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Original article

A Research of Color and Fastness Values on Silk, Wool and Cotton Fabrics Dyed with Pulp of Oregano (*Origanum onites*) and Sage (*Salvia tomentosa*)

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Abstract

One of the most important problems of natural dyeing is the lack of raw material. In recent years, researchers are aimed to solve the deficiency of raw material. The pulp of plants, which are obtained from the steam distillation, is used as a dyestuff. We assume that the dyestuff in the pulp of oregano and sage after steam distillation remain stable. Wool yarns and silk fabrics have been dyed in previous works done as a master's and a Proficiency in Art thesis. The results were showed that our assumption is correct. Based on these results further researches were done. The textile materials were dyed with %100 of the pulp of oregano and sage. Mordant dyeing method was applied and three different mordant materials were applied with the pre-mordant process. Alum [Kal(SO4)2.12H2O], ferrous (II) sulphate [FeSO4.7H2O] and potassium dichromate [K2Cr2O7] were used as a mordant and potassium bitartrate [KC4H5O$_6$] as an auxiliary agent in alum mordanting. Rubbing (dry and wet) and washing fastness values were determined. Also, the depths of shade were evaluated in terms of K/S and CIELAB colour difference values of the dyed fabric samples.

According to the results obtained from the dyeing, the capability of pulp of plants is adequate and will supply the lack of dyestuff. While the colors obtained from silk and wool fabrics are more vivid and dark, the colors of cotton fabrics are quite pale. This study will allow other pulp of plants to be evaluated in this way.

Keywords: Origanum onites, Salvia tomentosa, Natural Dye, Fastness, Color Values..

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INTRODUCTION

The destiny of natural dyes, which are widely used in textiles and which have been found in almost every culture, has changed with the discovery of synthetic dyes in the 19th century (Saxena & Raja, 2014). Natural dyeing is important in many aspects of the cultural memory of societies. Turkish culture has a very great memory of natural dyes and dyeing techniques. They used the techniques of natural dyeing, which was about to fade because of migrations in the Middle Ages, and introduced them to the world (Dogan et al. 2003). The use of natural dyes in carpet and rug weaving products in traditional Turkish handicrafts has an important place. The dyeing of wool and cotton yarns on these weavings with natural dyes increases the quality and prolongs the life of the products. In addition, toxic and allergenic side effects are not observed in products dyed with the appropriate natural dye plants. The wastes of dyeing do not harm the environment (Bhuyan et al. 2016)

One of the most important problems of natural dyeing is the lack of raw material. Besides, color standardization and industrialization of dyeing techniques' problems are trying to be solved. In recent years, researchers tend to use pulp of plants to solve the deficiency of raw material. Despite these problems, recent researches on natural dyes give attention the use of them, after antimicrobial, antifungal (Shahid et al. 2012a,b), antifeedant (Kato et al. 2004) and UV protective properties discovered (Eser et al. 2017). In this study, the dyeing properties of the pulp after the removal of the essential oils from *Origanum onites* and *Salvia tomentosa* plants were investigated. Wool yarns and silk fabrics have been dyed and the results were showed that the dyestuff in pulp of oregano and sage after steam distillation remains stable. Based on these researches, color and fastness values were examined on silk, wool and cotton fabrics. Also, three different mordants were used and for comparison only one of the samples was dyed without any mordants.

The number of medical and aromatic plants is estimated that there were 1000 herbs are used for medical purposes (Baser, 2000). The use of these plants is not only limited to pharmaceuticals, food, beverages, cosmetics industry and veterinary medicine, but also organic farming, animal husbandry and bioaccumulators in phytochemistry (Bagdat, 2006). Sage and oregano species have an important place in these industries. At the same time, sage and oregano species have been used in natural dyeing (Dogan et al. 2003; Karadag, 2007; Bechtold and Mussak, 2009).

Steam distillation and solvent extraction are currently the most common methods used for the isolation of essential oils from natural products (Ozel and Kaymaz, 2004). These methods have advantages and disadvantages for many aspects, but superheated (or subcritical) water extraction (SWE) method is a new and powerful technique at temperatures between 100 and 374°C and pressure high enough to maintain the liquid state (Asl and Khajenoori, 2013). Essential oil from O. onites and S. tomentosa can be obtained by steam distillation method (Ozel and Kaymaz, 2004; Länger et al. 1996).

Members of Lamiaceae family, represented by 45 genera, are important in pharmacology and perfumery because of its essential and aromatic oil contents (Sokmen et al. 2005; Ipek and Gurbuz, 2010). Lamiaceae family includes many medicinal plant species which are well known and economically important such as sage, pepermint, lemon balm, lavander and sweet basil (Bagdat 2006). *Salvia* genus has 900 spices in the worldwide and 97 in natural for Turkey. In Turkey, 51 of *Salvia* genus are endemic. *S. tomentosa* Miller which were collected from Zorkun Plateau, Turkey, constituents were determined and β-pinene (37.28 %) and α-pinene (5.73 %) were the predominant chemical constituents, followed by trans-pinocarveol (3.05 %), myrtenol (2.81 %), caryphyllene oxide (2.68 %), D-camphor (2.08 %) (Ulukanli et al. 2013).

The systematics and taxonomy of the genus *Origanum* (Labiatae) were given in the "A Taxonomic Revision of the Genus *Origanum* (Labiatae)" by J.H. Ietswaart in 1980. A total of 46 of the 49 taxa have limited distributions within the Mediterranean basin with 21 representatives of the taxon endemic to Turkey (Bokov et al. 2015). Turkey has an important and dominant position in the worldwide trade of oregano with the high percentage of production (Kintzios, 2004). Origanum species usually inhabit mountain regions and rocky places with calcareous stone. Origanum species are generally rich in volatile oils containing considerable quantities of carvacrol and thymol. (Ietswaart, 1980). Turkish oregano typically accumulates essential oil, phenolic compounds (rosmarinic acid, acacetin), flavonoids (cinaroside, luteolin, thymusin, thymonin, cosmosiin, apigenin, etc.), tanning agents, phenolic glycosides, ascorbic acid, glycolipids, phospholipids, and steroids (sitosterol) (Bokov et al. 2015).

Genus of *Salvia* and *Origanum* have been studied for many aspects of EO and their properties and activities such as antimicrobial, antifungal, antioxidant, antibacterial and antitumoral etc. have been revealed by many researchers, but studies about dyeing properties or potentials are very limited. Dyeing properties, washing and rubbing fastness values of pulp of *O. onites* and *Salvia tomentosa* on wool pile yarn and silk fabric were examined (Teker, 2012, 2016). These studies showed the dyeing potential of pulp of these plants. In this study, we aimed to determine the color and fastness values of washing and rubbing (wet and dry) of pulp of *O. onites* and *S. tomentosa* on cotton, wool and silk fabrics.

MATERIALS and METHODS

Dried pulp of *Origanum onites* and *Salvia tomentosa* were obtained from İnan Tarım ECODAB in Antalya. The company produces EO of these plants by steam distillation. Plant leaves were separated from their stem parts and air-dried. Pure silk fabric weighing 64 g/m^2 (satin), wool (gabardine) weighing 245 g/m^2 and pure cotton (voile) weighing 72 g/m^2 (bought from Erol Kumaşçılık, Antalya) were used. The dyeing was carried out mordants, using pre-mordanting method. Alum [Kal(SO4)2.12H2O], ferrous (II) sulphate [FeSO4.7H2O] and potassium dichromate [$K_2Cr_2O_7$] mordants were used. Also potassium bitartrate was used as an auxiliary agent in alum mordanting. Dry and wet rubbing fastness values determined according to TS EN ISO 105-X12 and wash fastness values were determined according to

TS EN ISO 105-C06/A1S. According to Grey Scale, 1-2-3-4-5 are rating classes also half rating values can be used as 4-5 (1- very poor, 2-poor, 3-fair, 4-good and 5-excellent). Fastness test results are listed below according to dyestuff in Table 3 and Table 4. Color strength and colorimetric values were measured by 3nh NS800 (D65, specular inclusion, 10°) spectrophotometer (Table 1, Table 2).

Pre-mordanting method was applied. %20 alum and % 15 potassium bitartrate were used together, %0,5 ferrous (II) sulfate for medium darkness and %1 potassium dichromate were used. Mordanting and dyeing temperature was 80°C with 1:100 flotte ratio for cotton fabrics and 60°C with 1:100 flotte ratio for silk and wool fabrics. The dyebath was held constant for 15 minutes when the dye bath temperature was reached at 80 / 60°C. Then left to decrease to room temperature (~20°C) and then left in the dye bath for 24 hours. After 24 hours fabrics were rinsed with tap water, squeezed and dried. Mordanting and dyeing processes are shown in Fig.1-4.

Pulp of plants were weighted according to fabric weight. % 100 of pulp were used. Pulps were put in tap water for 24 hours in separated pots and then boiled for half an hour. Flotte ratio was kept at 1:100. After filtration, it was ready for the dyeing process.

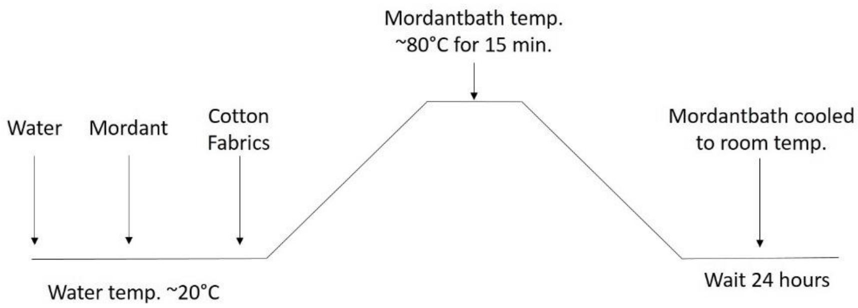

Figure 1. Mordanting diagram of cotton fabrics

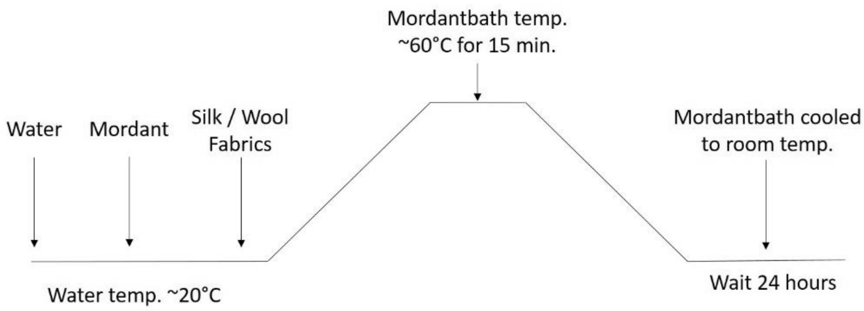

Figure 2. Mordanting diagram of silk and wool fabrics

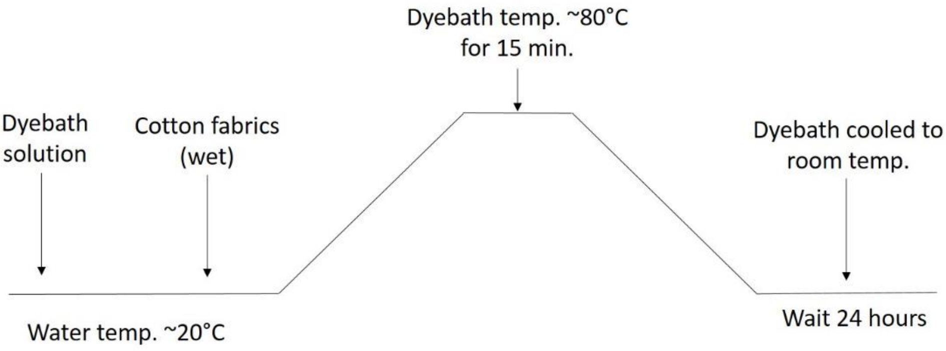

Figure 3. Dyeing diagram of cotton fabrics

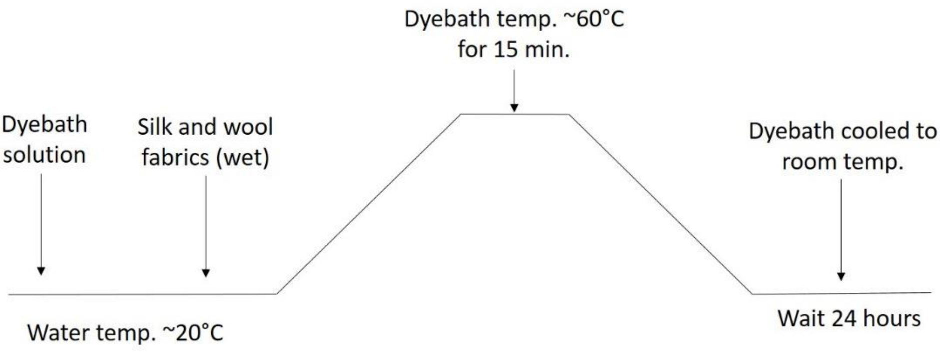

Figure 4. Dyeing diagram of silk and wool fabrics

Tests for color fastness to washing and rubbing were applied. Dry and wet rubbing fastness tests were applied by manual crockmeter. Color fastness values were evaluated according to ISO 105-X12:2016. The amount of color that stains the reference cloth is evaluated against "grey scale" (grade 1 to 5). Dyed fabrics were washed with detergent without optical brightener (WOB) at 40°C and washed with pure water at 37°C, then color fastness values were evaluated according to TS EN ISO 105-C06:2010.

RESULTS and DISCUSSION

Color Evaluation

In this study, three different mordanting agents, an auxiliary substance and pulp of *O. onites* and *S. tomentosa* were used. These mordanting agents were selected because of their acceptance according to ecotextile (Horrocks, 1999; Shahid et al., 2012b; Yusuf, Shabbir, & Mohammad, 2017; Burkinshaw, & Kumar, 2009).

The colorimetric measurements of fabrics were performed using a 3nh NS800 (Test Condition: Illuminant: D65 Observer, Angle: 10°, Color Space: CIE LAB, LCh, Color Formula: CIE 1976) spectrophotometer. The values used by CIE are called L*, a* and b* and the colour measurement method is called CIELAB. L* represents the difference between light (where L*=100) and dark (where L*=0). a* represents the difference between green (-a*) and red (+a*), and b* represents the difference between yellow (+b*) and blue (-b*) (Seif and Hijji, 2019). The colour difference ΔE was calculated by the Equation (1):

$\Delta E=\sqrt{(\Delta L)2 +(\Delta a)2+(\Delta b)2}$ (1)

The K/S (color strength) value was calculated by Equation (2):

K/S= (1-R)2 / 2R (2)

where R is the observed reflectance. The colorimetric values for dyed fabrics are listed in Table 1 and Table 2.

Table 1. Colorimetric values of samples dyed with *Origanum onites*

No	Fabric	Mordant	L*	a*	b*	C*	h°	ΔE	K/S
1	Silk (Satin)	Undyed	94,514	0,972	2,406	2,595	68,011	-	-
2		Unmordanted	72.604	5.605	19.721	20.502	74.134	28.307	3,43
3		Alum-potassium bitartrate	71.355	6.115	24.287	25.045	75.868	32.273	4.45
4		Ferrous (II) sulphate	52.079	2.228	15.202	15.364	81.661	44.340	1.39
5		Potassium dichromate	74.931	4.277	21.151	21.579	78.568	27.309	3.00
6	Wool (Gabardine)	Undyed	90,502	2,524	11,859	12,125	77,984	-	-
7		Unmordanted	77.997	3.751	17.928	18.316	78.181	13.954	5.14
8		Alum-potassium bitartrate	74.788	3.485	24.509	24.755	81.906	20.196	8.49
9		Ferrous (II) sulphate	62.932	1.931	12.348	12.498	81.113	27.581	1,60
10		Potassium dichromate	76.760	1.226	26.051	26.079	87.306	19.797	7,72
11	Cotton (Voile)	Undyed	94,871	1,559	-2,348	2,818	303,575	-	-
12		Unmordanted	72.778	3.520	13.680	14.126	75.569	27.366	2.13
13		Alum-potassium bitartrate	71.077	3.049	16.474	16.753	79.515	30.375	2.64
14		Ferrous (II) sulphate	70.453	3.986	15.192	15.706	75.297	30.162	2.67
15		Potassium dichromate	73.618	3.120	14.290	14.627	77.686	27.037	2.03

According to (Table 1) analysis of the data shows that using ferrous (II) sulphate mordant gives darker shades. On all fabric types, ferrous (II) sulphate mordant causes the darkest shades, silk fabric is the darkest samples. The silk fabrics showed more reddish shades. This can be attributed slightly the structure of silk and fabric surface brightness. Generally, all fabric samples have clouse lightness values with alum and potassium dichromate mordants. When b* values are examined, fabric samples mordanted with alum+potassium bitartrate show more yellowish shades than unmordanted, ferrous (II) sulphate and potassium dichromate mordanted fabric samples, only one fabric sample which is gabardine mordanted with potassium dichromate has more yellowish shade. Cotton fabrics are duller especially the unmordanted dyed cotton fabric sample. It was observed that, the color strength (K/S) value was good at alum+potassium bitartrate mordant. The value h° varies between 68-87, this indicates that samples have a yellow tone.

Table 2. Colorimetric values of samples dyed with *Salvia tomentosa*

No	Fabric	Mordant	L*	a*	b*	C*	h°	ΔE	K/S
1	Silk (Satin)	Undyed	94,514	0,972	2,406	2,595	68,011	-	-
2		Unmordanted	74.724	3.356	22.311	22.562	81.445	28.170	3.27
3		Alum-potassium bitartrate	71.699	5.669	26.910	27.500	78.104	33.808	4.75
4		Ferrous (II) sulphate	55.023	3.213	19.310	19.575	80.554	43.015	1.25
5		Potassium dichromate	73.725	3.401	24.003	24.243	81.934	30.075	3.73
6	Wool (Gabardine)	Undyed	90,502	2,524	11,859	12,125	77,984	-	-
7		Unmordanted	79.912	2.899	19.899	20.109	81.712	13.302	0.46
8		Alum-potassium bitartrate	74.516	4.055	26.536	26.844	81.311	21.756	0.93
9		Ferrous (II) sulphate	63.009	2.793	15.952	16.195	80.070	27.798	1.71
10		Potassium dichromate	75.014	2.371	30.445	30.538	85.547	24.194	1.00
11	Cotton (Voile)	Undyed	94,871	1,559	-2,348	2,818	303,575	-	-
12		Unmordanted	78.817	1.282	19.599	19.641	86.257	27.193	1.48
13		Alum-potassium bitartrate	76.494	1.353	23.148	23.187	86.655	31.430	2.06
14		Ferrous (II) sulphate	75.081	2.698	18.575	18.770	81.736	28.822	2.00
15		Potassium dichromate	78.760	1.534	18.761	18.824	85.325	26.555	1.45

According to (Table 2) analysis of the data shows that using ferrous (II) sulphate mordant gives darker shades as Table 1. On all fabric types, ferrous (II) sulphate mordant causes the darkest shades, silk fabric is the darkest samples dyed with both pulp of plants. a* and b* values are more close to each other than origanum onites dyeing. The silk fabrics showed more reddish shades, too. Generally, all fabric samples have close lightness values with alum and potassium dichromate mordants. When b* values are examined, fabric samples mordanted with alum+potassium bitartrate show more yellowish shades then unmordanted, ferrous (II) sulphate and potassium dichromate mordanted fabric samples, only one fabric sample which is gabardine mordanted with potassium dichromate has more yellowish shade, too. It was observed that, the color strength (K/S) values are olwer than *origanum* onites. The value h° varies between 68-86, this indicates that samples have a yellow tone. Generally, we can say that while the color tones in the fabrics dyed with salvia tomentosa are closer to each other, the color effects of mordants in origanum onites are more evident.

Washing and Rubbing (Dry/Wet) Fastness Values

The results of the tests performed on the manual crockmeter are given in the Table 3 and Table 4 classified accordint to dyestuff. Washing fastness tests are done with detergent without optical brightener (WOB) at 40°C and with pure water at 37°C. Generally, alum+potassium bitartrate mordant gives higher washing and rubbing fastness values. Potassium dichromate mordants come next. Washing and rubbing fastness values of ferrous (II) sulphate mordants are 4 to 4-5. According to test results, minimum value of wet rubbing fastness value was obtained from origanum onites with unmordanted wool fabric. The maximum values of dry rubbing fastness valus were obtained from alum+potassium bitartrate and potassium dichromate mordants for all types of fabric samples. The lowest change in color value is seen in cotton fabric with ferrous (II) sulphate mordant.

Table 3. Fastness values of samples dyed with *Origanum onites*

Fabric	Mordant	Washing Fastness							Rubbing Fastnes	
		Staining on wool	Staining on acrylic	Staining on polyester	Staining on polyamide	Staining on cotton	Staining on acetate	Change in color	Dry	Wet
Silk (satin)	Unmordanted	4	4-5	4-5	4-5	4-5	4-5	4	4-5	4
	Alum-potassium bitartrate	4-5	5	5	5	4-5	5	4-5	5	4-5
	Ferrous (II) sulphate	4	4-5	4-5	4-5	4-5	4-5	4	4-5	4-5
	Potassium dichromate	4-5	5	5	5	5	5	4-5	4-5	4-5
Wool (gabardine)	Unmordanted	4	4	4-5	4-5	4-5	4	4	4	3-4
	Alum-potassium bitartrate	4-5	5	5	5	5	4-5	4-5	5	4-5
	Ferrous (II) sulphate	4-5	4-5	4-5	4-5	4-5	4-5	4	4-5	4
	Potassium dichromate	5	5	5	5	5	5	4-5	5	4-5
Cotton (voile)	Unmordanted	4-5	4-5	5	5	5	5	4-5	5	5
	Alum-potassium bitartrate	5	5	5	5	5	5	4-5	5	4-5
	Ferrous (II) sulphate	4	4	4	4	4	4	3-4	4	4
	Potassium dichromate	4-5	4-5	4-5	4-5	4-5	4-5	4	4-5	4-5

Table 4. Fastness values of samples dyed with *Salvia tomentosa*

Fabric	Mordant	Washing Fastness							Rubbing Fastnes	
		Staining on wool	Staining on acrylic	Staining on polyester	Staining on polyamide	Staining on cotton	Staining on acetate	Change in color	Dry	Wet
Silk (satin)	Unmordanted	4	4-5	4-5	4-5	4-5	4-5	4-5	4	4
	Alum-potassium bitartrate	5	5	5	5	5	5	4-5	5	4-5
	Ferrous (II) sulphate	4-5	4-5	4-5	4-5	4-5	4-5	4	5	4-5
	Potassium dichromate	5	5	5	5	5	5	4-5	5	4-5
Wool (gabardine)	Unmordanted	4	4-5	4-5	4-5	4-5	4-5	4	4-5	4
	Alum-potassium bitartrate	5	5	5	5	5	4-5	4-5	5	4-5
	Ferrous (II) sulphate	4	4-5	4-5	4-5	4-5	4-5	4	4	4
	Potassium dichromate	4-5	5	5	5	5	5	4-5	4-5	4-5
Cotton (voile)	Unmordanted	4-5	4-5	5	5	5	5	4-5	4-5	4-5
	Alum-potassium bitartrate	4-5	5	5	5	5	5	4-5	5	4-5
	Ferrous (II) sulphate	4-5	4	4	4	4	4	4	4-5	4
	Potassium dichromate	5	5	5	5	5	4-5	4-5	4-5	4-5

Conclusion

Production of *Origanum* and *Salvia* genus in Turkey was made easy to access to pulp of these plants. Pulp of these plants can be obtained more economically after distillation of EO which has an important place in medical and aromatic plants.

In this study, silk, wool and cotton fabrics were mordanted with alum+potassium bitartrate, ferrous (II) sulphate and potassium dicromate dyed with pulp of O. onites and *S. tomentosa*. Test results show that silk and wool fabric color tones and fastness values are better than cotton fabrics. The use of naturally dyed textile products can be increased by solving one of the most important problems which the lack of dyestuff. The other advantage of using pulp of plants is decreasing dyestuff cost. In line with the results revealed by the antibacterial and antifungal properties of oregano and sage plant species, the scope of the study can be expanded by investigating whether there are similar effects on fabrics dyed with these plants.

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