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NATURAL DYEING OF SILK, WOOL AND COTTON FABRICS' COLOR AND FASTNESS PROPERTIES DYED WITH *DAPHNE SERICEA* VAHL COLLECTED FROM ANTALYA

Abstract

In this study, the dyeing properties of *Daphne sericea* Vahl plant with different mordants and washing, rubbing (dry and wet) and light fastness values were investigated. This plant is widely grown in Antalya region and can be considered as a natural dye source. *Daphne* (Thymelaeaceae) species have been widely used in traditional Anatolian medicine and locally called “ezentere” in Antalya. The dye capability of this plant has been studied on cotton, wool and silk fabrics with alum [$KAl(SO_4)_2 \cdot 12H_2O$], ferrous sulphate [$FeSO_4 \cdot 7H_2O$] and potassium dichromate [$K_2Cr_2O_7$] mordants. In order to see the effect of mordants, fabrics were dyed without any mordants. The CIE $L^*a^*b^*$ and K/S values of the fabrics were measured and are discussed.

Keywords: *Daphne sericea* Vahl, natural dye, fastness, color measurement

ANTALYA'DAN TOPLANAN *DAPHNE SERICEA* VAHL BİTKİSİ İLE BOYANMIŞ İPEK, YÜN VE PAMUKLU KUMAŞLARIN RENK VE HASLIK ÖZELLİKLERİ

Öz

Bu çalışmada *Daphne sericea* Vahl bitkisinin farklı mordanlarla yapılmış boyama özellikleri ve yıkama, sürtünme (kuru ve ıslak) ve ışık haslığı değerleri araştırılmıştır. Antalya bölgesinde bu bitki yaygın olarak yetişmekte ve doğal boya kaynağı olarak değerlendirilmeye imkan sağlamaktadır. *Daphne* (Thymelaeaceae) türleri geleneksel Anadolu tıbbında yaygın olarak kullanılmaktadır ve Antalya'da yerel olarak “ezentere” olarak adlandırılmaktadır. Bu bitkinin boyama kabiliyeti, şap $[KAl(SO_4)_2 \cdot 12H_2O]$, demir sülfat $[FeSO_4 \cdot 7H_2O]$ ve potasyum dikromat $[K_2Cr_2O_7]$ ile mordanlanmış pamuk, yün ve ipekli kumaşlar üzerinde incelenmiştir. Mordanların etkisinin görülebilmesi için mordansız boyamada yapılmıştır. CIE L * a * b * renk değerleri ve K / S ölçümleri yapılmış ve değerlendirilmiştir.

Anahtar kelimeler: *Daphne sericea* Vahl, doğal boya, haslık, renk ölçümü

1. INTRODUCTION

Natural dyes have been used in textiles for thousands of years in many different areas; textile, painting, food, cosmetics, etc... They have lost their importance after the discovery of synthetic dyes in textile industry. However, after upon emergence the negative effects of synthetic dyes on environmental pollution (wastewater) and human health, natural dyes became important again. With the world be-coming more conscious towards ecology and environment, there is emerging a greater need to revive the tradition of natural dyes and dyeing techniques (Sanjeeda and Taiyaba, 2014). At this point, it is aimed not only to reuse natural dyes but also to gain importance with correct and sustainable studies. Issues such as the high cost of using natural dyes, standardization or cultivation are still a problem, but these can be ignored when it comes to issues such as the environment, health and natural life.

Natural dyeing is important in many aspects of the cultural memory of societies. Turkish culture has a very great memory on natural dyes and dyeing techniques. They used the techniques of natural dying, which was about to fade because of migrations in the Middle Ages, and introduced them to the world (Doğan et al., 2003).

Dyestuffs are classified into three main groups; plants, Plants, animals and minerals. Plants are the main source of natural dyestuff sources (Shahid and Mohammad, 2013). It is possible to mention hundreds of plants used in natural dyeing in Turkey (Doğan et al., 2003; Karadağ, 2007; Özgökçe and Yılmaz, 2003; Parlak, 2007). The Mediterranean region is also included in this rich resource (Türkmen et al. 2004). Different properties can be obtained by natural dyes, such as antibacterial (Ren et al., 2017), antifungal (Shahid et al., 2012a; Shahid et al., 2012b), antifeedant (Kato, 2004), antioxidant (Amelia, 2013).

Including annual species, which spread in the world's tropical and temperate regions, one of the members of the Thymelaeaceae family, belonging to the genus *Daphne*, 7 species are known to grow in Turkey Flora (Borris et al., 2013). Tosun (2006) gave brief information about chemical

constituents and biological activities of daphne L. species on her article. The aerial parts of *Daphne sericea* yielded two new flavonoids, luteolin 7-methyl ether 5- β -d-glucoside and luteolin 7,3'-dimethyl ether 5- β -d-glucoside, as well as luteolin 7-methyl ether, isovitexin, apigenin and its 7- β -d-glucoside the following (Ulubelen, 1982).

Genus of *Daphne* has been studied for many aspects of their properties and contents, but studies about dyeing properties or potentials are not researched. The aim of the present work was to investigate the dyeing capability and fastness values of washing, rubbing (dry and wet) and light, determine the CIE L*a*b* and K/S values.

2. MATERIAL AND METHOD

2.1 Material

Pure silk fabric with different woven structure weighing 64 g/m² (satin) and 80 g/m² (mongol), wool (gabardine) weighing 245 g/m² and pure cotton (voile) weighing 72 g/m² (bought from Erol Kumaşçılık, Antalya) were used.

Daphne sericea Vahl. plant were collected from Ağırtaş, Döşemealtı, Antalya which coordinates are N 37°13'0,091" E 30°40'13,66" (Point A) and N 37°12'57,754" E 30°40'11,883" (Point B) showed in Figure 1. Plants dried in the shade and in an open airy place. Above ground part of the plant was used in dyeing process. Alum [KAl(SO₄)₂.12H₂O] and ferrous sulphate heptahydrate [FeSO₄•7H₂O] from Merck and potassium dichromate [K₂Cr₂O₇] from Zag were used. Also potassium bitartrate [KC₄H₅O₆] was used as an auxiliary substance in alum mordanting. Pre-mordanting method was applied.

Figure 1. Map of study area

2.2 Method

For dyeing process, plants were used as a 100% proportion of the quantity of the textile. Dried plants were put in a kettle with the ratio of 1:40. Boiled for an hour and let in the kettle for 24 hours then filtered. For each fabric samples four dyeings were done. First cotton fabric sample dyed without any mordant. Second sample was mordanted with 15% of alum and %5 of potassium bitartrate as a proportion of the quantity of the textile. Alum and potassium bitartrate were dissolved in warm (60°C) water and then added into the mordanting kettle. Then, the wetted fabric sample was added into the mordant solution. This was performed at 70 °C for 15 min. in a liquor ratio of 1:30. Then fabric leaved in the mordant bath for 24 hours at room temperature.

Third sample was mordanted with 0.5 % of ferrous sulphate in the same way. The fourth sample was mordanted with 5% of potassium dichromate in the same way. For silk fabric samples (satin and mongol), the temperature was set to 55°C and time for 30 min. provided the proportions remained the same for mordanting of other silk fabrics. For wool fabric sample (gabardine), the temperature was set to 70 °C and time for 30 min. provided the proportions remained the same for mordanting of other wool fabrics. After mordanting process fabrics were rinsed with cold water and left to dry.

For cotton fabrics dyebath temperature was increased to 80°C and let to decrease 40 °C and increased to 80°C again and leave in the dyebath for 24 hours. For silk and wool fabrics dyebath temperature was increased to 65°C and let to decrease 40 °C and increased to 65°C again and leave in the dyebath for 24 hours. After dyeing process fabrics were rinsed with cold water and left to dry.

3. RESULTS AND DISCUSSION

3.1 Color Measurements

The colorimetric measurements of fabrics were performed using a 3nh NS800 (D65, specular inclusion, 10°) spectro-photometer. 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):

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

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

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

where R is the observed reflectance.

The colorimetric values for dyed fabrics are listed in Table 1. Analysis of the data shows that using ferrous sulphate heptahydrate mordant gives darker shades. On all fabric types, ferrous mordant causes the darkest shades, but cotton fabric samples are generally lighter than the other fabric samples. The gabardine fabrics showed more reddish shades. This can be attributed slightly the structure of wool and fabric weaves density. Besides the gabardine fabric, mongol fabrics also showed more reddish shades. Also results showed that unmordanted dyed fabrics have more reddish shades. When b* values are examined, fabric samples mordanted with alum and potassium dichromate show more yellowish shades than unmordanted and ferrous mordanted fabric samples, and also b* values are close to each other. The values of a* were lowest and b* was maximal for silk fabric samples mordanted with alum. Gabardine fabrics have brighter shades than other fabrics. Cotton fabrics are duller especially the unmordanted dyed cotton fabric sample. It was observed that, the color strength (K/S) value was good at gabardine fabrics especially with potassium dichromate mordant. The value h° varies between 70-93, this indicates that samples have a yellow tone.

Table 1. Colorimetric values of samples

No.	Fabric	Mordant	L*	a*	b*	C*	h°	ΔE	K/S
1	Silk (Satin)	Unmordanted	83.274	2.509	20.690	20.841	83.085	-	-

2		Alum and potassium bitartrate	79.713	0.734	47.422	47.427	89.113	27.026	3,75
3		Ferrous sulphate heptahydrate	69.525	2.363	20.187	20.325	83.322	13.758	3,25
4		Potassium dichromate	78.817	1.659	43.858	43.890	87.834	23.608	3,37
5	Silk (Mon-gol)	Unmordanted	80.602	3.832	22.045	22.375	80.140	-	-
6		Alum and potassium bitartrate	78.932	1.995	44.487	44.531	87.433	22.579	2,67
7		Ferrous sulphate heptahydrate	67.958	2.992	20.022	20.244	81.502	12.832	2,71
8		Potassium dichromate	76.872	2.850	41.852	41.949	86.104	20.179	2,73
9	Cotton (Voile)	Unmordanted	91.175	0.763	9.165	9.197	85.239	-	-
10		Alum and potassium bitartrate	88.159	1.524	25.449	25.494	93.428	16.718	3,20
11		Ferrous sulphate heptahydrate	72.620	10.807	30.214	32.089	70.319	29.803	14,22
12		Potassium dichromate	87.753	-1.282	26.844	26.875	92.735	18.123	3,39
13	Wool (Gardine)	Unmordanted	76.553	5.951	24.984	25.683	76.601	-	-
14		Alum and potassium bitartrate	74.719	5.559	41.227	41.600	82.320	16.351	1,88
15		Ferrous sulphate heptahydrate	53.521	5.533	22.839	23.500	76.383	23.136	5,28
16		Potassium dichromate	63.684	3.289	48.592	48.703	86.128	27.019	6,68

3.2 Fastness Tests

Light, washing and rubbing fastness results are listed and classified according to mordants in Table 1-4. Tests for color fastness to washing, rubbing (dry and wet) and light were done in the Headship of TSE Test and Calibration Centre Aegean Regional Laboratories. Light fastness values were evaluated according to TS EN ISO 105-B02, rubbing fastness values (dry and wet) according to TS EN ISO 105-X12 and washing fastness values according to TS EN ISO 105-C06/A1S.

Rubbing fabric and samples were conditioned under standard atmosphere conditions for 24 hours and then 16 mm cylindrical rubbing foot used for rubbing fastness test. In wet rubbing, cotton rubbing cloth was used after absorbing 100% water. Environmental conditions of the experiment were 20±2°C and 65% relative humidity. According to Grey Scale, 1-2-3-4-5 are rating classes also half rating values can be used like 3-4 or 4-5 (1- very poor, 2-poor, 3-fair, 4-good and 5-excellent). Fastness test results are listed below according to mordant agent in Table 2,3,4,5.

Table 2. Fastness values of unmordanted dyed fabrics

Fabric	Light Fastness	Washing fastness							Rubbing fastness	
		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)	4-5	5	5	5	5	4-5	5	4-5	4-5	4-5
									4-5	4-5
Silk (mongol)	4	5	5	5	5	4-5	5	4-5	4-5	4-5
									4-5	4-5
Cotton (voile)	4	5	5	5	5	5	5	3-4	5	4-5
									5	4-5
Wool (gabardine)	4	5	5	5	5	5	5	4-5	5	4-5
									5	4-5

Table 3. Fastness values of fabrics mordanted with alum and potassium bitartrate

Fabric	Light Fastness	Washing fastness							Rubbing fastness	
		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)	4	5	5	5	5	4-5	5	3-4	5	5
									5	5
Silk (mongol)	4	5	5	5	5	4-5	5	4	5	5
									5	5
Cotton (voile)	4	5	5	5	5	4-5	5	4	5	5
									5	5
Wool (gabardine)	4	5	5	5	5	5	5	4	5	5
									5	5

Table 4. Fastness values of fabrics mordanted with ferrous sulphate heptahydrate

Fabric	Light Fastness	Washing fastness							Rubbing fastness	
		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)	4-5	5	5	5	5	5	5	4-5	4-5	4-5
									4-5	4-5
Silk (mongol)	4-5	5	5	5	5	5	5	4-5	4-5	4-5
									4-5	4-5
Cotton	4-5	5	5	5	5	5	5	4	5	4-5

(voile)									5	4-5
Wool	4-5	5	5	5	5	5	5	4-5	3-4	3-4
(gabardine)									3-4	3-4

Table 5. Fastness values of fabrics mordanted with potassium dichromate

Fabric	Light Fastness	Washing fastness							Rubbing fastness	
		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)	4-5	5	5	5	5	5	5	4-5	4-5	4-5
									4-5	4-5
Silk (mongol)	4	5	5	5	5	5	5	4-5	4-5	4-5
									4-5	4-5
Cotton (voile)	4	5	5	5	5	5	5	4-5	5	4-5
									5	4-5
Wool (gabardine)	4-5	5	5	5	5	5	5	4-5	4-5	4
									4-5	4

The light fastness values were ranged from 4 to 4-5 for all mordant varieties. Especially ferrous sulphate heptahydrate mordant gives the highest values. The washing fastness properties of the dyed samples varied between 4-5, staining on test fabrics are generally 5, except for staining on cotton mordanted with alum and ferrous sulphate heptahydrate. The rubbing fastness of the dyed samples varied generally between 4 and 5, wet and dry rubbing fastness values for unmordanted dyed fabrics are excellent.

4. CONCLUSION

Daphne sericea plant was selected for dyestuff because of its dyeability potential and lack of knowledge about this plant. During the field research, the villagers stated that they used this plant in dyeing. In particular, they used to obtain yellow colors, but also used as a mordant substance. However, they did not provide information about dyeing recipe. This species, which is locally named as “ezentere” in Antalya, has been searched for literature after species identification. Although there are many publications on *daphne* sp., no studies have been found on dyeing properties or color measurements. Three different mordant substances and four different fabric types are dyed (silk satin, silk mongol, cotton and wool). As a result of the dyeings made with this plant which was evaluated within the scope of the project, the results of washing, light and rubbing (wet and dry) fastnesses were found to be quite high. Especially the color results obtained from silk and wool fabrics are quite good. Dyeing on cotton fabrics, especially the color obtained as a result of unmordant dyeing is very pale. Fastness values and color measurements indicate that this plant is an important source of dyes. It is thought that it can offer an important alternative to plants such as *reseda luteola* L. and *rhannus petiolaris* Boiss which are widely used to obtain yellow color.

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