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Improvement of the traditional system of dyeing on wool fiber using eco-friendly natural dye

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ABSTRACT

The colour fastness properties of the seed coat of Juglans regia, Pinus roxburghii, Butea monosperma and Berberis aristata with with chemical mordants viz. $FeSO_4$, $CuSO_4$ and natural mordants viz. leaves of Symplocos racemosus and Artemisia japonica. Dyeing along with mordanting techniques which included pre-mordanting, simultaneous mordanting and post mordanting was carried out. Study about fastness tests of dyed clothes was undertaken. Large range of shades was obtained because of varying mordant. The washing, rubbing, light and perspiration fastness of the dyed samples was also evaluated, giving fair to excellent fastness grades and this evaluation also useful for textile industries.

Keywords: Wool, Colour fastness, Mordants and Natural dyes.

INTRODUCTION

Natural dyes produce an extraordinary diversity of rich and colors that complement each other. Natural dyes from plants may also have dozens of compounds and their properties vary with soil type and the weather. In 1856, William Perkins accidently synthesized a basic dye, with the advent of synthetic dyes, the use of natural dyes declined tremendously because the existing natural dyes failed to full fill the demand of the market. Natural dyes are permanent than other colorant. In India, Rajasthan and Kutch still possess a rich tradition in the use of natural dyes for textile dyeing. Dyes are one of the most important uses of the plants, as it relates with cultural practices, rituals, arts and crafts, fabrics and to satisfy personal embodiment, however, dye yielding plants have not received significant attention. The widely and commercially used synthetic dyes impart strong colors but causes carcinogenicity and inhibition of benthic photosynthesis [1]. Indians have been considered as forerunners in the art of natural dyeing. Natural dyes find use in the colouring of textiles, drugs, cosmetics, etc. Owing to their non- toxic effects, they are also used for colouring various food products. India has a rich biodiversity and it is not only one of the world's twelve mega diversity countries, but also one of the eight major centers of origin and diversification of domesticated texa. It has approximately 490,000 plant species of which about 17,500 are angiosperms; more than 400 are domesticated crop species and almost an equal number their wild relatives one. Thus, India harbours a wealth of useful germplasm resources and there is no doubt that the plant kingdom is a treasure-house of diverse natural products [2-7]. A close look at the chemical structures of natural dyes

will show that these dyes like synthetic dyes, also consist of vat, acid, basic, disperse, direct and mordant dyes (i.e. dyes capable of forming a complex with metal). For instance, in the case of mordant dyes, there are dyes which have affinity for the fiber; however, their uptake as well as hue can be modified by pre-treatment or post treatment with so called mordants. One such product from nature is the dye. Mordants are metal salts which produce an affinity between the fabric and the dye [8-9]. Alum, chrome, stannous chloride, copper sulphate, ferrous sulphate etc. are the commonly used mordants [10-12]. Use of mordants for colour fastness and also result into variation in dye colour, even same dye can produce different colours using different mordants [12]. *Terminalia chebula* (Harar), *T. bellirica* (Bahera), *Phyllanthus emblica* (Anwala), Tea leaves, Adhatoda, Solanum, Artemisia, Geranium, cow dung, urine, cream of tarter, curd water, ash of wood or bark, rock salt, lemon juice, lime water are some natural mordants.

Though there is a large plant resource base, little has been exploited so far. Due to lack of availability of precise technical knowledge on the extracting and dyeing technique, it has not commercially succeeded like the synthetic dyes. Although indigenous knowledge system has been practiced over the years in the past, the use of natural dyes has diminished over generations due to lack of documentation. Also there is not much information available on databases of either dye-yielding plants or their products. Natural dyes have been used for thousands of years by the people of the border district of Uttarakhand viz, Chamoli, Uttarkashi and Pithoragarh but their knowledge on natural dyes remains unutilized. The tribal community of these high hills has long traditional knowledge on natural dye but the methods of processing of dye on wool is very poor and the mordants are limited because of lack of knowledge on mordants, both natural and chemicals. Mordants are metal salts which produce an affinity between the fabric and the dye[8,11]. Mordants give different shades to the fabric. Similarly, wide range of soft and light colors was obtained on silk using the dye extracted from flower of *Spathadia campanulata* [13]. An Application of Eco-Friendly Natural Dye obtained from *Cordia sebestena* on Cotton using Combination of Mordants with respect to their colour and light fastness properties [13]. The local method of dye processing is time consuming and shades are limited that's why the synthetic dyes is getting more access in local market due to essay availability, with the result that the traditional system of this region is day to day getting down and the people like Bhotia and other tribal community are loosing their own identity. The various ethnic communities, including Bhotiyas of Chamoli District are well known for their traditional expertise in making a range of woolen garments and materials, besides processing and colouring of wool [14]. Before 1962, there was trans-border trade between India and former Tibet, and the import of wool was the major source of income for the Bhotiya's woollens-based indigenous cottage industry [14-16]. But after 1962, trade was stopped due to conflicts between China and India [17]. However, due to the visit of a sizeable number of pilgrims and tourists to the Badrinath and Kedarnath shrines in Garhwal region, the Bhotiya community still has market to sell the woolen products, so that this age-old tradition of preparing woolen products and processes to make them colourful through indigenous methods are alive in the remote area of Garhwal.

The present work envisage that if a low cost and less time consuming improved technique would be provided to local owners then it can uplift the quality of the products and can again lead the traditional market of the state. After adaptation of high-tech product, it will soon gain access to national market and the people could get the actual cost of their products. Moreover, by using the natural dyes instead of synthetic one we can ensure the best uses of the natural resources of the state and can upgrade the economic growth of the region and ultimately the state. Therefore, there is need to upgrade the indigenous method of dyeing and providing proper precautionary measures are considered for sustainability, conservation and value based selection of use pattern.

MATERIALS AND METHODS

Seed coat of *Juglans regia* (Akhrot), *Pinus roxburghii* (Chir), *Butea monosperma* (Dhak), *Berberis aristata* (Kingore), leaves of *Symplocos racemosus* and *Artemisia japonica*) were collected from different parts of Chamoli district. The Plant parts were dried under shade and made into fine powder for extraction techniques of extraction of dyes depend upon the specific part of the plant resource. The extraction

methods depend basically on medium in which the dye is extracted. There are mainly four methods used in extraction of natural dyes.

Aqueous Method: The powdered mass soaked in water for several hours followed with gentle heating. After squeezing and filtration, the whole extract is boiled and concentrated by repeated heating, subject to specific requirement.

Alkaline Method: Prepare 1% alkaline solution (sodium carbonate/sodium hydroxide) and pour dye material into it and boil then filter.

Acidic Method: Prepare 1% of acidic solution (HCl) and mix the dye material, Boil the solution and filter it.

Alcoholic Method: Prepare the solution of dye material in alcohol and boil it then filter.

The white wool samples were free from traces of protein, gums and oil impurities by washing it with a lot of tap water, till it was free from traces to detergents. It was then dried in shade and ironed. Before dyeing and mordanting the wool samples, were soaked for half an hour, the optical density was measured by using digital spectrophotometer. The optimum concentration of dye materiel and time for extraction of dye and dyeing of wool were found out by having varying concentration. For this purpose 2, 4, 6 and 8 gm were taken separately in beakers, each containing 100/150 ml of water for an hour and solution was filtered.

A sample of 2 ml was taken from each beaker and optical density was measured by diluting it at 100 times. Five wool samples of 1 gm each were dyed in the solution for 15 min. at 100^{o}C and the optical density of the left over dye solution was recorded at 420 nm. The optimum time for extraction of dye was found out by extracting the dye material in five beakers containing 100 ml water at 100^{o}C for 15, 30, 45 and 60 minutes. Optical density for each was measured. In order to find the optimum time for dyeing 1gm wool was added to four beakers and dyed for 15, 30, 45 and 60 minutes. The sample was stirred occasionally to obtain an even dyeing. The samples were taken out from the beakers and dried in shade. The evenness of dye, depth of shade and overall appearances are evaluated by panel of judges, the highest percentage rating was calculated by judgment. Mordanting helps the fabric grabs the dye better and improves the light and wash fastness of most dye colors. Three methods were applied for mordanting according to plants properties:

1. Premordanting- In this method the fabric/yarn mordanted in the first stage and then dyed in the second stage. The required amount of mordant was dissolved in 10 ml of boiling water and poured in a beaker containing 100 ml of water. The presoaked and weighed sample was placed in this beaker and the bath was gradually brought to boiling point. The yarn was stirred from time to time and the mordanting was continued for 30 minutes to 1 hr. The samples were allowed to cool in the bath, removed, rinsed and dried in shade.
2. Simultaneous mordanting-In this method the mordant and the dye are applied simultaneously in the same bath. The weighed amount of mordant was dissolved in 10 ml of boiling water and the solution was transferred to a beaker containing 100 ml of dye solution and stirred for a few minutes. The pre-soaked sample was placed in it and the temperature was slowly raised to boiling point and the dyeing and mordanting was carried out for 30 minutes to 1 hr. The samples were cooled in the bath, rinsed and dried in shade.
3. Post mordanting- In this method the fabric/yarn is first dyed and then mordanted. The pre-soaked sample was placed in beaker containing 100 ml of dye solution. The dyeing was carried out for 45 minutes to 1.5 hrs at boiling point. The samples were removed from the dye bath with a glass rod. The weighed amount of mordant was dissolved in 10 ml of boiling water, transferred to bath and mixed thoroughly with the dye solution. The sample were placed in the dye bath and treated for 30 minutes to 1 hr. Finally allowed to cool, rinsed and dried in shade.

Evaluation of Colour Fastness: Colour fastness to washing of the dyed fabric samples was determined as per IS: 764 – 1984 methods using a Sasmira launder-O-meter following IS-3 wash fastness method. The wash fastness rating was assessed using grey scale as per ISO-05-A02 (loss of shade depth) and ISO-105-AO3 (extent of staining) and the same was cross-checked by measuring the loss of depth of colour and

staining using Macbeth 2020 plus computer-aided colour measurement system attached with relevant software. Colour fastness to rubbing (dry and wet) was assessed as per IS: 766-1984 method using a manually operated crock meter and grey scale as per ISO-105-AO3 (extent of staining). Colour fastness to exposure to light was determined as per IS: 2454- 1984 method. The sample was exposed to UV light in a Shirley MBTF Microscale fade-O-meter (having 500 watt Philips mercury bulb tungsten filament lamp simulating day light) along with the eight blue wool standards (BS1006: BOI: 1978). The fading of each sample was observed against the fading of blue wool standards (1-8). Colour fastness to perspiration assessed according to IS 971-1983 composite specimen was prepared by placing the test specimen between two adjacent pieces of wool fabric and stitched all among four sides. The sample was soaked in the test solution (acidic/alkaline) separately with MLR 1:50 for 30 minutes at room temperature. The sample was then placed between two glass plates of perspirometer under load of 4.5kgs (10 lbs). The apparatus was kept in the oven for four hours at 37˚C. At the end of this period the specimen was removed and dried in air at a temperature not exceeding 60˚C. The test samples were graded for change in colour and staining using grey scales.

RESULTS AND DISCUSSION

The evaluation of colour fastness to light, washing, rubbing and perspiration of seed coat of *Juglans regia* (Akhrot) dyed wool samples treated with chemical mordants viz. $FeSO_4$, $CuSO_4$ and natural mordants viz. leaves of *Symplocos racemosus* (lodh) and *Artemisia japonica* (Kingora) is presented in Table-1. Almost all the treated samples subjected to light showed fair to good (3-4 to 4) light fastness for chemical mordant viz. $FeSO_4$, $CuSO_4$ and good (4) light fastness for natural mordants. The washing fastness grades ranged good to very good (4 to 4-5) for chemical mordants and fair to good (3-4 to 4) for natural mordants. There was no colour staining. The colour change to dry and wet rubbing for all the treated samples was very good to excellent (4-5 to 5). There was no colour staining to negligible colour staining (5 to 4-5) in dry rubbing. Most of the treated samples showed excellent fastness grade to colour change in both acidic and alkaline media. There was no color staining (5) for almost all the treated samples in acidic and alkaline media. The simultaneous and post mordanting methods gave best result in comparison of pre mordanting. The different shades of colour obtaining in simultaneous mordanting method are presented in (fig.1)

Mordant	Mordanting Method	Light Fastness Grades	Washing Fastness Grades		Rubbing Fastness			Perspiration Fastness			
					Grades Dry		Grades Wet	Acidic		Alkaline	
			CC	CS	CC	CS	CC	CC	CS	CC	CS
Ferrous sulphate	Pre Mordanting	3-4	4-5	5	4-5	4-5	5	5	4-5	5	4-5
	Simultaneous Mordanting	3-4	4-5	5	5	5	5	5	5	5	4-5
	Post Mordanting	3-4	4	5	4-5	3-4	5	5	5	4-5	4-5
Copper sulphate	Pre Mordanting	4	4	3-4	5	5	5	4	5	4	5
	Simultaneous Mordanting	4	4-5	4	5	5	5	3	5	4	5
	Post Mordanting	4	4-5	4	4-5	5	4-5	4	5	4-5	5
Symplocos racemosus (Leaves0	Pre Mordanting	4	3-4	5	5	4-5	3	4-5	5	3	5
	Simultaneous Mordanting	4	4	5	5	5	5	4	5	4	5
	Post Mordanting	3-4	4	5	5	5	5	4	5	4-5	5
Artemisia japoinica (leaves)	Pre Mordanting	4	3-4	4-5	5	5	5	5	5	4	5
	Simultaneous Mordanting	4	4	4-5	5	4-5	5	5	5	5	5
	Post Mordanting	4	4	4-5	5	5	5	4	5	4-5	5

Table 1: Fastness Grades of seed coat of *Juglans regia* (Akhrot) dyed on wool at optimum dyeing conditions (Wavelength 420 nm. Dye Extraction Time 60 min, Material to liquor ratio 1:20, Dyeing Time 90 min.)

Fig. 1. Seed Coat of *Juglans regia* (Akhrot) and Dyed Wool

The bark of *Pinus roxburghii* (Chir) dyed wool samples treated with mordants viz. $FeSO_4$, $CuSO_4$, leaves of *Symplocos racemosus* and *Artemisia japonica,* the evaluation of colour fastness to light, washing, rubbing and perspiration is presented in Table-2. Almost all the treated samples subjected to light showed good (3-4) light fastness for all mordant. The washing fastness of treated samples ranged good to vary good (4 to 4-5) for chemical mordants and good (4) for natural mordants. There was negligible colour staining. The colour change to dry and wet rubbing for all the treated samples was good to excellent (4-5 to 5). There was no colour staining ranged between no staining to negligible staining (5 to 4-5) in dry rubbing. The perspiration fastness grades ranged between good to excellent (4 to 5) for all samples in both acidic and alkaline media and no colour staining. The simultaneous and post mordanting methods gave best result in comparison of pre mordanting. The different shades of colour obtaining in simultaneous mordanting method are presented in (fig.2)

Mordant	Mordanting Method	Light Fastness Grades	Washing Fastness Grades		Rubbing Fastness			Perspiration Fastness			
					Grades Dry		Grades Wet	Acidic		Alkaline	
			CC	CS	CC	CS	CC	CC	CS	CC	CS
Ferrous sulphate	Pre Mordanting	3-4	4	4-5	5	5	5	5	5	5	4-5
	Simultaneous Mordanting	3-4	4-5	4-5	5	5	5	5	5	5	5
	Post Mordanting	3-4	4-5	4-5	5	4-5	5	5	4-5	5	4-5
Copper sulphate	Pre Mordanting	4	4-5	4	4-5	4	5	4	5	4	5
	Simultaneous Mordanting	4-5	4-5	4	5	5	5	4	5	4	5
	Post Mordanting	4	4	4	5	4	5	4	4-5	4	5
Symplocos racemosus (Leaves0	Pre Mordanting	3-4	4	4-5	4-5	5	3	5	5	3	5
	Simultaneous Mordanting	4	4-5	4-5	5	5	5	5	5	4	5
	Post Mordanting	3-4	4	4-5	5	4-5	5	4	4-5	4	5
Artemisia japoinica (leaves)	Pre Mordanting	3-4	4	4-5	4-5	5	5	5	5	4	5
	Simultaneous Mordanting	3-4	4	4-5	5	5	5	5	5	5	5
	Post Mordanting	3-4	4	4-5	5	4-5	5	4	4-5	4	4-5

Table 2: Fastness Grades of bark of *Pinus roxburghii* (Chir) dyed on wool at optimum dyeing conditions (Wavelength 420 nm. Dye Extraction Time 90 min, Material to liquor ratio 1:20, Dyeing Time 120 min.)

Fig. 2. *Pinus roxburghii* (Chir) and Dyed wool

Butea monosperma (Dhak) flowers dyed wool samples treated with chemical and botanical mordants i.e. $FeSO_4$, $CuSO_4$, leaves of *Symplocos racemosus* and *Artemisia japonica*. All the treated samples were subjected to light showed fair to good (3-4 to 4) light fastness for all mordant. The washing fastness grades showed good to very good (4 to 4-5) for chemical mordants and fair to good (3-4 to 4) for natural mordants.There was no colour staining. The colour change to dry and wet rubbing for all the treated samples was very good to excellent (4-5 to 5). The colour staining ranged between negligible staining to no staining (4-5 to 5) in dry rubbing. Most of the treated samples showed very good fastness grade to colour change in acidic and alkaline media. There was no colour staining (5) for all treated samples in both acidic and alkaline media. Results showed that all mordanting methods gave similar result and different shade produced in simultaneous method are presented in (fig.3)

Mordant	Mordanting Method	Light Fastness Grades	Washing Fastness Grades		Rubbing Fastness			Perspiration Fastness			
					Grades Dry		Grades Wet	Acidic		Alkaline	
			CC	CS	CC	CS	CC	CC	CS	CC	CS
Ferrous sulphate	Pre Mordanting	3-4	4-5	5	4-5	4-5	5	5	4-5	5	4-5
	Simultaneous Mordanting	3-4	4-5	5	5	5	5	5	5	5	4-5
	Post Mordanting	3-4	4	5	4-5	3-4	5	5	5	4-5	4-5
Copper sulphate	Pre Mordanting	4	4	3-4	5	5	5	4	5	4	5
	Simultaneous Mordanting	4	4	4	5	5	5	3	5	4	5
	Post Mordanting	4	4	4	4-5	5	4-5	4	5	4-5	5
Symplocos racemosus (Leaves0	Pre Mordanting	4	3-4	5	5	4-5	3	4-5	5	3	5
	Simultaneous Mordanting	4	4	5	5	5	5	4	5	4	5
	Post Mordanting	3-4	4	5	5	5	5	4	5	4-5	5
Artemisia japoinica (leaves)	Pre Mordanting	4	3-4	4-5	5	5	5	5	5	4	5
	Simultaneous Mordanting	4	4	4-5	5	4-5	5	5	5	5	5
	Post Mordanting	4	4	4-5	5	5	5	4	5	4-5	5

Table 3: Fastness Grades of flowers Butea monosperma (Dhak) dyed on wool at optimum dyeing conditions (Wavelength 420 nm. Dye Extraction Time 60 min, Material to liquor ratio 1:20, Dyeing Time 120 min.)

Fig. 3. *Butea monosperma* (Dhak) and Dyed wool

In case of heartwood (root and stem) of *Berberis aristata* (Kingore) dyed wool sample treated with different mordants ($FeSO_4$, $CuSO_4$, leaves of *Symplocos racemosus* and *Artemisia japonica*. The dyed samples were subjected to light showed fair to good (3-4 to 4) light fastness for all ratio mordant. The washing fastness grades ranged between good to very good (4 to 4-5) for all the treated samples. There was negligible colour staining. The colour change to dry and wet rubbing for all the treated samples was very good to excellent (4-5 to 5) for all mordants. There was negligible to no staining (4-5 to 5). The treated samples showed good to excellent (4 to 5) fastness grade to colour change in both acidic and alkaline media. Overall there was no clour staining (5) for all treated samples in both acidic and alkaline media. A number of shades were obtained by simultaneous mordanting the wool with different mordants (Fig.4)

Mordant	Mordanting Method	Light Fastness Grades	Washing Fastness Grades		Rubbing Fastness			Perspiration Fastness			
					Grades Dry		Grades Wet	Acidic		Alkaline	
			CC	CS	CC	CS	CC	CC	CS	CC	CS
Ferrous sulphate	Pre Mordanting	3-4	4	4-5	5	5	5	5	5	5	4-5
	Simultaneous Mordanting	3-4	4-5	4-5	5	5	5	5	5	5	5
	Post Mordanting	3-4	4-5	4-5	5	4-5	5	5	4-5	5	4-5
Copper sulphate	Pre Mordanting	4	4-5	4	4-5	4	5	4	5	4	5
	Simultaneous Mordanting	4-5	4-5	4	5	5	5	4	5	4	5
	Post Mordanting	4	4	4	5	4	5	4	4-5	4	5
Symplocos racemosus (Leaves0	Pre Mordanting	3-4	4	4-5	4-5	5	3	5	5	3	5
	Simultaneous Mordanting	4	4-5	4-5	5	5	5	5	5	4	5
	Post Mordanting	3-4	4	4-5	5	4-5	5	4	4-5	4	5
Artemisia japoinica (leaves)	Pre Mordanting	3-4	4	4-5	4-5	5	5	5	5	4	5
	Simultaneous Mordanting	3-4	4	4-5	5	5	5	5	5	5	5
	Post Mordanting	3-4	4	4-5	5	4-5	5	4	4-5	4	4-5

Table 4: Fastness Grades of heartwood (root and stem) of *Berberis aristata* (Kingore) dyed on wool at optimum dyeing conditions (Wavelength 420 nm. Dye Extraction Time 90 min, Material to liquor ratio 1:20, Dyeing Time 50 min.)

Fig. 4- *Berberis aristata (*Kingora) with dyed samples

APPLICATIONS

These results concluded that the seed coat of *Juglans regia* (Akhrot), *Pinus roxburghii* (Chir), *Butea monosperma* (Dhak) and *Berberis aristata* (Kingore) can be successfully used for dyeing of wool to obtain a wide range of soft and light colours by using natural (botanical) as well as chemical mordants. These results give us different data for evaluation of fastness properties under consideration and which are helpful for textile industries.

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