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Green dyeing of wool and cotton fabrics using Juglans regia L. (leaves) and Salix alba L. (wood ash)

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ABSTRACT

The work was conducted with an aim to extract natural dye and mordant from plant materials. The natural dye was extracted from Juglans regia L. (leaves) and material of mordant was prepared from Salix alba L. (wood ash). The dye was extracted using Soxhlet apparatus. The extracted material was tested on wool and cotton fabrics which were dyed by adopting different methods. The dyed material appeared with beautiful light brown, dark brown and yellowish colours with varying shades and tones, which was subjected to various quality and retention tests like percent absorption, colour coordinates (CIELAB), colour strength (K/S), relative K/S and fastness properties as per standard ISO test methods. The results showed excellent absorption of dye, Colour strength (K/S), CIELAB values and retention grades in case of wool fabric. However, cotton fabric appeared with dull and poor colour quality with low grades of fastness and retention. The usage of the mordant affected the uptake of the dye by the wool fabric which enhanced the colour quality of the fabric and showed different shades and tones of colour in each mordanting method by just using a single dye source. The cotton fabric does not show much affinity for the dye and mordant which resulted in poor colour quality of the fabric.

***Key words*: *Cotton, dye, Juglans regia* L., *Salix alba* L., *Wool*.**

I. INTRODUCTION

Colour is an important factor which governs the fabric choice of the consumers. The colour was used since earlier in pre historic times. The colouring agents of food and textiles were procured from natural sources like plants and animals. Dyeing was practiced during the Bronze age in Europe [6]. Though, earliest written record of the use of natural dyes was found in China dated 2600 BC, however dyeing was known as early as 2500 BC in the Indus valley period. The commerce of natural dye was considered as a measure of economic prosperity of nations. Accidental discovery of Mauvine - coloured coal tar products by Sir Henry Perkin was the starting point for synthetic dye industry. The increasing market demand for dyes and the dwindling number of dye yielding plants forced the emergence of synthetic dyes like aniline and coal-tar, which threatened total replacement of natural dyes. Environmental problems from the dyeing of textiles arose after industrialization, when traditional natural dyes were replaced by synthetic dyes. Synthetic dyes are designed to resist chemicals, and improve the quality of the product but are persistent in the environment [2]. The chemicals used to produce synthetic dyes

are highly toxic, carcinogenic, allergic, explosive and dangerous to work with. The production of synthetic dyes involves many violent reactions, which are considered health hazardous [14]. In textile industries, 50% of the dyes are lost in effluents [11] are mostly toxic and carcinogenic to living organisms especially aquatic animals and humans [15]. Textile dyeing is recognized as one of the most environmentally unfriendly industrial processes, it is of extreme importance to understand the critical points of the dyeing process so as to find alternative, ecofriendly methods. Considering the importance of the coloured products in present day societies, it is of relevance to optimize the colouring process with the objective of reducing the environmental impact of the textile industry. As a result of the worldwide concern over the carcinogenic effects, toxicity and allergic reactions associated with synthetic dyes interest in the revival of natural dyes in textile colouration is increasing [9]. Moreover, many countries have already imposed stringent environmental standards over synthetic dyes. Germany was the first to take initiative to put ban on numerous specific Azo dyes for their manufacturing and applications. Netherlands, India and some other countries also followed the ban. The natural dyes are clinically safer than their synthetic analogues, in handling and use because of non carcinogenic and biodegradable nature [2]. Natural dyes exhibit better biodegradability and generally have a better compatibility with the environment. They are less toxic, polluting, health hazardous, non carcinogenic, easily available and renewable [1]. Thus the research work was conducted with an aim to explore the natural wealth for dye dye exploration and its availability in industries for utilization.

II. MATERIALS AND METHODS

2.1. COLLECTION OF MATERIAL

The material of the dye (*Juglans regia* L. leaves) was collected from university campus at Shalimar, Srinagar and material of material (*Salix alba* L.) wood was collected from city outskirts "Plate 1". The test materials for the application of the dye (wool and cotton) were purchased from Poshish (JKHDC) outlet at Srinagar (J&K) "Plate 2".

Plate-1: Test materials of dye and mordant

Plate-2: Test fabrics for dyeing

2.2. Preparation of plant material

The shade dried fresh plant material was washed with cold tap water to remove the adherent materials and then dried in tray drier at 80 ^{o}C for 2 hours and was grinded in a grinding machine. The material was then passed through a standard test sieve (BSS-14) to obtain a fine powder.

2.3. Preparation of Mordant

The willow (*Salix alba* L.) stem was cut into small pieces and was burnt completely into ash and was soaked in distilled water for 10 days. 1 litre of distilled water was used for 100 gm of ash. The ash solution was then filtered and kept under refrigeration for future usage.

2.4. Extraction of dye

The natural dye was extracted using Soxhlet apparatus. 1 L of distilled water was used for 100g of plant material. The dyeing 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.

2.5. Scouring of fabrics

All the test fabrics were cut into 5×6 cm, washed with 2% non-ionic soap (Labolene) and were heated at 50^{o}C for 20 min, keeping the material-to-liquor ratio of 1:50. The fabric was then washed thoroughly with tap water and dried at room temperature.

2.6. Dyeing of Fabrics

The samples of the wool and cotton were soaked in distilled water for 30 minutes prior to the dyeing process. The dyeing of the test fabrics was carried in a water bath by maintaining the material to liquor ratio of 1:60 for wool and 1:40 for cotton respectively. The test fabrics were dipped in 250 ml beaker containing 100ml of dyeing solution and 4% dye (OWM) was heated and temperature was raised to 85^{o}-90 ^{o}C with gentle stirring continued for 1 hour. The material was then washed 2-3 times with 1% of detergent and water. The dyed samples were then dried at room temperature. Dyeing of the wool fabric was done at acidic pH by adding acetic acid (CH_3COOH) and cotton fabric was dyed at basic pH by adding Sodium carbonate (Na_2CO_3) [3].

2.7. Mordanting

The method of mordanting was carried out by adopting pre mordanting, simultaneous mordanting and post mordanting methods. The process of mordanting was performed by using 4% of the mordant solution keeping

the M:L ratio of 1:60 and 1:40 for wool and cotton respectively. The mordanting of the samples was carried at 60-75^{o}C and continued for 1 hour.

2.8. Determination of percent absorption of dye

The absorption of the dye by the fabrics was recorded both before and after dyeing process. By recording the optical density of the dye solution. 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:

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

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

The Colour coordinates (CIELAB) values of the dyed wool and cotton fabric was determined by Chromometer (Model CR-2000, Minolta, Osaka, Japan) equipped with 8mm 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 [3].

$\text{Chroma} = (a^{*2} + b^{*2})^{1/2}$

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

Total colour change (ΔE) 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

2.10. Determination of Colour strength (K/S) value

The value of the Colour strength (K/S) of both dyed and undyed fabrics was observed using JAYPAK 4802 colour matching system (Jay Instruments Ltd, Mumbai, India) at D65 illuminate/10 Deg observer. The

reflectance of the dyed samples was measured at 360-760 nm. The colour strength in the visible region of the spectrum (400-700 nm) was calculated based on the Kubelka-Munk equation, mentioned below:

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

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.

2.11. Relative colour strength

The Relative colour strength (K/S) was determined by using the following equation below:

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

2.12. Evaluation of colour fastness properties.

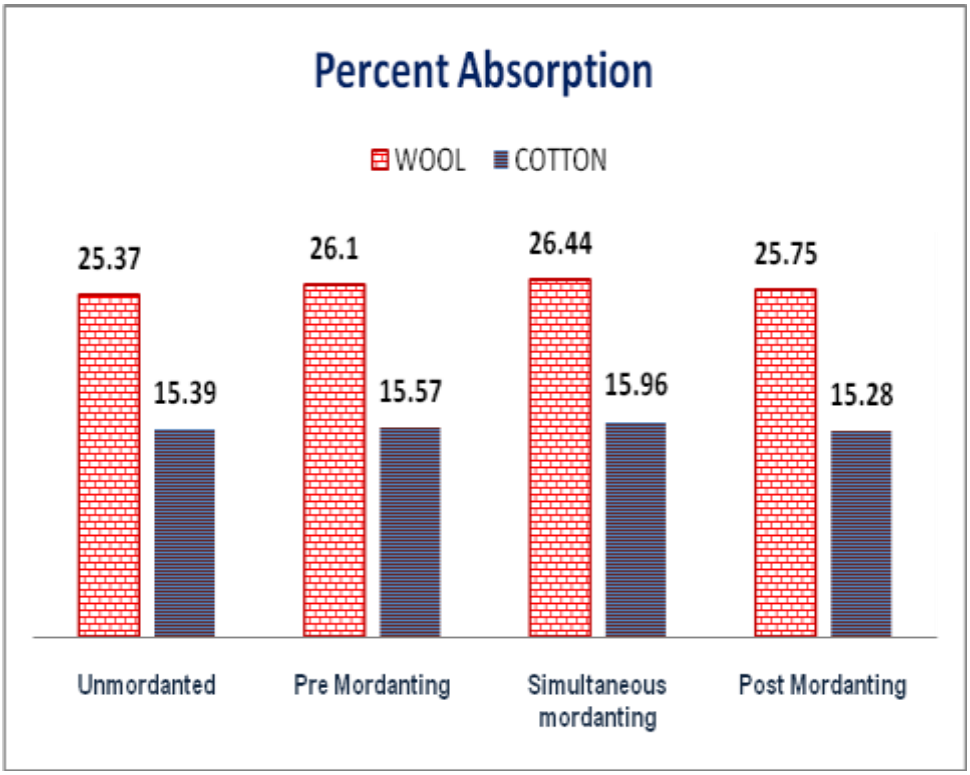
The fastness and retention of the dyed fabrics was recorded by different fastness tests and results were assigned as per the grades of the reference grey scale. The quality and retention of the dye was calculated by washing, rubbing and light fastness tests, carried out as per ISO 105-B02 for light, ISO 105 X-12 for rubbing and ISO 105-C01 for washing respectively. Grading for colour change and colour staining were assigned as per ISO 105-AO2 and ISO 105-A03 [4].

III. RESULTS

3.1. PERCENT ABSORPTION OF DYE

The absorption of the dye greatly affects the colour quality of the dyed fabric. The examination of the absorption of the dye by the fabrics showed variation in each mordanting method which, recorded greater in case of wool fabric than the cotton fabric. In case of the wool fabric highest absorption of the dye was recorded by adopting simultaneous mordanting method followed by pre, post and unmordanted sample respectively. However, cotton fabric showed lower absorption of the dye and recorded highest absorption in case of the simultaneous mordanting method followed by pre, post and lowest in case of unmordanted dyed sample "Fig. 1". These results may be attributed to the presence of multifunctional groups in the protein polymer (-NH2,-OH,-COOH) in the pashmina fabric which binds a bridge between the dye and fabric, resulting in the efficient absorption of the dye than the other fabrics [13]. The lowest absorption of the dye by the cotton fabric may be due to weak coordination complexes developed between cellulosic polymer and dye molecules. With the result, low coordination tendency of cotton with the dye and mordant may have resulted in low dye absorption.

Fig. 1: Percent absorption of dye by the wool and Cotton fabric.



3.2. COLOUR COORDINATES (CIELAB) OF DYED TEST FABRICS

The colour coordinates of each fabric in each method of mordanting varied significantly, which indicates variations in the colour and tones of the each dyed samples. The lightness (L*) values of all the dyed fabrics depicted coloured samples which ranged from 57.77 to 64.12 in case of wool fabric and 71.66 to 80.22 in case of cotton fabric respectively. The higher values of L* in case of cotton fabric depicts less coloured samples. The value of a* in both the wool and cotton fabrics recorded positive and showed bright brown shades with varying tones. However, the positive values of b*, represented yellowish colour of the samples. The dyed fabric appeared with light and dark shades of yellowish, brownish and reddish shades as shown in "Plate 3". The colour of all the dyed wool fabrics showed excellent brightness as depicted by the values of the C*. However, cotton fabric showed low brightness of the colour and appeared with dull and pale colour shades. All the dyed samples of cotton and wool fabrics showed a difference in colour with reference to the undyed and unmordanted sample, which is also confirmed by the values of the ΔE. The ΔE value of the cotton fabric recorded lower than the values of the wool fabric. "Table 1". There is pronounced difference among the dyed samples. This could be correlated with complex forming ability of the metal ions with dye molecules on the fabric [5].

Table-1: Colour coordinates of selected test fabrics.

Colour coordinates / Methods	L*	a*	b*	C*	h^o	ΔE
Wool fabric						
Without Mordant	64.12	2.68	33.50	33.61	85.40	30.93
Pre mordanting	63.81	3.38	34.25	34.42	84.40	31.72
Simultaneous mordanting	61.00	3.89	30.61	30.86	82.70	31.65
Post mordanting	57.77	3.34	32.51	32.68	84.10	35.32
Cotton fabric						
Without Mordant	71.66	3.15	22.29	22.51	81.90	26.53
Pre mordanting	80.22	2.29	19.23	19.37	83.20	17.93
Simultaneous mordanting	77.25	1.92	28.20	28.27	86.10	26.59
Post mordanting	76.95	2.72	23.27	23.43	83.30	23.28

Plate-3: Test fabrics dyed with Juglans regia L.(leaves) and Salix alba L.(Ash)

3.3. COLOUR STRENGTH AND RELATIVE COLOUR STRENGTH OF DYED TEST FABRICS (K/S)

The colour strength (K/S) is a single numerical value related to the amount of light-absorbing material (colourant) contained in the sample usually based on spectral data. The colour strength (K/S) value of both wool

and cotton fabric varied significantly in each mordanted sample. The values K/S values of wool fabric recorded higher than the K/S values of cotton fabric. In wool fabric highest K/S value was recorded in post mordanted dyed sample and lowest was recorded in case of pre mordanted dyed sample. The highest value of K/S in case of cotton fabric was recorded in unmordanted sample and no such effect of mordant was visual due to mordant. However, K/S value of unmordanted samples in both wool and cotton fabric recorded satisfactory which depicts the efficiency of the natural dye in absence of the mordant also "Fig. 2". The relative colour strength value with respect to the undyed and unmordanted samples also followed the same trend which is much evident by the "Fig. 3". The increase in colour strength of dyed wool may be due to fiber swelling and the breakdown of the dye molecule aggregates in the solution became more, thus the diffusion of the dye molecules to the fiber became easier causing increase in colour strength value of the wool fabric [2].

Fig. 2: Colour Strength (K/S) and Relative Colour Strength (K/S)

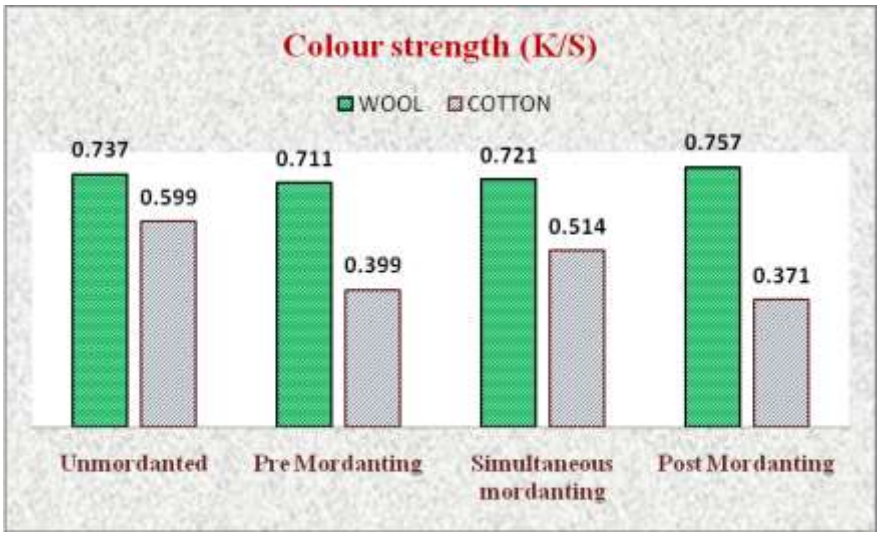
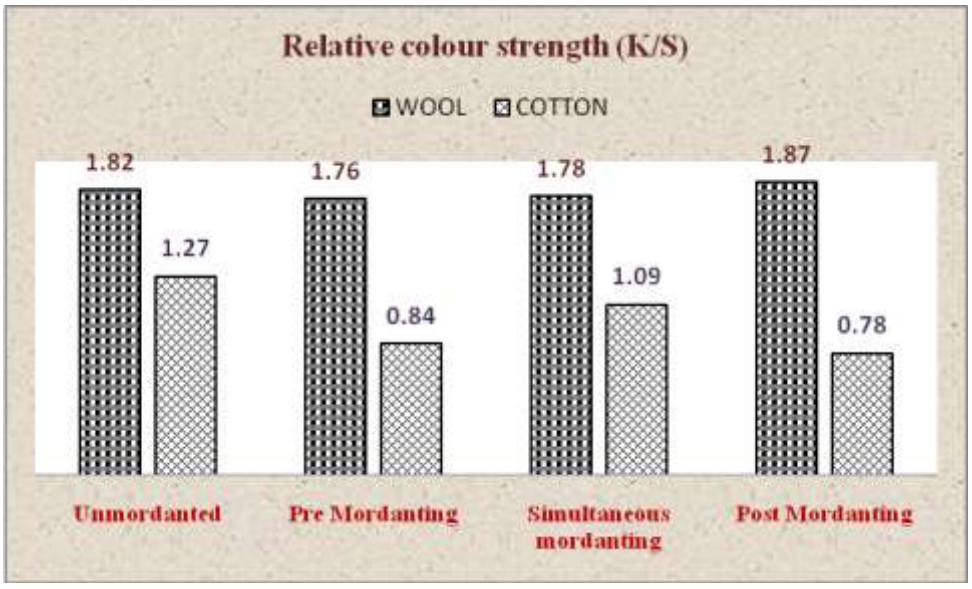


Fig-3: Relative colour strength of dyed fabrics



3.4. COLOUR FASTNESS OF DYED FABRICS

Colour fastness is the resistance of the dyed material to change any of its colour characteristics or extent of transfer of its colourants to adjacent white materials in touch. The performance of the natural dyes and mordants on the dyeing of the fabrics were evaluated by testing the colour fastness of the dyed fabrics. Among all types of colour fastness properties, light fastness, washing fastness and rubbing fastness are considered generally for any textile [12]. The results of colour fastness test were rated visually by comparing the difference in colour or the contrast between the untreated and treated specimens with the differences represented by the grey scale. The colour fastness grade is equal to the grey scale step which is judged to have the same colour or contrast difference [10]. "Table 2" shows the colour fastness values of both in the wool and cotton fabric respectively. Where, both the test fabrics recorded satisfactory fastness grades. In wool fabric mordanted samples recorded higher grades of fastness in washing, light and rubbing fastness respectively which varied ranged from Fair to Good (¾) to Excellent (5) in washing fastness, Excellent in light fastness and Good (4) to Excellent (5) in both fastness tests. In cotton fabric mordanted samples also recorded higher grades which ranged from Fair (3) to Excellent (5) in washing, Good to Excellent (4/5) to Excellent (5) in light and Good (4) to Excellent (5) in rubbing respectively "Table 2". The grades of all the mordanting methods in all the retention tests varied with each other and in each combination. Mordants play a very important role in fastness properties of the fabrics and are used to increase the colour fastness behavior of the natural dyes [12]. The increase in the fastness properties of the dyed fabrics due to mordants may be attributed to increase in size of dye molecules when connected to tannin molecules into the fibre [8]. The lower grade of light fastness implies that metal ligand chealates formed by the mordant are not much resistant to photo degradation as by other mordants having Excellent (5) grades [7].

Table-2: Fastness grades of fabrics dyed with *Juglans regia* L. (leaves) and *Salix alba* L. (ash) adopting different methods.

Method	Washing fastness		Light fastness	Rubbing fastness			
				Staining		Fading	
	CC	CS		Dry	Wet	Dry	Wet
Wool							
Without mordant	4	4/5	5	4/5	4	5	4/5
Pre mordanting	3/4	5	5	5	4/5	5	5
Simultaneous mordanting	4/5	5	5	5	4/5	5	5
Post mordanting	4	5	5	4/5	4/5	5	5
Cotton							
Without mordant	4/5	5	5	5	4/5	5	4/5
Pre mordanting	¾	5	4/5	5	5	5	5
Simultaneous mordanting	3	5	4/5	5	4/5	5	5
Post mordanting	4/5	5	4/5	5	5	5	4/5

CC: Colour change

CS: Colour staining

IV.CONCLUSION

The test materials used as a source of dye and mordant proved to be an efficient and potential dye sources for the application of wool fabric. However, cotton fabric does not show much affinity for the dye and mordant. The dye and mordant applied on wool fabric produced beautiful colours with different shades and tones. However, cotton fabric does not produced beautiful shades. The colour produced on the test fabrics proved to be of excellent colour quality and dye retention with bright and beautiful shades and tones. The colour quality of wool fabric recommends the usage of *Juglans regia* L. (leaves) dye and *Salix alba* L. (wood ash) mordant for industrial applications which can be effectively explored both at national and international level.

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