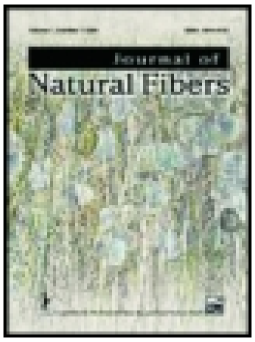

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Dyeing Properties of Natural Dyes Extracted from the Junipers Leaves (*J. excelsa* Bieb. and *J. oxycedrus* L.)

Halil Özdemir

Osmaniye Korkut Ata University, Osmaniye Vocational School, Osmaniye, Turkey

ABCTRACT

In this paper, two natural dyes were extracted from Junipers leaves (*Juniperus oxycedrus* L. and *Juniperus excelsa* Bieb.). Wool yarn (for carpet) was dyed with the extract using ferrous (II) sulfate, potassium dichromate and sodium sulfate as mordant. Then, CIELab (L*, a*, b*, c,* and h) values, color differences (ΔE) and color strength (K/S) values of dyed wool yarns were examined and the fastness properties of dyed wool against dry and wet rubbing were evaluated. According to the experimental results, the use of mordant increased the color strength of dye goods. All mordant increased the rub fastness of dyed samples such as similar scientific studies previously. Compared to two junipers, dyeing samples of *J. excelsa* are dyed darker than that of *J. oxycedrus*.

摘要

在本文所涉的研究中，从刺柏（大果刺柏和棕竹刺柏）叶子中提取两种天然染料。通过使用亚铁（Ⅱ）硫酸盐、重铬酸钾和硫酸钠作为媒染剂提取物对羊毛纱（地毯用）进行染色。然后，检测经过染色的羊毛纱的 CIELab（L＊、 a＊、b＊、c＊和 h）值、色差（ΔE）和颜色强度（K/S）值，并对染色羊毛的干摩擦和湿摩擦耐磨色牢度进行了评估。根据实验结果，媒染剂的使用提高了染料产品的色牢度。所有媒染剂都提高了染色样本的耐摩色牢度，这与以前的类似科学研究结果一致。具体到这两种刺柏，棕竹刺柏的样品染色深度高于大果刺柏。

KEYWORDS
Color properties; junipers; mordant; natural dyeing; quercetin; rub fastness

关键词
颜色性质；刺柏；媒染剂；天然染色；槲皮素；耐摩色牢度

Introduction

A variety of climate and ecological conditions in Anatolia (Asia Minor) is reflected in the generosity of the nature which offers an extensive diversity of both fauna and flora. Many plants have been found here as endemic species and others are represented by numerous genera and species. Juniper is the forest tree which is extensively available in Turkey. It takes an important place as an area and stem volume especially in Mediterranean Region. Juniper can grow on poor areas that other forest trees cannot grow. It's so hard to bring the other kinds of trees to Juniper areas (Carus 2004; Uçar and Balaban 2002).

Juniperus excelsa grows in the Mediterranean region, central and south Balkans, Anatolia, Caucasus, central and southwest Asia, Iran, Iraq and the Arabian Peninsula. This plant is used both medicinally and non-medicinally. The most common use is concerned with wood as fuel and material for building houses and fences as well as making household items. Gum is mainly used for medicinal purposes such as relieving pains. Leaves are sometimes used for incense and producing natural dyestuffs (Milios et al. 2007; Pirani et al. 2011; Topçu et al. 1999). Mishra and Agrawal

CONTACT Halil Özdemir halilozdemir@osmaniye.edu.tr Osmaniye Vocational School, Osmaniye Korkut Ata University, 80000 Osmaniye, Turkey.

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revealed some pharmacological properties of *J. excelsa* extracts including depressant activity in albino rats. I. Muhammed and friends, report the results on the bioactivity—guided isolation of the antimicrobial compounds from a local collection of *J. excelsa* leaves (Mishra and Agrawal 1989; Muhammed et al. 1992).

Juniperus L. is represented in Turkey by eight taxa. *Juniperus oxycedrus* has two subspecies—*subsp. oxycedrus* and *subsp. macrocarpa*—in Turkey. The main use of *J. oxycedrus* is to prepare the so-called oil of cade (also known in pharmacy as juniper tar) by destructive distillation of the branches and wood of the plant. This empyreumatic oil has been widely employed in human and veterinary dermatology in order to treat chronic eczema and other skin diseases as well as rectified cade oil is used as a fragrance component in soaps, detergents, creams, lotions, and perfume (Salido et al. 2002; Sezik et al. 2005).

As the name suggests, natural dyes are derived from natural resources. Coloring materials obtained from natural resources of plant, animal, mineral, and microbial origins were used for coloration of various textile materials. Leaves, flowers, wood, bark, and so on of several plant materials have been utilized for the dyeing of various textile substrates with varying results in terms of deepness of color produced on the substrates and their colorfastness properties. Natural dyes are mostly non-substantive and must be applied on textiles by the help of mordants, usually a metallic salt, having an affinity for both the coloring matter and the fiber. These metallic mordants after combining with dye in the fiber, it forms an insoluble precipitate or lake and thus both the dye and mordant get fixed to become wash fast to a reasonable level (Samanta and Agarwal 2009; Samanta and Konar 2011; Saxena and Raja 2014). Mordants are often polyvalent metallic ions, which form a complex with the fiber and the coloring components of the dye. Most commonly used mordants are metallic salts such as alum, iron (II) sulfate, potassium dichromate, copper (II) sulfate, tin (II) chloride, etc., which help to fix the colorant to the fiber resulting in deeper shades with excellent fastness properties (Yusuf et al. 2013). In natural dyeing mordants increase the fixation of the dyestuff in various ways. In many cases mordants are metal salts that can form a metal complex with the natural colorant, which exhibits increased affinity to the substrate. Depending on the metal character the complex formation does not only strengthen dyestuff fixation on the substrate but also changes the color of the dyeing. In some cases the resulting change in shade can be seen as an opportunity to steer color in a wider range (Mussak and Bechtold 2009). Yellow or yellowish-brown tints are obtained on wool mordanted with alum and tartar using the essence of the juniper leaves. The tints possess a good fastness to washing and a good light- fastness as well using the essence of the leaves on wool mordanted with alum one obtains green (Lacasse and Baumann 2004). The bark, berries and twings are suitable for dyeing purposes on wool. Juniper berries give khaki color when applied with alum and copper sulfate. Juniper berries do not dye cotton (Furry and Morrison 1935).

Information about the mordants used in the study is given below;

Iron (ferrous sulphate)

Iron, one of the oldest mordants and easily available, is also known as green vitriol and is readily soluble in water. This mordant is used for darkening /browning and blackening of the colors /shades which is widely used to get grey to black shades.

Chrome (potassium dichromate)

It is also referred to as red chromate. However, Cr^{3+} or Cr^{6+} is considered to be detrimental to the human skin as objectionable heavy metal beyond a certain limit of its presence. Its use is limited as per the norms of the eco-standards. The dichromate solution is light sensitive, and thus, it leads to changes in the color under light exposure (Samanta and Konar 2011).

Sodium sulfate

It is known as an auxiliary chemical and increases an affinity between the wool and dyestuff in the dye bath. Although generally not known as a mordant in the literature, sodium ion is shown to be used as a mordant in some articles (Basalma et al. 2008; Cao et al. 2014; Li et al. 2015).

Tavares et al. (2012) putatively identified the catechins, procyanidins, flavonol derivatives, flavones and biflavones in the four wild Juniperus sp. (*Juniperus navicularis, J. oxycedrus badia, Juniperus phoenicea*, and *Juniperus turbinata*). As a result, all four species contained flavonol derivatives mainly quercetin. Keskes et al. (2014) investigated chemical composition of various extracts of *J. phoenicea* which are rich in flavonoids and polyphenols. Asili et al. (2008) determined that leaves of male and female plants of *J. communis subsp. hemisphaerica* and *J. oblonga* were both rich in tannins and flavonoides while the amounts of alkaloids and saponins were mainly not significant in these plants.

Considering related publications, major flavonols (colorants) in the leaves of Juniper may be quercetin, myricetin and their structural formula are given below (Figure 1).

Main phenolics have carbonyl (C = O) and hydroxy (—OH) groups that are more important and proper to form strong chemical bonds between mordant salt cation and protein molecules in fibers. The acquired dyeing and fastness properties of wool yarns are very important characteristics in terms of users. The interaction of mordant with wool fibers affects the affinity to fibers of dyestuffs (Eser and Onal 2015).

Özgür et al. (2013) determined that dyeing properties of quercetin, extracted from *Punicagranatum* L. rind, are used as different mordants on wool fabrics. The color coordinates of dyed samples were found to lie in the yellow-brown quadrant of the color space diagram. In a similar study, quercetin from acacia bark can also be seen reddish-brown synthetic dyes (Gulzar et al. 2015). Furthermore, Ali et al. (2007) determined that the major coloring component of Eucalyptus bark is quercetin and the color, obtained from dyestuff, is extracted from Eucalyptus and it is yellowish-brown. Also, Narayanaswamy et al. (2013) studied dyeing behavior of natural dye extracted from the leaves of *Psidiumguajuva* L and it is rich in terms of tannin that bears antiseptic properties and flavonoids are made up of quercetin, hyperin, and myricetin. The color coordinates of dyed samples lie in the yellow-red quadrant of color space diagram.

Taking previous studies into consideration about Junipers, we can observe that there is not enough study about extraction and natural dyeing. So, this study is concerned with the extraction of dye from Junipers leaves and its application on wool fiber using different mordants.

Material and methods

%100 pure straygarn woolen yarns (Yarn count: Nm 4/2, Twist per meter: 256 t/m) were supplied from Karatepe Kilim Cooperative in Osmaniye, Turkey. Wool, used to be dyed, obtained from sheep

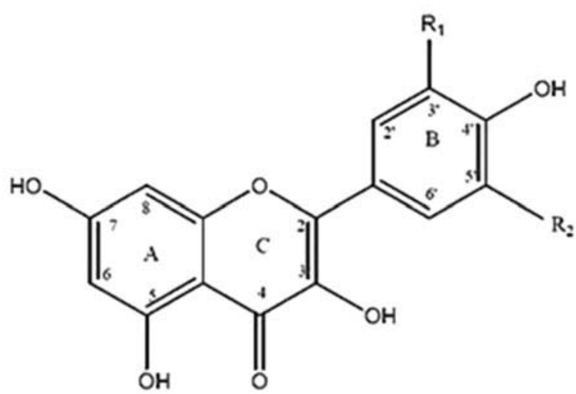

Figure 1. Chemical structure of flavonols (Quercetin R_1 = OH R_2 = H, Myricetin R_1 = OH R_2 = OH).

which is native to the region, Eastern Mediterranean. The hank samples (each 3 gram) were produced using hank winder for dyeing.

A number of Junipers leaves growing in the same conditions (*J. excelsa* and *J. oxycedrus*) were collected from Osmaniye Korkut Ata University without damaging the trees on dry and damaged boughs in July (Figure 2). They were first washed and sun-dried and then chopped. In order to prepare the original solution of the dye, each 20 g of plant samples was added to 1 L distilled water and boiled for 60 minutes and filtered (Figure 3). The concentration of the resultant solution is 2%.

Metallic Mordants can be classified as the Dulling mordants (copper and iron) and the Brightening mordants (Alum, Chrome and Tin) (Samanta and Konar 2011). The commercial ferrous (II) sulfate ($FeSO_4$) as the Dulling mordants, potassium dichromate ($K_2Cr_2O_7$) as the Brightening mordants and sodium sulfate (Na_2SO_4) used in natural dyeing processes were selected as the mordants for this study. The wetted wool yarns were mordanted using 3% owf of ferrous (II) sulfate, potassium dichromate and sodium sulfate. The liquor to goods ratio of the bath was kept at 50:1 for 45 min. at 80 °C. Commonly, mordanting is applied prior to dyeing. After mordanting, each sample was not rinsed but only squeezed.

150 ml of original dye solution (extract) was used for each a hank sample at liquor ratio 1:50. No chemical was used to maintain pH in dyeing process because the pH of the extracts was 5. Dyeing was started at 40 °C and the temperature was raised to 80 °C. Then, the samples remained for 60 min. The dyed wool yarn samples were washed for 30 min in a bath containing 2 g/L of a nonionic detergent (Setalan HE, Setas-Chem) at 80˚C. Finally, the samples were then rinsed and dried in shade at room temperature (Figure 4). The swatch cards were manufactured using yarn sample winding machine for color measurement (spectrophotometer). Dyed woolen yarn samples

Figure 2. *Juniperus oxycedrus, Juniperus excelsa*, and leaves (University of Osmaniye Korkut Ata Campus).

Figure 3. Natural plant extracts (*J. excelsa* and *J. oxycedrus*).

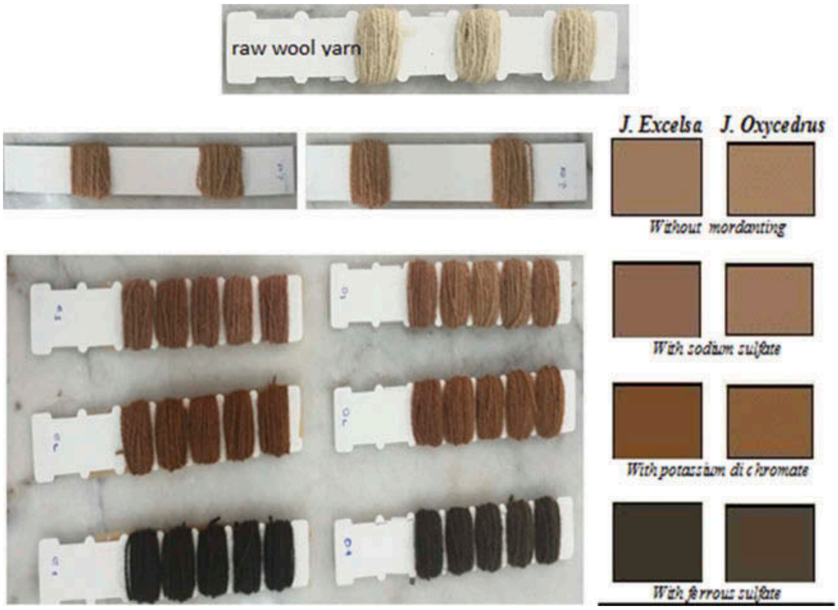

Figure 4. Pictures of dyed and raw woolen yarn samples and images on spectrophotometer.

(five replication) were carried out for three different mordants in the study. All mordanting and dyeing processes were fulfilled using a laboratory dyeing machine.

The color coordinates of dyed yarn were measured on a Minolta CM 3600 D model spectrophotometer coupled to a PC between 400–700 nm under D65/10° illuminant. K/S (Color Strength) values were obtained from Kubelka Munk equation;

$$K/S = (1 - R)^2/2R \quad (1)$$

where K is a constant associated with the light absorption of the fabric, predominantly determined by dyestuff, S is the constant associated with the light scattering of the fabric, determined only by the textile material, and R is the reflectance of the dyed fabric measured at the wavelength of maximum light absorption.

The CIELAB formula is used to assess small color differences and is recommended for use by DIN 6174. The color difference is determined using a color difference formula from the colorimetric measures L*, a*, b* which result from the CIE tristimulus values X, Y, Z. The L*-value indicates the position on the light/dark axis in the L*,a*, b* system, the a*-value gives the position on the red/green axis and the b*-value the position on the yellow/blue axis. According to DIN 6174, the ΔE*ab color difference is calculated as follows: (Needles 1986; Vigo 2005)

$$\Delta E^*_{ab} = \left[(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2\right]^{1/2} \quad (2)$$

Dry and wet rubbing fastnesses of the samples were tested after conditioning in standard atmospheric conditions (20°C±2 temperature and 65 ± 4 % RH) for 24 hours, according to ISO 105-X12 method.

Result and discussion

The colors, which were formed as a result of the dyeing performed with the mordant salts of $K_2C_r2O_7$ and $FeSO_4$, were observed to be brown and khaki green respectively. Moreover, it was

Table 1. Color measurement results of wool dyed with extracts of *Juniperus excelsa* leaves without and with mordanting.

	Color measurement results					
Dyeing	L*	a*	b*	c*	h	K/S
Dyeing without mordanting	54,52	10,06	21,01	23.29	64,41	8,36
Dyeing with premordanting (sodium sulfate)	46,91	12,84	18,58	22,59	55,36	10,18
Dyeing with premordanting (potassium dichromate)	37,59	14,87	27,77	31,5	61,84	20,47
Dyeing with premordanting (ferrous (II) sulfate)	23,29	1,51	7,57	7,72	78,7	25,05

Table 2. Color measurement results of wool dyed with extracts of *Juniperus oxycedrus* leaves without and with mordanting.

	Color measurement results					
Dyeing	L*	a*	b*	c*	h	K/S
Dyeing without mordanting	55,86	9,61	21,16	23,24	65,56	8,99
Dyeing with premordanting (sodium sulfate)	51,92	11,36	20,7	23,61	61,24	10,62
Dyeing with premordanting (potassium dichromate)	43,02	13,6	25,09	28,54	61,53	15,04
Dyeing with premordanting (ferrous (II) sulfate)	29,79	2,69	10,49	10,83	75,62	19,34

observed that the color was light brown as a result of the dyeing performed with the Na_2SO_4. Taking the quercetin studies into consideration, it is observed that the results of the study are similar to the literature (Ali et al. 2007; Gulzar et al. 2015; Narayanaswamy et al. 2013; Özgür et al. 2013).

CIELab color values and color strength (K/S) values of dyed yarns using extracts of *J. excelsa* leaves and *J. oxycedrus* leaves are listed Table 1 and Table 2. As can be seen from the Table 1, while yarns mordanted with ferrous sulfate have the highest K/S (color strength) and the lowest L* values (darker color appearance), dyeing yarns without mordanting have the lowest K/S and the highest L*values (Figure 5 and 6). Ferrous (II) sulfate can form complexes with the wool fiber on one site and with phenolic colorants of juniper leaves extract. High K/S values can be due to the ability of mordant metals to form coordination complexes between both the hydroxyl groups of the dye molecules and wool functional groups such as amino and carboxylic acid groups. Such strong coordination tendency can enhance the interaction between the fiber and the dye resulting in high dye uptake. This strong coordination tendency of Fe enhances the interaction between the fiber and the dye, resulting in high dye uptake, while all other metals show similar coordination (Eser and Onal 2015; Jothi 2008).

Low K/S values for sodium sulfate can be due to the decrease of solubility of juniper leaves extract coloring substances when this mordant were added in dye bath.

As can be seen from Figures 5 and 6, while yarns mordanted with ferrous sulfate have the highest K/S (color strength) and the lowest L* values (darker color appearance), dyeing yarns without mordanting have the lowest K/S and the highest L*values.

Change in total color difference (ΔE) value for two different extracts and three mordants are given in Table 3. The samples using extract of *J. oxycedrus* were taken as a reference for comparison. Table 3 indicates that there are significant color differences among extracts of two Junipers for all dyed samples with extract without and with mordanting. Dyeing samples with extract of *J. excelsa* are dyed darker and redder than that of *J. oxycedrus*.

Table 4 shows the rub fastness properties of mordanted and non-mordanted dyed wool. Rub fastness properties of dyed samples, when mordanted were higher than non-mordanted. This situation can be due to increase in size of dye molecules when connected the mordant molecules in to the fiber. Wet rub fastness values were less than dry rub fastness values because the water molecules can dissolve some of water-soluble dye molecules and make them easier to be removed from the fiber by rubbing (Haji 2010, 2012).

The darker samples mordanted with ferrous sulfate having lowest L* values have similar fastness properties with sodium sulfate and potassium dichromate.

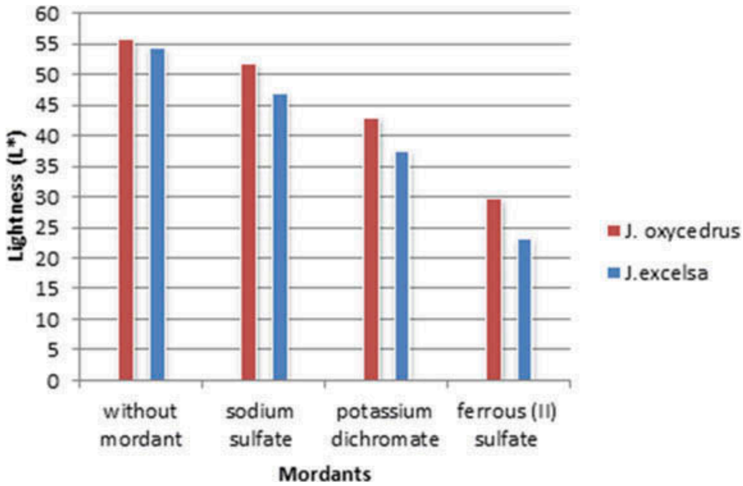

Figure 5. The effect of different mordants on lightness properties.

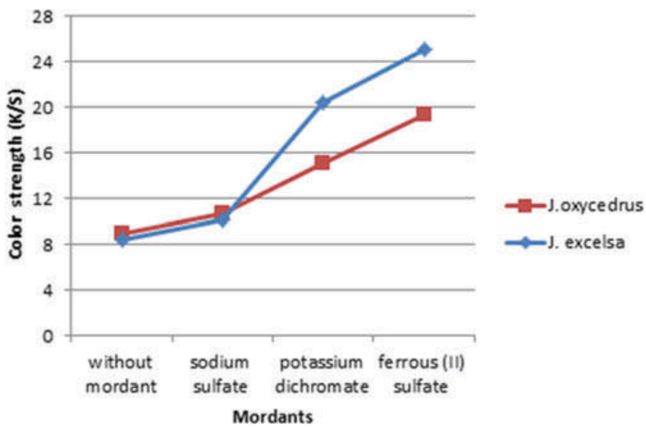

Figure 6. The effect of different mordants on color strength.

Table 3. Comparing of color difference measurement values.

Dyeing					
Juniperus oxycedrus*	Juniperus excelsa	ΔL	Δa	Δb	ΔE
without mordanting	without mordanting	−1,34	0,44	−0,14	**1,42**
sodium sulfate	sodium sulfate	−5,01	1,48	−2,11	**5,64**
potassium dichromate	potassium dichromate	−5,43	1,26	2,68	**6,19**
ferrous (II) sulfate	ferrous (II) sulfate	−6,50	−1,17	−2,92	7,23

* Reference for comparison

Table 4. Rub fastness properties of dyed samples.

	Juniperus oxycedrus		Juniperus excelsa	
Mordant	Rub Fastness (dry)	Rub Fastness (wet)	Rub Fastness (dry)	Rub Fastness (wet)
Sodium sulfate	4/5	4	4/5	4
Potassium dichromate	4/5	4	4/5	3–4
Ferrous (II) sulfate	4/5	3–4	4/5	3–4
Without Mordant	3/4	3	3/4	2–3

Conclusion

It can be concluded from the study that extract of Junipers leaves can be used as a natural dye for wool. Mordanting with ferrous (II) sulfate, potassium dichromate and sodium sulfate before dyeing increased the dye uptake and fastness properties of the samples. Furthermore, in this study, it is observed that the ferrous sulfate (metal salt) exhibited the highest K/S value, due to their ability to form coordination complexes with the dye molecules and mordanting with sodium sulfate before dyeing did not have an important impact on dyeing results compared to ferrous sulfate, potassium dichromate.

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