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Effect of Mordants on Color Shade and Color Fastness of Silk Dyed with Kikar and Madder Barks

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The silk fabric was dyed with aqueous extract of Kikar bark *(Acacia arabica)* and Madder bark *(Rubia cordifolia)* by using various metal sulphates as mordants. The three techniques of mordanting were used for dyeing the fabric and are known as pre-mordanting, where the fabrics were first mordanted and then dyed with natural dye extract, meta-mordanting (i.e. dyeing in the presence of mordants) and post- mordanting where the dyed fabrics were treated with mordants. The fastness properties i.e. crock fastness, washing fastness and light fastness of the dyed samples were determined and comparison was made for control and samples dyed in the presence of the metal ions. The three fastness of the dyed samples were found to be good to excellent. The color of the dyed silk was investigated on computer color matching system in terms of Reflectance, K/S and CIE lab color values. The effect of different metal ions have been studied with respect to their influence on color shade and fastness properties. The mechanism of mordant interactions with the fabric has been briefly discussed.

Key words: Natural dyes, Mordants, Fastness.

Introduction

Natural dyes are generally understood to be colorants (dyes and pigments) that are obtained from animal or vegetable matter without chemical processing. They are mainly mordant dyes, although some natural vat, solvent, pigment, direct and acid types are known (Gulrajani and Gupta 1992a). In recent years concern for the environment has created an increasing interest in natural dyes. Conventional wisdom leads to the belief that natural dyes are amiable to the environment than their synthetic counterparts, although the issue is not necessarily quite so straight forward (Smith and Wagner 1995). Nevertheless, natural dyes do have tremendous commercial potential (Verma and Gupta 1994).

In recent years, the world has become increasingly aware of environmental issues. Synthetic dyestuffs in particular have come under severe criticism on the grounds of being highly polluting in their manufacturing and application. A search for safer alternatives has created a widespread renewal of interest in natural dyes. Studies conducted on the color characteristics and fastness properties of natural dyes show that the colors obtained are soft and varied and several shades have wash fastness ratings similar to those of acid dyes on wool and silk (Minagawa and Kawahara 1983; Grierson *et al* 1985; Taylor 1986; Gulrajani and Gupta 1992b).

There have been some investigations on the theoretical basis of dyeing with natural dyes (Arshad *et al* 1954; Gupta and Gulrajani 1994; Gulrajani *et al* 1999). Recently there has been growing interest in the use of natural dyes in textile applications. This is a result of the stringent environmental standards improved by many centuries in a response to the toxic and allergic reactions associated with synthetic dyes. Natural dyes exhibit better biodegradability and are generally more compatible with the environment. In spite of their inferior fastness, natural dyes are more acceptable to environmentally conscious people around the world (Deo and Desai 1999).

The present study focuses on the dyeing of silk with the Kikar and Madder bark extract which is sparingly soluble in water but is freely soluble in alcohol. For dyeing three different techniques named as pre-mordanting, meta-mordanting and post-mordanting were used. The fastness properties of silk samples dyed with natural dye with and without mordants were determined. Methods are intended for determining the resistance of the color of silk when exposed to sunlight (light fastness), washed with soap water at the given temperature (wash fastness) and rubbing off and staining other materials (crock fastness). Grey scale of society of dyers and colorist (SDC) is used as the standard rating scale (1 to 5) to determine the change in color of these fastness properties. The rating 1-2 shows poor fastness, 3-4 moderate and 4-5 good and very good fastness properties.

The changes in color properties will be discussed in terms of reflectance , K/S and CIE Lab values of the dyed substrate. The reflectance is actually the ratio of the light leaving and an

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operat versus the total light that was hitting the object and K/S is the determination of colorant strength from reflectance measurement. C1E Lab Cartesian coordinates are L*, a* and b*, where L* values represent the difference of lightness of the color, the a* value corresponds to the difference of color's position on the red-green axis and the b* value is the difference of its position on the yellow-blue axis.

Materials and Methods

A commercial sample of Kikar and Madder Bark was purchased from Akbari Mandi, Lahore. Commercially bleached silk (all 3% o.w.f) cut into rectangular pieces of 10 cm x 12 cm, weighing 2.0 and 2.5 g, respectively were used in dyeing. The fabric was washed using non-ionic detergent to remove any impurities present. Aqueous solutions containing 5 and 10g/l ferrous sulphate hepta hydrate, aluminium potassium sulphate dodecahydrate (alum) and copper sulphate pentahydrate were used as mordants. Dried barks of Kikar and Madder were crushed (10g/l) and soaked for 16 h followed by boiling for 2 h. The extract was filtered and used for dyeing silk using the same liquor ratio as for mordanting. Multifibre test fabric was used for assessment of staining.

Dyeing of silk with Kikar bark. To achieve a 1% owf shade on unmordanted silk, fabric was entered into the dye bath at 60°C; this temperature was held for 10 min and then raised to 85°C over 35 min at liquor ratio 30:1. After dyeing the cloth was removed and raised, soaped at the boil for 15 min, washed thoroughly and dried. Similar procedures were used for dyeing 2% and 4% (owf) shades.

The three different methods of dyeing with mordants were pre-mordanting, meta-mordanting and post-mordanting. Mordant concentration of 5 and 10g/l were used.

In the pre-mordanting method, the fabrics were first immersed in an aqueous solution of alum, copper sulphate or ferrous sulphate for 45 min at 30°C. All of the mordanted fabrics were then dyed by the above method.

For the meta-mordanting method (i.e. dyeing in the presence of mordants) the fabrics were immersed in bath containing a mordant and the dye extract. The temperature was raised to 90°C over 30 min and held for 1 h. The fabrics were rinsed at 60°C, washed with water, squeezed and dried.

In post-mordanting method, dyeing was carried out in the absence of a mordant, followed by mordanting in a separate bath containing a mordant at 30°C for 45 min. Further processing was the same as described in the meta-mordanting method.

Fastness and color measurements. Wash fastness of all dyed samples was determined by ISO CO2 method and Crock fastness was carried out according to ISO X 12 test method. Light fastness of dyed samples was determined by exposing them to sunlight according to ISO BO2. The change in color and staining on multifibre test fabric was assessed by comparing sample with Grey scale. The results have been summarized in Table 1 for Kikar bark.

Dyed samples were prepared for color measurement, which was carried out by following a standard procedure (Bryan 1987). Color values were evaluated by means of K/S and CIE Lab color-difference values by spectrophotometer with data master V 2.3 software (Data color, international, USA). Four measurements were made on each of the four samples and the variation in percentage reflectance values over a range of 400-700 nm was recorded. The reproducibility of the results was also checked and found to be satisfactory in all cases. The dyeing performance in the various processes was measured in terms of K/S value at their λ_{max} using spectrophotometer. The reflectance and K/S values were recorded for all the dyed samples by using a colorimeter and the result is recorded in Table 1. Changes in color were also measured on the basis of CIE Lab color space in terms of L^* a^* b^* (Cartesian coordinates) and the difference in values obtained for original and faded samples were measured at ΔE and the results are given in Table 3.

Dyeing of silk with madder bark. The controlled and mordanted samples of silk dyed with madder extract were prepared and their fastness properties and color measurements were determined. The results of these studies are indicated in Table 2 and 4.

Results and Discussion

In the actual dyeing process a mordant combines chemically with a soluble dye to form a very complex, aggregated, insoluble lake of high molecular weight within the textile fibre. Lake is formed when solubilized natural dye is rendered insoluble by complexing with mordanting salts and makes the fibre resistant to the external influence in washing and finishing processes. The fastness depends on the formulation of lake inside the textile fibres (Ali 1993). The silk fabric was dyed with Kikar bark and Madder bark at 2% and 4% dyeing concentrations with different mordants (alum, copper sulphate and ferrous sulphate) at 5g/l and 10g/l concentrations. The results of fastness properties of the two natural dyes are given in Table 1 and Table 2.

The metals used as mordants are well known for their ability to form co-ordination complexes and readily chelated with the dye. As the co-ordination numbers of copper and iron are 4 and 6 respectively, some co-ordination sites remained unoccupied where they interacted with the fibre. Functional groups such as amino and carboxylic acid groups on the fibre can

Table 1
Fastness properties of silk dyed with Kikar bark

Samples	Mordant concentration g/l	Crock fastness		Washing fastness						Light Fastness
		Dry	Wet	Cellulose acetate	Cotton	Nylon	Polyester	Acrylic	Wool	
Control										
Natural		3-4	3-4	2-3	2	2	3-4	3	3-4	3-4
Pre-mordanting										
Alum	5	2-3	2-3	4-5	4-5	4-5	4-5	4-5	4-5	4
Alum	10	3	3	4-5	4-5	4-5	5-5	4-5	4-5	4
Copper	5	3-4	3-4	4-5	4-5	4-5	4-5	4-5	4-5	4
Copper	10	3-4	3-4	4-5	4-5	4-5	4-5	4-5	4-5	4
Ferrous	5	3-4	3-4	4-5	5-4	5-4	5-4	5-4	5-4	4-4
Ferrous	10	4	4	4-5	5	4-5	4-5	4-5	4-5	4-5
Meta-mordanting										
Alum	5	2	2	2-3	3	3	3-4	3-4	2-3	4
Alum	10	3	3	2-3	3	3	4-5	4-5	2-3	4-5
Copper	5	2	2	4-5	2	2	4	4-5	4-5	4-5
Copper	10	2	2	4-5	2	2	4	4-5	4-5	4-5
Ferrous	5	3	3	3-4	3	2-3	4-5	4-5	4	4-5
Ferrous	10	3	3	4	4-5	4	4-5	4-5	4	4-5
Post-mordanting										
Alum	5	2-3	2-3	5	5	4-5	4-5	4-5	4-5	4
Alum	10	3	3	5	5	4-5	4-5	4-5	4-5	4
Copper	5	4	4	4-5	4-5	4-5	4-5	4-5	4-5	4
Copper	10	4	4	4-5	4-5	4-5	4-5	4-5	4-5	3-4
Ferrous	5	4	4	5	5	4-5	4-5	5	4-5	4-5
Ferrous	10	4	4	5	5	4-5	4-5	5	4-5	4

occupy these sites. These metals can form a ternary complex on one site with the fibre and on the other site with the dye. Such a strong co-ordination tendency can enhance the interaction between the fibre and the dye, resulting in high dye uptake.

*Fastness and color properties of silk dyed with Kikar bark.*The samples dyed were washed with soap water and the staining on multifibre test fabric was determined. Significant changes were noted for cellulose acetate, cotton, nylon, polyester, acrylic and wool and were compared against the original samples by Grey scale measurement. The results of wash fastness are given for each mordant in Table 1.

As shown in Table 1 the staining on the substrates mentioned above is high for control samples showing poor fastness. The staining on cotton after dyeing silk with the technique called pre-mordanting showed good wash fastness property, having high fastness value when dyed with ferrous sulphate. The samples dyed with the technique meta-mordanting showed low fastness properties as had been shown by high staining on cotton and nylon when dyed with copper sulphate as compared to alum and ferrous sulphate where the fastness is also poor. The silk dyed with these mordants by using the technique named as post-mordanting showed good fastness property for all three mordants, the Grey scale being very good for cellulose acetate, cotton and acrylic fabric for alum and ferrous sulphate.

As shown in Table 1 the crock fastness of controlled sample dyed with natural dye was found to be poor for both dry and wet crocking. The silk samples dyed under pre-mordanting technique showed poor dry and wet crock fastness for alum and copper sulphates. With ferrous sulphate dry and wet crock fastness properties were also poor. In the case of meta-mordanting the samples showed again poor dry and wet crock fastness ratings. The lowest crock fastness was obtained with copper sulphate. In the case of post-mordanting good crock fastness was found for alum and copper but for ferrous sulphate the fastness was very poor.

The results of light fastness of dyed samples are presented in Table 1. Mordanting with mordant concentrations at 5g/l and 10g/l were compared with the values found for the control samples of natural dyes. As shown in Table 1 the light fastness showed high rating of Grey scale which is good to very good.

Table 2
Fastness properties of silk dyed with Madder bark

Samples	Mordant concentration g/l	Crock fastness		Washing fastness						Light fastness
		Dry	Wet	Cellulose acetate	Cotton	Nylon	Polyester	Acrylic	Wool	
Control										
Natural		3-4	3-4	2-3	2	2	3-4	3	3-4	3-4
Pre-mordanting										
Alum	5	4	4	4	4-5	4-5	4-5	4-5	4-5	4-5
Alum	10	2-3	2-3	4-5	4-5	4-5	4-5	4-5	4-5	4
Copper	5	4	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Copper	10	4	4	4	4-5	4	4-5	4-5	4-5	4-5
Ferrous	5	4	4	4	4-5	4	4-5	4-5	4-5	4
Ferrous	10	2-3	2-3	4	4-5	4	4-5	4-5	4-5	4
Meta-mordanting										
Alum	5	3-4	3-4	4-5	4	4-5	4-5	4-5	4-5	4-5
Alum	10	2	2	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Copper	5	4	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Copper	10	4	4	4	4-5	4	4-5	4-5	4-5	4-5
Ferrous	5	4	4	4-5	4-5	4-5	4-5	4-5	4-5	4
Ferrous	10	4	4	4	4-5	4	4-5	4-5	4-5	4-5
Post-mordanting										
Alum	5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4
Alum	10	4	4	4-5	4-5	4-5	4	4-5	4-5	4
Copper	5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Copper	10	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Ferrous	5	4-5	4-5	4	4-5	4-5	4-5	4	4-5	4
Ferrous	10	4-5	4-5	4	4	4-5	4	4-5	4-5	4

Reflectance of the dyed silk with Kikar (control sample) has been found to be 9.67 and the K/S value calculated from this reflectance is 1.19.The dyeing of silk with Kikar by pre-mordanting with alum, copper and ferrous sulphate (5g/l) had shown reduction in the reflectance values for alum, copper and ferrous sulphate, respectively. By increasing the strength of mordant to 10g/l the reflectance value decreased. These values showed an increase in the strength of color of the dyed sample which is confirmed by an increase in the K/S values at 5g/l concentration of alum, copper sulphate and ferrous sulphate respectively as compared to the value of control sample.

The results also showed high values for ferrous sulphate at the concentration of 5g/l as compared to alum and copper sulphate. For meta-mordanting similar trends have been followed for the reflectance and K/S values. In this case ferrous sulphate showed high reduction in reflectance value and highest increase in K/S values at 5g/l concentration. In the case of post-mordanting the reflectance and K/S values increased at 5g/l concentration of ferrous sulphate. With the increase in the concentration of ferrous sulphate to 10g/l the values decreased for reflectance and K/S, respectively.

The color co-ordinates of dyed samples have been determined by spectrophotometer with data master V 2.3 software (Data colour, International, USA) against the control sample of natural dye and inter comparison of different techniques used for dyeing were made by measuring the difference in coordinates L*, a* and b*. The results of these studies are presented in Table 3.

As shown in Table 3 the values of L* were higher for the concentration of mordants at 5g/l as compared to 10g/l showing that color depth is maximum up to 5g/l and decreased after increasing the concentration of mordants. Similar trend was observed in all the techniques of mordanting. The variation in a* values of sample dyed with pre-mordanting and post-mordanting techniques showed negative values as compared to the a* values of samples dyed with the meta-mordanting which had positive values. The negative values of a* showed that the color moved towards the green side along the red-green axis and the positive value of a* showed that the color

Table 3
Color properties of silk dyed with Kikar bark

Sample		K/S values	Reflectance	Color coordinates CIE Lab difference		
				DL*	Da*	Db*
Control						
Natural		4.22	9.67			
Pre-mordanting						
Alum	(5 g/l)	6.34	2.72	15.91	-(4.09)	-(3.71)
Alum	(10 g/l)	5.82	2.49	9.08	-(6.05)	-(6.32)
Copper	(5 g/l)	5.12	2.20	9.54	-(9.21)	-(8.75)
Copper	(10 g/l)	4.78	2.05	8.01	-(6.50)	-(9.94)
Ferrous	(5 g/l)	7.66	3.29	8.78	-(4.45)	+11.00
Ferrous	(10 g/l)	7.50	3.22	8.46	-(2.70)	+13.55
Meta-mordanting						
Alum	(5 g/l)	12.56	5.43	13.73	+14.62	+8.53
Alum	(10 g/l)	8.54	3.67	13.82	+12.41	+14.60
Copper	(5 g/l)	14.02	6.02	9.81	+6.77	-(5.17)
Copper	(10 g/l)	9.66	3.29	11.53	+6.31	-(4.07)
Ferrous	(5 g/l)	15.27	6.56	2.26	+2.99	+6.01
Ferrous	(10 g/l)	11.36	4.88	1.75	+1.03	+8.88
Post-mordanting						
Alum	(5 g/l)	4.78	2.05	13.76	-(5.04)	+2.20
Alum	(10 g/l)	4.56	1.96	19.00	-(2.24)	+3.74
Copper	(5 g/l)	5.19	2.21	22.52	-(4.50)	-(4.15)
Copper	(10 g/l)	8.84	2.08	25.52	-(3.93)	-(3.10)
Ferrous	(5 g/l)	6.45	2.77	16.49	-(3.64)	+5.29
Ferrous	(10 g/l)	6.25	2.68	20.06	-(3.22)	+5.20

was shifted towards black axis of the CIE L a* b* space. The changes in b* values of samples dyed with pre-mordanting technique showed negative values with alum and copper indicating color shift towards the blue side along the yellow-blue axis. As compared to the ferrous sulphate which showed positive values indicating color towards the yellow side. Whereas in the case of meta-mordanting and post-mordanting, the alum and ferrous sulphate showed positive values of b* as compared to copper sulphate which showed negative b* values in the aforementioned dyeing techniques.

Fastness and color properties of silk dyed with Madder bark. Fastness to washing, crocking and light was determined as for the Kikar bark and the results presented in Table 2 showed good to very good fastness values of silk dyed with mordants as compared to the untreated or controlled sample. The post-mordanting technique of dyeing with mordants at 5g/l and 10g/l concentrations showed high rating of fastness to washing, crocking and light as compared to pre-mordanting and meta-mordanting. Dyeing of silk with the three dyeing techniques showed high rating of fastness properties with ferrous sulphate and copper sulphate as compared to alum.

The reflectance and the K/S values were determined for controlled and mordanted samples by following the same procedure as given for Kikar bark and the data obtained is presented in Table 4. The reflectance of controlled sample was decreased when the dyeing was performed with mordants by using these three techniques whereas the K/S values increased for the mordanted samples. For all the three techniques, of dyeing the samples dyed with copper sulphate at 10g/l concentration showed highest values of K/S. Increase in the K/S values of all the mordanted samples of silk showed that the strength of color was increased as compared to the controlled samples.

The color co ordinates L*, a* and b* of the controlled and mordanting samples with Madder bark are reported in Table 4. The L* values of dyed silk increased when the dyeing were performed at 10g/l concentration under pre-mordanting and post-mordanting techniques and indicated a reduction in color strength. The L* values for the mordanted samples showed an increased value at 5g/l as compared to 10g/l concentration.

The values of a* for samples dyed with pre-mordanting and post-mordanting techniques were negative indicating

Table 4
Color properties of silk dyed with Kikar bark

Sample		K/S values	Reflectance	Color coordinates CIE Lab difference		
				DL*	Da*	Db*
Control						
Natural		1.19	24.17			
Pre-mordanting						
Alum	(5 g/l)	3.02	10.23	(18.26)	-(1.07)	+7.46
Alum	(10 g/l)	4.43	9.29	(22.05)	-(0.90)	+4.73
Copper	(5 g/l)	3.94	12.65	(8.81)	-(0.56)	+13.26
Copper	(10 g/l)	7.23	6.10	(10.45)	-(2.22)	+9.12
Ferrous	(5 g/l)	3.38	11.56	10.18	-(2.02)	+8.83
Ferrous	(10 g/l)	5.87	7.32	(15.87)	-(0.89)	+6.22
Meta-mordanting						
Alum	(5 g/l)	2.13	24.09	8.01	-(3.66)	+7.84
Alum	(10 g/l)	2.83	18.28	7.11	-(2.75)	+9.82
Copper	(5 g/l)	3.68	24.01	10.44	+15.58	-(15.98)
Copper	(10 g/l)	4.85	23.02	8.68	+20.60	-(15.74)
Ferrous	(5 g/l)	2.83	10.81	4.95	+3.40	+21.66
Ferrous	(10 g/l)	1.82	8.60	4.72	+7.33	+23.76
Post-mordanting						
Alum	(5 g/l)	3.04	12.09	(5.98)	-3.71	+2.63
Alum	(10 g/l)	3.79	10.55	(7.62)	-3.59	+5.72
Copper	(5 g/l)	5.19	16.28	(18.64)	-(1.57)	-(3.43)
Copper	(10 g/l)	5.57	10.56	(24.09)	-(2.08)	-(1.34)
Ferrous	(5 g/l)	2.15	8.13	(18.77)	-3.65	+17.73
Ferrous	(10 g/l)	3.79	3.21	(21.65)	-3.08	+20.61

the shift of color on the green side along the red-green axis. On contrast the a* values for samples dyed with meta-mordanting remained positive showing the position of color on the red side of red-green axis except with alum which showed negative values. The change in the b* values of the samples dyed with pre-mordanting and post-mordanting techniques showed positive values indicating yellowish colour along the yellow-blue axis, whereas for meta-mordanting with copper sulphate at 5g/l and 10g/l negative values of b* were recorded which indicated the shift of color toward blue on yellow-blue axis. The dyeing with alum and ferrous sulphate under meta-mordanting technique, on the other hand, showed positive values of b* at the both mordants concentrations.

Conclusion

Silk fabric was dyed with Kikar and Madder bark dye extract by three different techniques named as pre-mordanting, meta-mordanting and post-mordanting using metal salts as mordant. The dyeing behaviour has been assessed by measuring K/S values and different fastness properties. In general the fastness properties obtained can be sufficiently good for practical dyeing. From the above findings that natural dyes from the two sources exist in a highly aggregated form in silk fibres, thereby exhibiting good resistance to washing, crocking and light exposures. Following these techniques this work can be expanded to other plant that yield colorants to examine the possibility of using the dye commercially as a safer substitute for synthetic dyes.

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