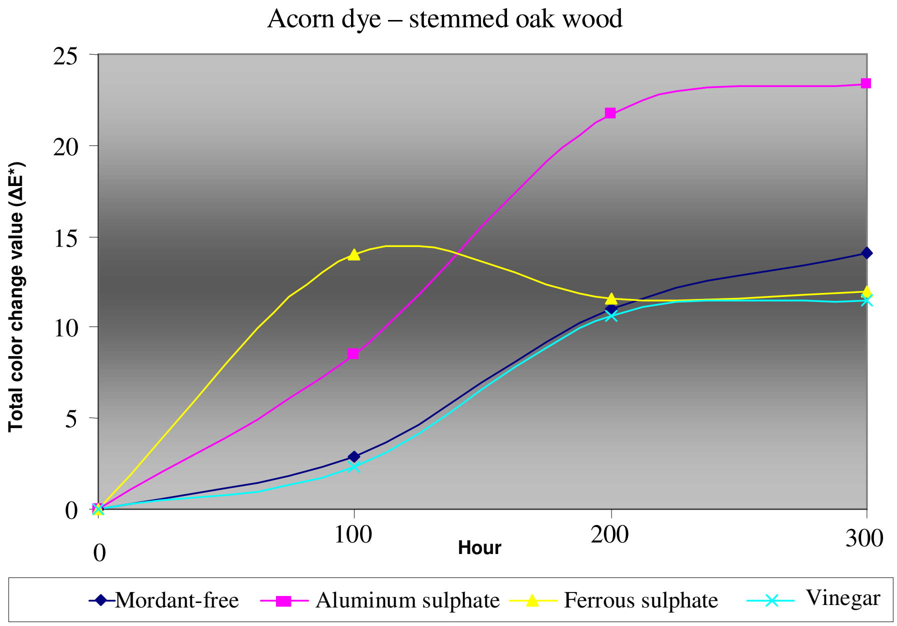

Figure 4. Graphics of total color change in Valonia oak.

sectors, a contribution will be made to the development of natural wood surface treatment materials as an alternative to the chemical substances today. Therefore, subsequent studies can concentrate on natural dyestuffs like synthetic based dyestuffs with nanotechnology, and they can be tested in accelerated weathering environment and color change values can be compared. Different researches can be conducted to increase availability thereof on wood products used outdoors.

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