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UTILIZATION OF *QUERCUS ROBUR* L. (FRUIT CUPS) AND *SALIX ALBA* L. (WOOD EXTRACT) ASDYEING AGENTS FOR SILK AND COTTON FABRICS

SYED MAQBOOL GEELANI*[1], S.J.A. BHAT [2], GAZALA QAZI[1], SHAZMEEN QASBA [2], SUJA N QURESHI[3], AFAQ A PARRAY[2], P.K. MISHRA[4], SHOUKAT ARA[1], ASIF H. SOFI [5] AND P.A. KHAN [2]

[1] Division of Environmental Sciences, SKUAST-K, Shalimar, (J&K), India
[2] Faculty of Forestry, SKUAST-K, Wadura Campus, (J&K), India
[3] Division of Fruit Sciences, SKUAST-K, Shalimar (J&K), India
[4] Department of Chemical Engineering and Technology, IIT (BHU), Varanasi (UP), India
[5] Division of LPT, SKUAST-K, Shuhama Campus, (J&K), India
[6] Division of Agricultural Statistics, SKUAST-K, Shalimar, (J&K), India

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ABSTRACT

Investigation was carried out to study the dyeing potential of *Quercus robur* L. and *Salix alba* L. of which fruit cups and wood extract were used as source of natural dye and mordant. Dyeing potential of the extracted material was tested on silk and cotton fabrics. Keeping in view the negative impacts of chemical agents, extraction and application was carried out using distilled water. Natural mordant used in combination with natural dye showed satisfactory results on silk fabrics both including and excluding mordant, however poor quality of dye and mordant was recorded on cotton fabric. The naturalmordant gave silk fabrics rapid dye adsorption, enhancedcolour coordinates, K/S, ΔE and also showed improvement in fastness properties with regard to washing, light and rubbing of the dyed silk fabric. Cotton fabric does not showed much affinity for the natural dye and mordant.

INTRODUCTION

One of the most environmentally unfriendly industrial processes includes textile dyes, which points us towards the extreme and adverse of the dyeing processes with an alternativeof ecofriendly methods (Wanyama, 2014). Coloured products have gained a lot of importance in present day societies. It is of relevance to optimize the coloring process with the objective of reducing the environmental impact of the textile dye industry. Increase in the demand of the dyed textile products with proportional increase in its manufacturing by utilizing synthetic dyes have contributed to the severe pollution of water, soil and as a whole to our environment (Dos *et al.*, 2007). Over 10,000 different dyes and pigments are used industrially and over 7×10^5 tons of synthetic dyes are annually produced worldwide (Ogugbue and

* **Corresponding author's email :** geelani111@gmail.com; (Syed Maqbool Geelani)

Sawidis, 2011). Global concerns over the carcinogenic effects, toxicity and allergic reactions due to synthetic dyes have gained interest in the revival of natural dyes in textile coloration. (Kulkarni *et al.*, 2011). Vast number of colours can be obtained from plants of which, Leaves, roots, flowers, barks, fruits and stems can be used. Due to non-carcinogenic and biodegradable nature, natural dyes are clinically safer (Aminoddin and Haji, 2010). Safety of natural dyes needs to be proved if they are used more widely and commercially (Kumar and Bharti, 1998). Natural dyes are gaining demand in international market which is about 10,000 tonnes, which is equivalent to 1% of the world synthetic dye consumption (Sachan and Kapoor, 2007). Natural dyes are excellent for long endurance and retain great beauty and charm. In view of advancements in technology and urban development, there is dire need to assess the environmental pollution problems caused due to synthetic dyes (Samanta and Agarwal, 2009). Considerable research work is being conducted throughout the world to search out new cheaper dye sources of which the explored natural dye and mordant can be an important sources which can be utilized commercially.

MATERIALS AND METHODS

Collection of Material

The plant material of *Quercus robur* L. (fruit cups) as dyewas collected from Dachigam wild life sanctuary, Srinagar and material of *Salix alba* L. (wood) as mordant was collected from university outskirts. Silk and cotton were purchased from Govt. silk weaving factory and Poshish (JKHDC) outlet at Srinagar (J&K).

Quercus robur L. (fruit cups)

Salix alba L. (wood)

Preparation of dyeing material

Quercus robur L. (fruit cups) were used as natural dye source. The shade dried fruit cups were washed with water to remove dirt and other adhering materials followed by drying in tray drier at 80 ^{o}C for 2 hours, finely powdered with the help of a grinding machine. The material was then passed through a standard test sieve (BSS-14).

Preparation of Mordant

The stem of willow (*Salix alba*L.) was cut into small pieces and soaked in distilled water for 30 days. 10 L of distilled water was used for 1 Kg of Willow stem wood. The solution was filtered and kept under refrigeration for further usage.

Extraction of dye

The dyeing material was extracted using Soxhlet apparatus using distilled water as solvent. 1 L of distilled water was used for 100g of plant material. The material was kept for reflux for about 8 hrs. at 80-85 ^{o}C. Liquid extract was evaporated at 65 ^{o}C in a rotary vacuum evaporator to one fourth of its original volume to obtain the final dyeing extract.

Scouring of fabrics

Fabric was cut into 5×6 cm, followed by washing with 2% non-ionic soap (Labolene) at 50 ^{o}C for 20 min, maintaining the material-to-liquor ratio at 1:50. The scoured material was then washed thoroughly with plenty of tap water and dried at room temperature. The scoured material was soaked in distilled water for 30 minutes prior to dyeing.

Dyeing of Fabrics

Process of dyeing was carried out in a water bath maintaining material to liquor ratio 1:50. The scoured and washed fabric wasdipped in 250mL beaker containing 100mL of dyeing solution and 4% dye (OWM) at room temperature andraised to 85 ^{o}C - 90^{o}C with gentle stirring continued for 1 hour. The material was then removed and washed 2-3 times with 1% of detergent and water. Dyed samples were squeezed and dried at room temperature. Dyeing of the silk fabric was done at acidic pH by adding acetic acid (CH_3COOH), whereas cotton fabric was dyed at basic pH by adding Sodium carbonate (Na_2CO_3).

Mordanting

The mordanting was carried out by the following three methods, i.e. pre mordanting, simultaneous mordanting and post mordanting.

Recipe of mordanting

Mordant	*Salix alba* L. (wood extract)
Concentration (%)	15
Temperature (oC)	60-75
Material to Liquor ratio (MLR)	1:50
Time (hrs)	1

Determination of percent absorption of dye

The absorption of the natural dye by the selected fabrics was calculated by recording the optical density of the dye solution both before and after dyeing process. The ultraviolet-visible adsorption spectra (UV-VIS) was recorded on PC based double beam spectrophotometer.

(Systronics 2202) over the range of 200-800 nm. The percent absorption of natural dye was calculated by using the following equation-

$$\text{Percent absorption} = \frac{\text{O.D before dyeing - O.D after dyeing} \times 100}{\text{O.D before dyeing}}$$

Evaluation of CIE L*a*b* values of dyed fabrics

CIE L*a*b* values of the dyed and undyed fabrics was determined by chromometer (Model CR-2000, Minolta, Osaka, Japan) equipped with 8 mm measuring head and AC illumination (6774 K) based on CIE system (International Commission on Illumination). The meter was calibrated using the manufacturer's standard white plate.

L*, a* and b* coordinates, Chroma (C*) and hue angle (h^o) values were calculated by the following equations.

Chroma = $(a^*2 + b^*2)^{1/2}$
Hue = $(h = \tan^{-1} b^*/a^*)$

Total colour change of the dyed fabrics was calculated from the L*, a* and b*coordinates by applying the following equation.

Total Colour change (ΔE):

$(\Delta E) = [(\Delta L^*)2 + (\Delta a^*)2 + (\Delta b^*)2\,]^{1/2}$
Where,
ΔL* = L* sample - L* standard
Δa* = a* sample - a* standard
Δb* = b*sample - b*standard

Determination of Colour strength (K/S) value

The colour strength (K/S values) of the undyed and dyed fabric samples was evaluated using JAYPAK 4802 colour matching system (Jay Instruments Ltd, Mumbai, India) at D65 illuminate/10 Deg observer. The reflectance of the samples was measured at 360-760 nm. The colour strength value in the visible region of the spectrum (400-700 nm) was calculated based on the Kubelka-Munk equation.

$$\frac{K}{S} = \frac{(1-R\lambda)^2}{2\times R\lambda}$$

Where, K is the coefficient of absorption, S is the scattering coefficient and R is the surface reflectance value of the sample at a particular wavelength, where maximum absorption occurs for a particular dye/ colour component.

Relative colour strength

Relative colour strength (K/S values at maximum wavelength) was determined by adopting the following equation.

$$\text{Relative strength} = \frac{\text{(K/S) Extracted}}{\text{(K/S) Raw}}$$

Evaluation of colour fastness properties

The Colour fastness tests of the dyed fabrics were carried out as per ISO 105-B02 for light, ISO 105 X-12 for rubbing and ISO 105-C01 for washing respectively. Grading for colour change and colour staining were evaluated as per ISO 105-AO2 and ISO 105-A03.

RESULTS AND DISCUSSION

Percent absorption of dye

Percent absorption of dye is an important factor governing the colour quality of the fabric. Dye and mordant combinationsshowed satisfactory results on the silk fabric however, cotton fabric does not showed any satisfactoryresult. In both cotton and silk fabric, mordanting methods followed the same trend and enhanced the absorption of the dye where, simultaneous method recorded highest value of dye absorption in both cotton and silk fabric followed by pre, post and without mordanted samples recording the lowest values of absorption(Fig. 1). Increase in percent absorption of dye may be attributed to the formation of the chemical bridge between the dye and fabric through mordant, which get fixed on the fibre and help in fixation of the dye (Prabhu and Bhute,

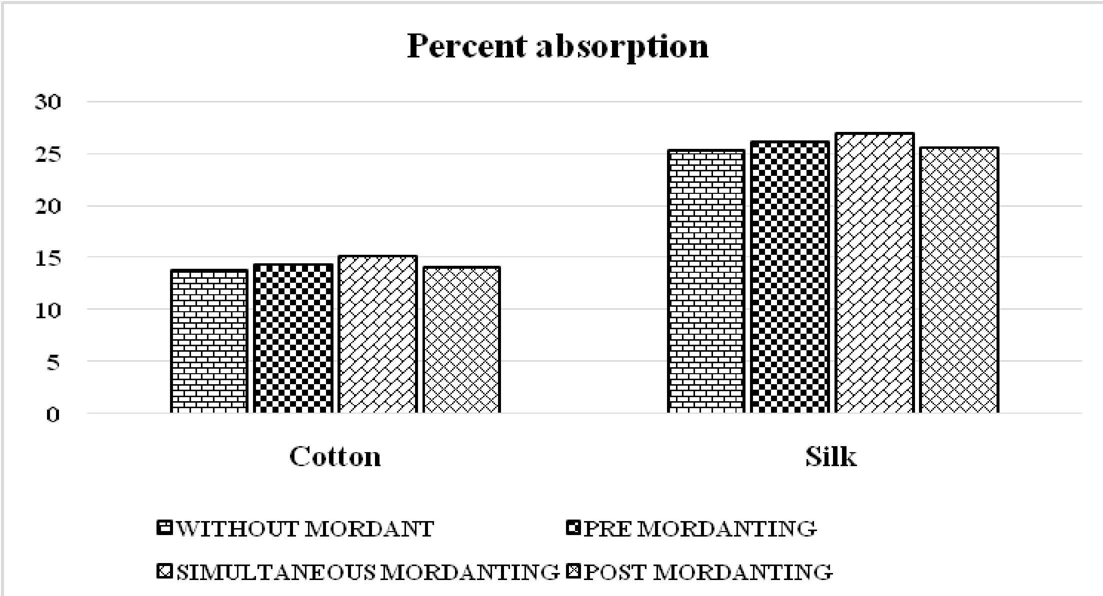

Fig. 1 Percent absorption of dyed fabrics

2012). The low coordination tendency of cotton fabric with the selected dye and mordant causes low dye absorption (Tiwari *et al*., 2010).

Colour quality of dyed fabrics

Mordant type and method of mordanting showed significant difference in colour coordinates of the dyed fabrics.All the dyed fabrics showed variation in L*, a*, b*, C*, h^o, ΔE, K/S and relative K/S values. Where, the cotton fabric recorded higher values of lightness and lower values of a*, b* and and C* as compared to silk fabric which depicts poor dyeing quality of the cotton fabric. The tone of the dyed samples were much yellowish and less reddish as indicated by the values of the a* and b*. In silk fabric hovalue represented light brownish, brownish and dark brownish colour of the silk fabric and shades of cotton fabric represented light yellowish. Chroma value of the silk fabric recorded higher representing bright colours (Almela, 1995). Colour change recorded higher in silk fabric. Simultaneous mordanting method recorded higher values of total colour change in both cotton and silk fabric which shows efficient results of dye and mordant when used in combination during the process of the dyeing (Table 1). Varying shades of the silk fabric may be attributed to the unique complex formation due to ions of the dye, mordants and different techniques results in the varying shades of the silk fabric (Kampeerapappun *et al*., 2010).

Colour strength of dyed Samples

Colour strength of the dyeing material is an essential parameter governing the extent of the coloring material in the substrate and describes the colour quality of the fabric.Colour strength and relative colour strength of the silk fabric recorded higher than that of the cotton fabric. In both silk and cotton fabrics unmordanted dyed samples recorded higher value than that of the mordanted samples. Mordanting methods adopted also showed variation in values of colour strength with the highest value recorded in without mordanted cotton and silk samples. However pre mordanting in cotton and simultaneous mordanting in silk fabric showed the successive higher values of K/S among all the methods adopted (Fig. 2 & 3).which is due to stripping of the dye molecules during the process of mordanting, which subsequently forms an insoluble complex with dye molecules in solution resulting in poor colour strength (Deo and Desai, 1999). Negatively charge of cotton in water exhibits poor absorption of natural dyes due to its repulsion effects leading poor colour strength (Kampeerapappun, 2010).

Fastness properties of dyed fabrics

Fastness requirements are largely determined by the end use of dyed fabrics. Washing, rubbing and light fastness grades of dyed samples recorded satisfactory both in cotton and silk fabrics. However, colour of the

Table 1. Colour coordinates of fabrics dyed with *Quercus robur* L. (fruit cups) dye and *Salix alba* L. (wood extract) mordant.

CIELAB/Methods	L*	a*	b*	C*	ho	ΔE
Cotton sample						
Without mordant	80.44	4.50	19.73	20.24	77.10	18.17
Pre mordanting	85.09	2.82	18.35	18.57	81.30	13.99
Simultaneous mordanting	80.76	3.13	16.95	17.24	79.50	16.03
Post mordanting	80.55	3.25	14.99	15.24	77.70	15.04
Silk sample						
Without Mordant	65.81	4.84	28.98	29.39	80.50	30.03
Pre mordanting	62.17	9.28	23.80	25.55	68.70	30.42
Simultaneous mordanting	67.45	7.06	24.16	25.14	73.70	25.72
Post mordanting	71.00	5.78	24.62	25.29	76.80	23.05

Table 2. Fastness grades of dyed fabrics dyed with Quercus robur L. (fruit cups) and Salix alba L. (wood extract) adopting different mordanting methods.

Method	Washing fastness		Light fastness	Rubbing fastness			
				Staining		Fading	
	CC	CS	CC	Dry	Wet	Dry	Wet
Cotton fabric							
Without mordant	4/5	4/5	4	5	5	5	4/5
Pre mordanting	4/5	5	4	5	5	5	4/5
Simultaneous mordanting	4/5	5	4/5	5	5	5	4/5
Post mordanting	4	5	4/5	5	5	5	4/5
Silk fabric							
Without mordant	4/5	5	4/5	5	5	5	5
Pre mordanting	4/5	5	4/5	5	5	5	5
Simultaneous mordanting	4/5	5	4/5	5	5	5	5
Post mordanting	4/5	5	4/5	5	5	5	5
CC: Colour change							
CS: Colour staining							

silk fabric showed more satisfactory grades than cotton fabric. Washing fastness of the cotton fabric showed excellent grades (5). Where, mordanted samples recorded higher grades than unmordanted dyed samples with lower gradesof colour change and higher grades of colour staining. In silk fabric colour change recorded Good to Excellent (4\5) grade and staining recorded Excellent grade (5) both in unmordanted and mordanted samples. Light fastness of the dyed silk fabric recorded good to excellent (4\5) to excellent (5) grade in silk fabric and good to excellent (4\5) to excellent (5) grade adopting simultaneous and post mordanting method with good (4) in case of without and pre mordanted dyed samples. Rubbing fastness of silk fabric recordedExcellent (5) grade in all types of dyed fabrics. However, in cotton fabric wet fading recorded slight lower grade of good to excellent (4\5) to excellent (5). Assigned grades in Table 2 shows satisfactory fastness quality of the dyed fabrics both without and with mordanted samples with best quality in case of the silk fabric.Formation of the mordant and dye complexes which are able to form coordination linkages between the dye and mordant results in higher fastness grades of the dyes and mordants when applied on the fabric (Ali *et al.*, 2009). The washing fastness of the dye is influenced by the rate of the diffusion of the dye and state of the dye inside the fibre, (Jothi, 2008). Good light fastness is due to the formation of complex with the mordant which protects chromatophore from photolytic degradation (Kulkarni *et al.*, 2011). The higher grades of the wet rub fastness than dry rub fastness may be due to dissolving of water soluble dye molecules which make them easier to be removed from the fibre by rubbing (Haji, 2010).

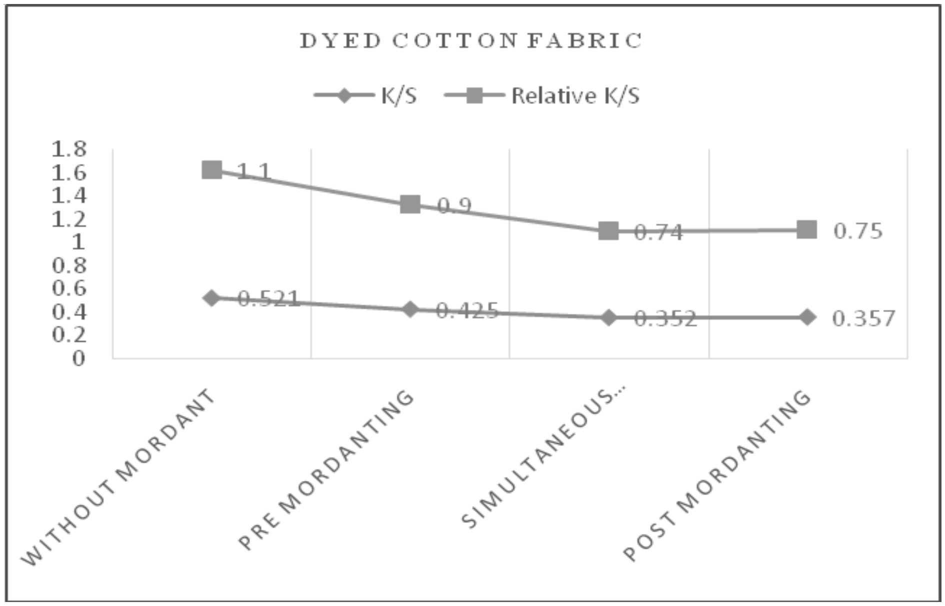

Fig. 2 Colour strength of dyed Cotton fabric

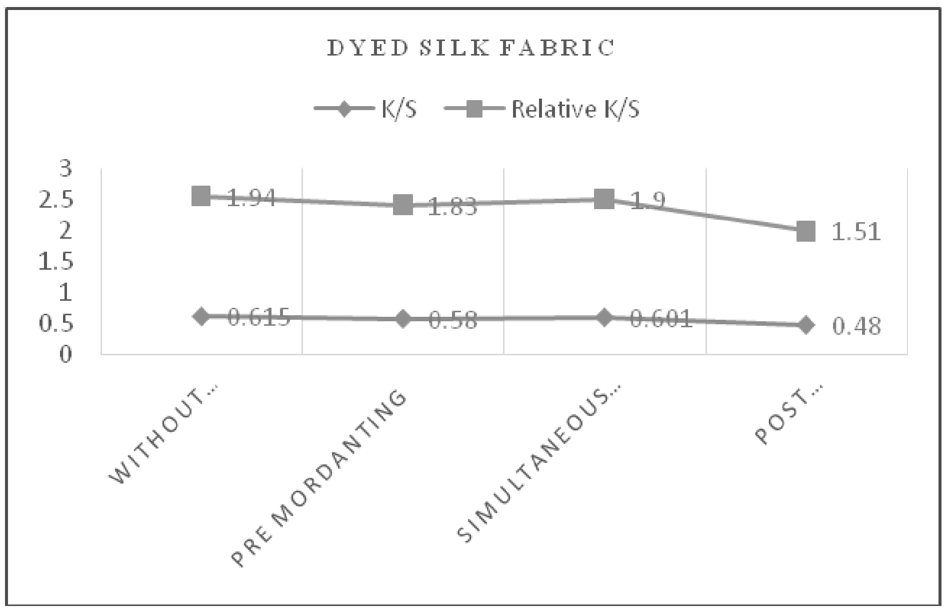

Fig. 3. Colour strength of dyed Silk fabric

CONCLUSION

Extracted dye and mordant was successfully applied on cotton and silk fabric which produced beautiful shades by adopting different mordanting methods. The dye and mordant alone and in combination does not showed much affinity for the cotton fabric. Experimental results revealed significant association of bio mordant when used in combination with natural dye. Percent absorption recorded best results in combination with and without mordant also in silk fabric. However, cotton fabric recorded poor values of absorption. CIELAB, K/S and relative K/S values reported excellent results in case of silk fabric with different shades and poor dyeing quality of cotton fabric. Fastness tests of the dyed samples also recorded satisfactory grades. The results obtained for silk fabric justifies the good quality of the extracted

Test fabrics for dyeing

Dyed cotton samples

Dyed Silk samples

natural dye and mordant which suggests its commercial usage in textile industries.

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