Extraction and dyeing of eco-friendly natural dye from Lodh bark on wool fabrics and optimization of procedure for dyeing using herbal mordants

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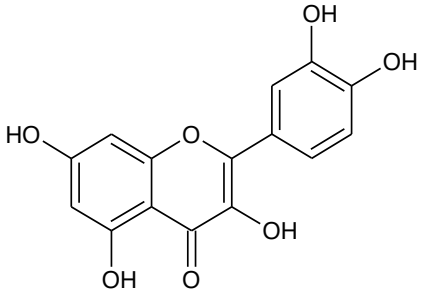
Abstract- The current study deals with the soaked wool fabrics were dyed with natural dyes extracted from the bark of *Symplocos racemosa*. The optimization was carried out for the different variables; concentration of dyematerial, time for extraction of dye, dyeing time, concentration of mordants methodsof mordanting. Out of the three methods of mordanting, the best shades for dye was obtained using pre-mordanting with lemon juice and vinegar andpost-mordanting with aqueous leaves extract of *Rhus parviflora* and aqueous leaves extract of *Erythrina suberosa* . The colour fastness tests indicated that all samples change in colour on exposure to light. The light coloured samples were affected more rapidly as compared to the dark ones. The Lodh bark dyed samples exhibited fairly excellent fastness to light and the results of washing fastness tests showed that dyed samples had good to excellent fastness to washing. **Keywords:** *Symplocos racemosa* (Lodh), *Erythrina suberosa*, *Rhus parviflora*, Wool fabrics, Fastness, Herbal mordants, Natural dye.

Introduction

The art of dyeing with natural dyes[2,3,4] is very ancient[20]. There is very much evidence to indicate that dyeing had been practised in India[17], China and Persia, as far back as 2500 B. C. It is believed that some of the earliest traces of dyed textiles are found. Due to its eco-friendly environmental nature, it creates non-toxic environment and these plant pigments[5] have been used for dyeing textile, wool and fibers across the world by different human societies. In fact, natural dyes have been synonymous with traditional Indian textiles like the Kalamkari and Madhubani paintings and Pashmina shawls. In District Chamoli, Uttarkashi and Pithoragarh of Uttarakhand, traditional wool[11,12,15,16] and woolen products were still used for dyeing by the local villagers but there are certain problems with the use of natural dyes in

textile dyeing[7,8,9,10] viz., colour yield, complexibility of dying process, reproducibility results, limited shades, blending problems and inadequate fastness properties. 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 in format ion available on data bases of either dye-yielding plants or their products. Mordants either metallic salts or herbal mordants like extraction of leaves, roots, bark and flowers etc. may be used which produce an affinity between the fabric and the dye. Extract[6] of various plants, vinegar and lemon juices are the commonly used natural or herbal mordants. Colour fastness is the resistance of a material to change any of its colour characteristics or extent of transfer of its colorants to adjacent white materials in touch. The natural dyes[19] present in plants and animals are pigmentary molecules which impart colour to the materials. There are several plants that provide natural dyes[13,14,18] which are used in the textile industry. However, the common drawbacks of natural dyes are their non-reproducible and non-uniform shades, poor to moderate colour fastness and lack of scientific information on the chemistry of dyeing and standardized dyeing methods. Many reports are available on application of natural dyes on wool. *Symplocos racemosa* species is very rare to give natural dyes properties and this species give us good fastness grades with respect to grey scale. Isolation of natural dyes from these species under optimization[1,21] could not be done till now because recent data of the literature did not show these results of natural dyes properties in the past. The present study has been undertaken so as to revive the age-old are of dyeing with natural dyes. The present investigation deals with the aqueous extraction of natural dyes from the bark of *Symplocos racemosa* grow in almost all cold and dense parts of Garhwal Himalaya in Uttarakhand, India. The aim of present work has been carried out to prepare eco-friendly natural dyes the bark of *Symplocos racemosa* and then apply them on wool fabrics. The main aim of study is to get a cheap and easily available natural colourant for textile industry as well as to alter colour fastness of wool fabric sunder optimized condition and also to visualize the effect of metallic mordants have been undertaken. The structures of pre-isolated major colourant from this species have been mentioned below, **Figure-1**.



Quercetin

Figure-1 Chemical structures of pre-isolated dye bearing yellow colourant from the bark of *Symplocos racemosa*

Material and Methods

Collection of plant materials

Lodh bark were collected from the forests of District Chamoli of Uttarakhand, were dried in shade and crushed into fine powder form.

Wool- Wool in bulk was purchased from the tribal community of Gopeshwar, District Chamoli of Uttarakhand.

Mordants used as herbal mordants

The following non-toxic herbal mordants as aqueous extract of some plants materials like leaves of *Rhus parviflora* and flowers of *Erythrina suberosa*, vinegar and lemon juices were used as herbal mordants for the study.

Instruments used to analysis

The colour strength values of un-irradiated and irradiated dyed fabrics were investigated by CIE lab system using the Spectra flash (SF 660) at Chemistry Division, FRI, Dehradun, India. 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 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 Microsal fade-O-meter (having 600 watt Philips mercury bulb tungsten filament lamp simulating day light) along with the eight blue cotton 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 as well as Grey scale was used for determining respective shades of colour of the dyed materials.

Scouring of wool

Wool yarn contain much grease and other impurities which affect the dye take-up by the fabrics, therefore it is desirable to give a thorough scouring treatment to the yarns, before being used for dyeing or mordanting. The scouring was done with a detergent solution prepared by mixing 0.5 of genteel with 100 ml of very hot water. The skeins were immersed in the prepared solution, after it had cooled to lukewarm temperature (40-50 ^{0}C). They were stirred with a wooden spoon for 30 minutes. The skeins were then removed, rinsed with lots of warm water again treated as above 3-4 and squeezing, till completely free of detergent. Care was taken not to scrub or mangle the skeins.

Preparation of skeins

The skeins were prepared so as to allow even preparation of the mordants and dye into each fiber. One gram of yarn was weighed and wrapped around a 10” cardboard piece. The resulting skein was removed and tied loosely at two places.

Optimization of different variables used in dyeing

The different variables like the concentration of dye material, time for extraction of the dye, dyeing time and the concentration of the mordants were optimized. The material-liquor ratio selected was 1:100. The optical density (O.D.) of the dye solution, before and after dyeing was recorded and the percent absorption was calculated by the following

formula:

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

Concentration of dyeing material

The dye materials, in four different concentrations (2g, 4g, 6g, and 8g) were taken in the beakers containing 100 ml water. The extraction of the dye was done for 1 hour in boiling water and the solution was filtered. A sample of 2 ml was taken from each beaker and the optical density was recorded by diluting it 100 times. Four wool samples of 1g each were dyed in the solution for 1 hour at 100 ^{0}C and the optical density of the left over dye solution was recorded like as above. The results are given in the **Figure-2**.

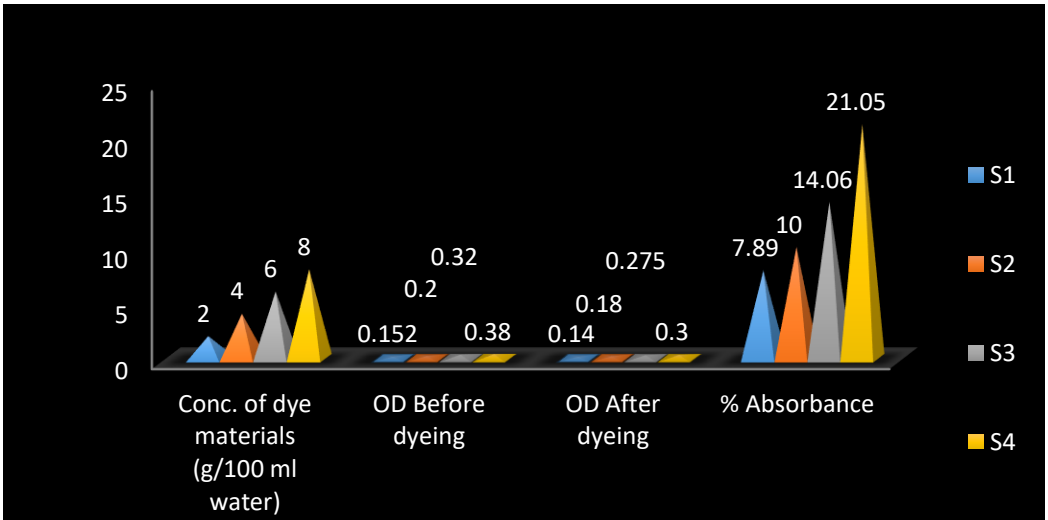


Figure 2. Percent absorption for different concentrations of Lodh bark dye

Time for extraction of the dye

Four beakers containing 100 ml of water were taken and the required amount of the dye material was put in each. The extraction of the dye was carried out at 100 ^{0}C for 15 minutes, 30 minutes, 45 minutes and 60 minutes. A sample of 2 ml was taken from each beaker and the optical density was measured. Four wool samples of 1g each were dyed in the dye bath and the optical density of the left over solution was recorded **Figure-3**.

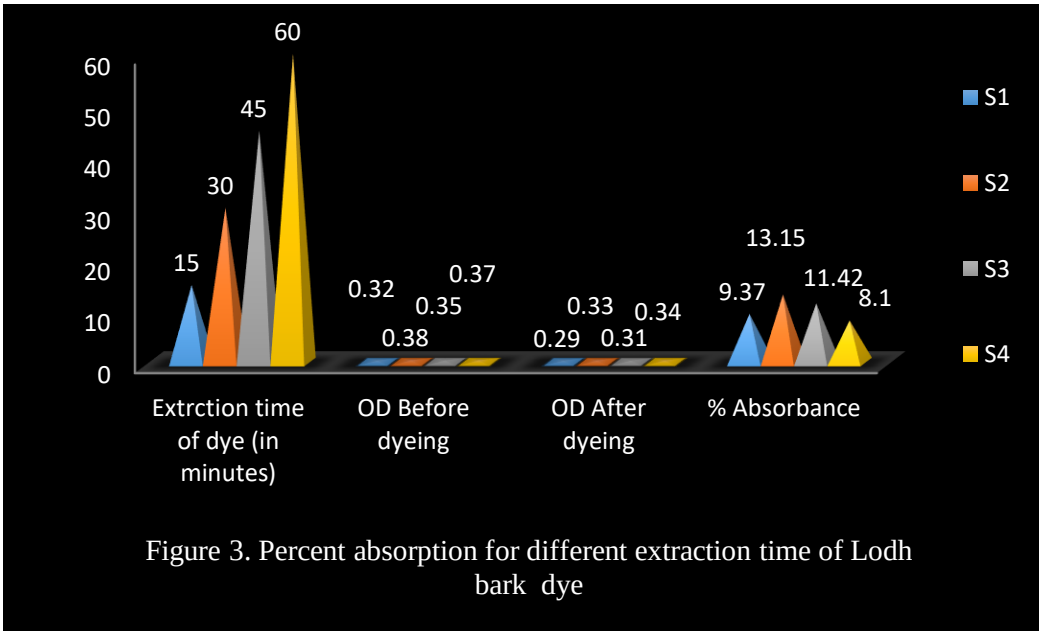


Figure 3. Percent absorption for different extraction time of Lodh bark dye

Dyeing time

The required amount of the dye material was put in four beakers containing 100 ml of water and the dye extraction was carried out at boiling temperature for the optimum time. The solution was filtered and a sample of 2 ml was taken from each beaker and the optical density was recorded. Wool samples of 1g each were put in the four beakers and dyeing was carried out at 100 ^{0}C for 15 minutes, 30 minutes, 45 minutes and 60 minutes. The optical density of each solution was recorded again **Figure-4**.

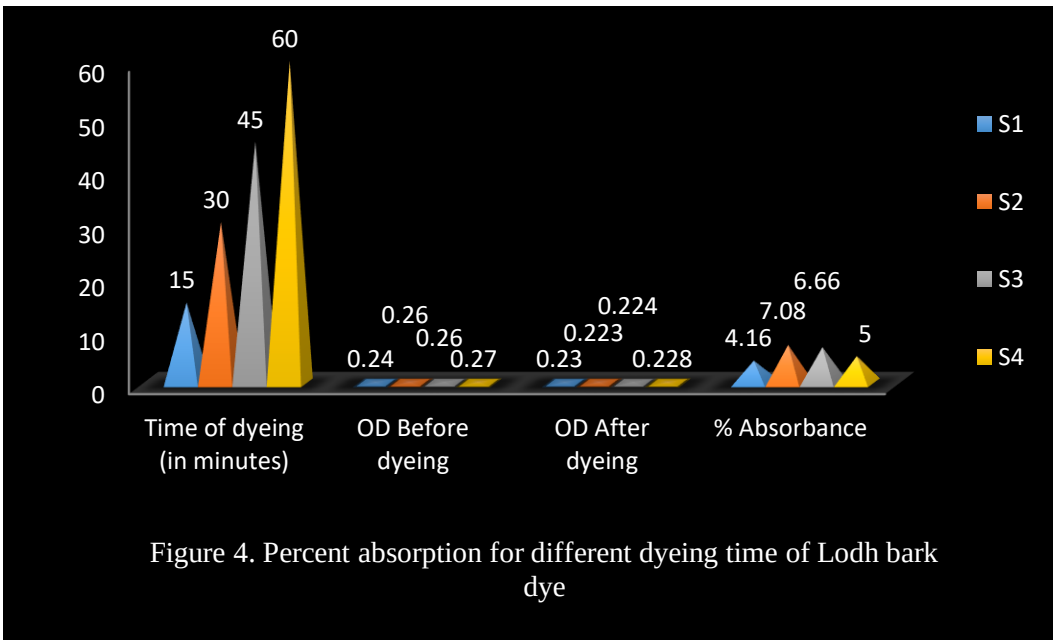


Figure 4. Percent absorption for different dyeing time of Lodh bark dye

Mordants

Organic dyes by their nature are fugitive and agents are therefore required to fix the dye molecules in the fabrics. These fixing agents are known as mordants. The mordant has a direct effect on the dye and governs the colour obtained, so that the same dye produces different colours when used with different mordants. The

following mordants were selected for the present study: leaves extract of *Rhus parviflora* and flowers extract of *Erythrina suberosa*, vinegar and lemon juice.

Concentration of mordants

The concentrations of all the mordants were taken 5 ml/100g of wool. The mordants in the above concentrations were put in beakers containing 100 ml of dye solution. After dissolving the mordant, 1g of wool was placed in each beaker and the dyeing and mordanting was carried out simultaneously at 100 ^{0}C for the optimum time. The percentages of ratings were calculated on the basis of the total marks obtained by each sample **Table-1**.

Table -1 Optimum concentration of different mordants for Lodh bark dye

S. No.	Mordants used	Conc. of Mordants (ml/100g of wool)	Percentage of rating
1.	Lemon Juice (LJ)	5	57.67
2.	Vinegar (V)	5	55.48
3.	Extract of *Rhus parviflora* (ERP)	5	58.45
4.	Extract of *Erythrina suberosa* (EES)	5	60.30

Preparation of dye liquor

Lodh bark dye: The required amount of dyes powder were put in beakers separately containing 100 ml of water and the extraction was carried out at 100 ^{0}C for one hour.

Preparation of blank samples

The weighed samples of wool yarns were soaked in water for 1-2 hours and added to the dye liquor prepared as above. The temperature of the bath was raised to boiling and the dyeing continued for 3/2 hours. The samples were allowed to cool in the bath, rinsed under tap water and dried in shade.

Methods of mordanting used

There are three methods of mordanting, out of which the optimum method was selected for each mordant, on the basis of visual evaluation, by a panel of judges. These are:

a) Pre-mordanting
b) Simultaneous mordanting and dyeing
c) Post-mordanting

Pre-mordanting

In this method, the required amount of each herbal mordants as 5 ml was poured in a beaker containing 150 ml of water. The pre-soaked and weighed samples were

placed in this beaker and the bath was gradually brought to boiling point. The yarns were stirred from time to time and the mordanting was continued for one hour. The samples were allowed to cool in the bath, removed, rinsed and dried in shade. The mordanted samples were dyed according to the methods prescribed for blank (3.7). The samples mordanted with non-toxic mordants as aqueous extract of leaves of *Rhus parviflora* and flowers of *Erythrina suberosa*, vinegar and lemon juice were dyed immediately after mordanting. Mordanting with aqueous extract of leaves of *Rhus parviflora* is very sensitive to light and hence special care was taken to ensure that the dye bath was well covered.

Simultaneous mordanting and dyeing

5 ml of each herbal mordant were used and the solution was transferred to a beaker containing 150 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 1:30 hours. The samples were cooled in the bath, rinsed and dried in shade.

Post-mordanting

The pre-soaked samples were placed in beakers containing 150 ml of dye solution. The dyeing was carried out for 1:30 hours at boiling point. The samples were removed from the dye bath with a glass rod. 5 ml of each herbal mordant were transferred to the bath and mixed thoroughly with the dye solution. The samples were replaced in the dye bath and treated for one hour. They were allowed to cool in the dye bath, rinsed and dried in shade **Table-2**.

Table-2 Optimum method of mordanting with Lodh bark dye

S. No.	Mordants used	Mordanting methods	Percentage of rating
1.	Lemon Juice (LJ)	Pre-mordanting	63.26*
		Simultaneous mordanting	48.75
		Post mordanting	57.00
2.	Vinegar (V)	Pre-mordanting	55.56
		Simultaneous mordanting	47.00
		Post mordanting	62.88*
3.	Extract of *Rhus parviflora* (ERP)	Pre-mordanting	62.00*
		Simultaneous mordanting	51.25
		Post mordanting	50.45
4.	Extract of *Erythrina suberosa* (EES)	Pre-mordanting	45.20
		Simultaneous mordanting	55.15
		Post mordanting	68.50*

*** Best method of mordanting**

Preparation of final samples

The final samples for lodh bark dye was prepared by using the optimum concentration of dye material, optimum extraction time, optimum dyeing time, optimum concentration of herbal mordants and the optimum method of mordanting. These samples were used for preparing the sample sheets and for conducting the colour fastness tests.

Testing colour fastness of dyed samples

The final sample of lodh bark dye was subjected to colour fastness tests:

Test for colour fastness to light

To carry out this test, the dyed samples of 3 x 6 cm were mounted on a cardboard frame along with blue standards rated 1-8 respectively. The cardboard frame was covered with a black sheet in such a way that all samples were half exposed and half

covered. This frame was placed inside the fadeometer fitted with mercury bulb tungeston fluorescent lamp (MBTF) and faded as per the ISO recommendation. The standards and specimens were checked after every few hours till the fading was equivalent to grade 3 on the grey scale. The samples were compared with the blue standards and rated **Table-3.**

Test for colour fastness to washing

For this test the yarn was made into a sheet form of parallel length measuring 10 x 4 cm and placed between two pieces of undyed fabrics (of the same size), one of which was wool. All the three layers were sewn from all sides. The washing solution was prepared by dissolving 5 ml of detergent with 1 liter of water and the test was carried out according to the ISO recommended test No. 2, in the standard washing machine (launderometer). Each sample was treated for 45 minutes at 50 ± 2^0C, using soap solution in the ratio of 50:1. The samples were rinsed for 10 minutes in running water and dried in shade. The samples were assessed on the basis of change in colour of the samples as well as staining of the adjacent fabrics with the help of geometric grey scale **Table-3.**

Table-3 Colour fastness of samples dyed with Lodh bark dye

S. No.	Samples (Mordanted and Non-mordanted)	Rating for Washing fastness	Rating for staining	Rating for light fastness
			Wool samples	
1.	Control	4-5	4	4-5
2.	Lemon Juice (LJ)	5	4-5	4
3.	Vinegar (V)	4	4-5	4
4.	Extract of *Rhus parviflora* (ERP)	4-5	5	5
5.	Extract of *Erythrina suberosa* (EES)	4-5	4	5

Results and Discussion

The preliminary experiments showed that the bark of *Symplocos racemosa* yields a wide range of colours on wool fabrics. Dyed and mordanted samples are revealing about dyed samples with lodh bark dye with different colour shades and so detailed experiments were conducted to standardize the methods of extraction and applications of the dyes on wool fabrics.

The results obtained by these tests are reported and discussed below.

Optimization of different variables

To optimize the dyeing procedure includes:

Concentration of dye materials, Time for extraction of the dye, Dyeing time concentration of mordants and Methods of mordanting.

Optimum concentration of dye materials

Four concentrations 2%, 4%, 6% and 8%, each of lodh bark dye was taken and the optical density (O.D.) of the dye liquor was recorded. The results obtained are reported in Figure-2 from which, it is evident that the percent absorption of lodh bark dye increased with increase in the concentration of dye up to 8% , on visual inspection of wool samples dyed in the above concentrations, it was observed that best colour obtained when 8% dye solution was used (21.05). Hence this concentration was selected as optimum for further

experiments. 8% dye solution is equivalent to 8gm of dye /100 ml of water/gm of wool.

Optimum time for extraction of dye

Crushed powder form of lodh bark was subjected to different periods of boiling and optical density of dye liquor for each period was recorded [Figure-3] which shows that maximum percent absorption (13.15), was obtained when lodh barks were boiled for 30 minutes, after which it steadily decreased as boiling was continued for 60 minutes (8.10). Wool samples gave best colour when the dye was extracted for 30 minutes and hence 30 minutes was selected as the optimum time for extracting lodh bark dye. Cavendish (1978) has reported that extraction time of natural dyes varies above 20 minutes. The observations of the present study are in accordance with those observations.

Optimum dyeing time

Wool samples were dyed for different periods of lodh bark dye solutions, optical densities for before and after dyeing were recorded. The results are given in Figure-4. It is clear from the Table-3 that initially percent absorption increased with the same time for dyeing wool samples, after some time it was decreased on continuation for the same time. Hence the best time for lodh bark dye was 30 minutes with maximum percent absorption as 7.08.

Optimum concentration of mordants

Wool samples with the same concentrations of mordants were used with lodh bark was visually evaluated by a panel of judges. There optimum concentrations were same but despite of that, samples revealed different percent of rating with different dyed and mordanted samples. There are four types of mordants were used as Lemon Juice (LJ), Vinegar (V), Extract of *Rhus parviflora* (ERP) and Extract of *Erythrina suberosa* (EES), among these four mordants Extract of *Erythrina suberosa* (EES) mordanted samples gave good percent of rating for lodh bark dye (60.30) and all above results are mentioned in the Table-1 respectively.

Optimum method for mordanting

The wool samples were mordanted with the three methods of mordanting: Pre-mordanting, Simultaneous mordanting and Post mordanting. Out of these the optimum method was selected for each mordant, on the basis of visual evaluation as in Table-2. It is clear from the Table-2, Lemon Juice (LJ) and extract of *Rhus parviflora* (ERP) were taken as mordants and applied with three mordanting methods like as above in which pre-mordanting method revealed maximum percent of rating, 63.26 and 62.00, which was the best mordanting method for lodh bark dye. Simultaneous mordanting with 48.75 and 51.25 and Post mordanting with 57.00 and 50.45 have low percent of rating than Pre-mordanting for the same. Secondly, Vinegar (V)and Extract of *Erythrina suberosa* (EES) were taken as mordants and applied with three mordanting methods in which maximum percent of rating was recorded in post mordanting, 62.88 and 68.50, rest two mordanting methods have less values than post-mordanting for the same.

Colour fastness of the dyed samples

Colour fastness to light

The rating of colour fastness to light was done with the help of geometric gray scale and the blue standards, by comparing the contrast between the exposed and unexposed position of the dyed samples. The results are reported in Table-3. It was observed that dyed samples with lodh bark

dye as in Table-3, were mordanted with Lemon Juice (LJ) and Vinegar (V) showed fairly good fastness (4) while blank, Extract of *Rhus parviflora* (ERP) and Extract of *Erythrina suberosa* (EES) mordanted dyed samples showed excellent fastness to light (5).

Colour fastness to washing

The rating of washing fastness was done on the basis of change in colour of the samples as well as staining of adjacent fabrics as wool. On visual inspection with the help of gray scale, it was found that in case of lodh bark dye, the washing fastness was excellent for blank, Extract of *Rhus parviflora* (ERP), Lemon Juice (LJ) and Extract of *Erythrina suberosa* (EES) mordanted samples and while washing fastness was good for Vinegar (V). It was observed that for blank, there was almost slightly staining of adjacent fabrics for wool while there was almost negligible staining of adjacent fabrics as mentioned in Table-3.

Conclusion

The present study was undertaken to develop the dyeing process for dyeing wool fabrics with natural dyes obtained from bark of *S. racemosa* (lodh). The optimization was carried out for the different variables; concentration of dye material, time for extraction of dye, dyeing time, concentration of mordants methods of mordanting. It was concluded that 8% solutions of lodh bark dye gave the best results. Thus, it was selected as optimum concentration for dyeing 1g of wool samples. The optimum time required for maximum extraction of bark of *S. racemosa* (lodh) was found 30 minutes. The dyeing of wool samples for 45minutes yielded bright and beautiful shades for bark of *S. racemosa* (lodh) and there are concentrations of different mordants, which were same and it was found that 5 ml of extract of *Erythrina suberosa* (EES)/100 g for Tung leaf dye gave the best results. Thus, the above concentrations were selected as optimum for lodh bark dye. In the mode of mordant application, it was observed that when the method of mordanting was varied, there was a marked difference in the shades obtained. Out of the three methods of mordanting, the best shades for lodh bark dye was obtained using pre-mordanting with Lemon Juice (LJ) and extract of *Rhus parviflora* (ERP) and post-mordanting with Vinegar (V) and extract of *Erythrina suberosa* (EES). The colour fastness tests indicated that all samples change in colour on exposure to light. The light coloured samples were affected more rapidly as compared to the dark ones. The lodh bark dyed samples exhibited fairly good to good fastness to light and the results of washing fastness tests showed that lodh bark dyed samples had good to excellent fastness to washing. It may be concluded that these natural dyes can be used for dyeing wool on a small scale and may encourage rural women and youths to start a cottage industry based on the above methods. The beautiful shades can be used to satisfy the artistic and creative urge of modern textile designers and can readily find a place in the colour schemes of today.

Disclaimer Statement

Authors declare that no competing interest exists. The products used for this research are commonly used products in research. There is no conflict of interest between authors and producers of the product.

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