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EXTRACTION OF NATURAL DYES FROM LEAVES OF *MYRICA ESCULENTA* AND DYEING OF WOOL AND COTTON FIBERS USING DIFFERENT MORDANTS : DETERMINATION OF OPTIMUM DYEING CONDITIONS AND ASSESSMENT OF COLOUR FASTNESS PROPERTIES

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Abstract

As the synthetic dye is associated with various negative eco-toxicological effects which is a serious global environmental concern, a need has been felt to explore the new dye-yielding plants. *Myrica esculenta* has proved to be a good dye yielding plant and wide range of color shades was obtained when different mordants were used with different mordanting methods. Bark of this plant was extracted with water under different optimized conditions such as extraction time, extraction temperature, pH, etc. and its extract then applied for dyeing wool and cotton fabric using different mordants, *viz.* Potassium Aluminium Sulphate, Ferrous Sulphate, Stannous Chloride and Tannic Acid. The washing, rubbing and light fastness properties of dyed samples were assessed and good to excellent fastness grades were obtained.

Key words: *Myrica esculenta,* natural dye, optimization, mordants, fastness properties, colour strength, K/S values.

Introduction

Color is one of the elements of nature that made the human living more aesthetic and fascinating in the world. The art of dyeing is as old as human civilization (Upadhyay and Choudhary, 2012). The age old art of dyeing with natural dyes was common in India, China, Egypt and Central Asia (Sinha Keka *et al.,* 2012). Natural colorants are dyes and pigmentary molecules that are obtained from plants, animals or mineral sources with or without chemical treatments (Kamel MM *et al.,* 2005; Wealth of India., 1952).

Dyeing with natural resources is an oldest technique and even today in most of the regions, despite the availability of synthetic dyes, many people continue to use plant extracts to colour fabrics, masks or food (Kundal Jyoti *et al.,* 2016). Natural dyes have also better biodegradability and generally have higher compatibility with the environment. They are non-toxic, non-allergic to skin, non-carcinogenic, easily available and renewable (Samanta AK, Agarwal P., 2009 and Adeel S, *et al.,* 2009).

The invention of the first synthetic dye by William Henry Perkin in 1856 changed the situation and later, the synthetic dyes received faster acceptability due to a wide range of applications in various fields like food (Fossen T. *et al.,* 1998), cosmetic (C.D. Calnan 1976), photodynamic therapy (L. Gao and X. Qian 2002), non-linear optical activity (A.K. Sinha *et al.,* 1995), more importantly in textile industries due to ease in dyeing and overall cost factor (P. Savarino *et al.,* 1999; P. Kongkachuichay *et al.,* 2002). However, during the last few decades, due to an increased environmental awareness the use of synthetic dyes is gradually decreasing as most of the synthetic dyes are toxic, allergic to skin, carcinogenic, non-renewable etc. As a result, recently a ban has been imposed all over the world including European Economic Community (EEC), Germany, USA and India on the use of some synthetic dyes. (Jyoti Kundal *et al.,* 2016). Again natural dye came into existence due to its eco-friendly nature and human need.

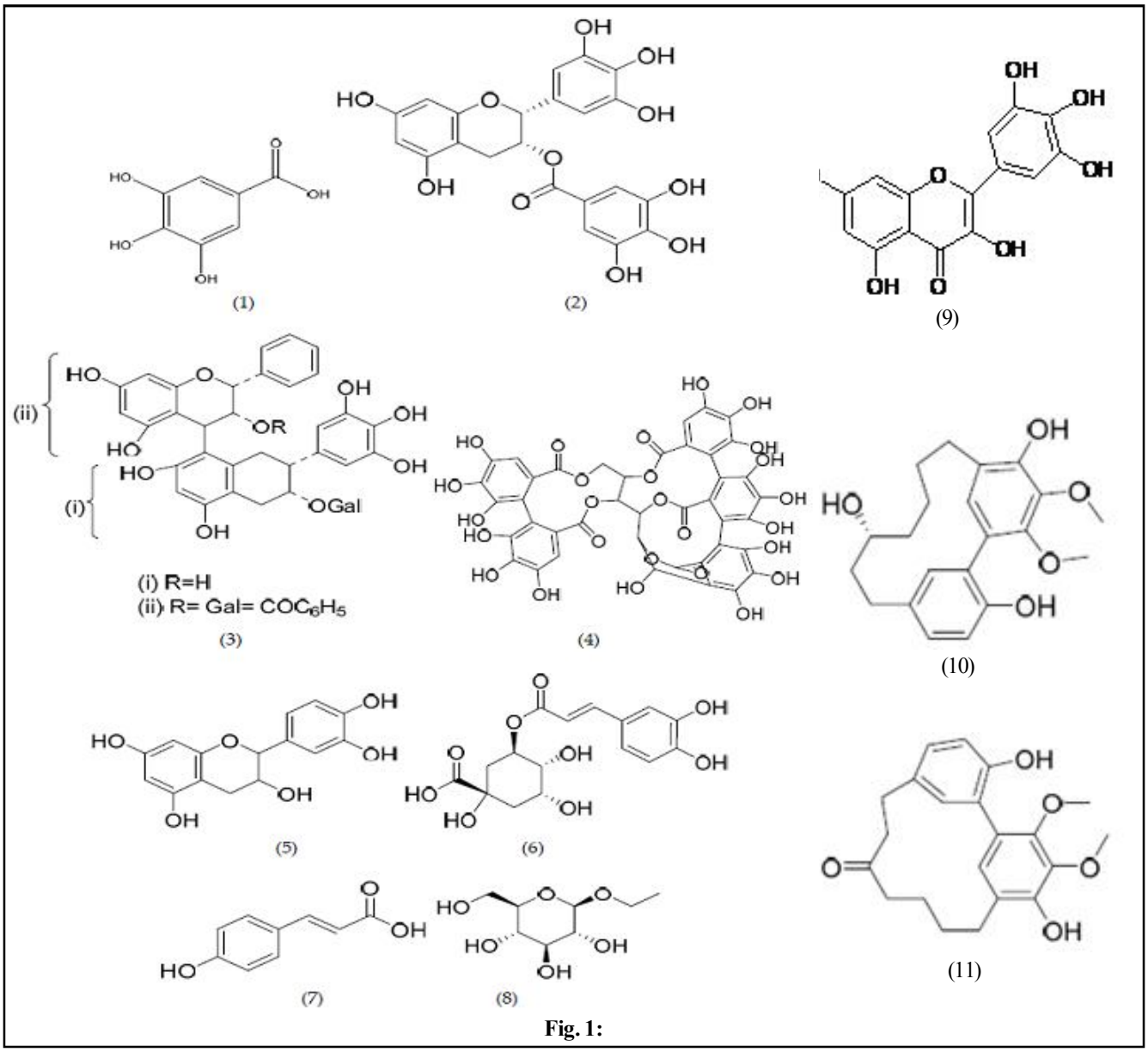


Fig. 1:

The present study deals with the dyeing of cotton and wool fabric with the bark extract (water extract) of *Myrica esculenta* along with some synthetic mordants which overcome the limitations associated with natural dye like limitations of shades and colours. *Myrica* is a genus of about 35-50 species of small trees and shrubs in the family Myricaceae, The genus is distributed widely from Africa, Asia, Europe, North America, South America to India (Gaur R.D, 1999). The barks are used in Vietnamese folk medicine to treat catarrhal fever, cough, sore throat, and skin disease (Bich D.H *et al.,* 2004). Some bioactive isolated compounds from different parts of M. esculenta are: (1) Gallic acid. (2) Epigallocatechin 3-O-gallate, (3) i) epigallocatechin- (4β→8)-epigallocatechin 3-O-gallate, ii) 3-O-galloyl-epigallocatechin -(4β→8) -epigalloc-atechin 3-O-gallate, (4) Castalagin, (D. Sun *et al.,* 1988; N. Singh, *et al.,* 2009). (5) Catechin, (6) Chlorogenic acid, (7) p-coumaric acid, (S. Rawat *et al.,* 2011). (8) Myricetin, (P. Panthari *et al.,* 2012), (9) Myricanol, (10) Myricanone (V. Krishnamoorthy and T.R. Seshadri 2001). Fig. 1.

Materials and Methods

Material

Bark of the plant *Myrica esculenta* was collected from forest region of Pauri Garhwal Uttarakhand (India). It was then dried under shade and crushed into fine powder. Fig. 2

Extraction of Dye

The powdered material was extracted with water at different optimized conditions.

Fig. 2:

Optimization of various parameters before dyeing

To reduce the extra efforts, extra time and wastage of resources certain parameters were optimized before dyeing *viz.* optimum pH, optimum concentration, optimum time of extraction and optimum time of dyeing. All these parameters were optimized by proper methods of optimization and their values are shown below in table 1.

Table 1:

S. No.	Parameter	Value for cotton	Value for Wool
1	Optimum pH	8-9	6-7
2	Optimum concentration	10 gm /100ml	10 gm /100ml
3	Optimum time of extraction	45 minutes	45 minutes
4	Optimum Time of dyeing	40 minutes	40 minutes

Preparation of Dye Solution

Aqueous dye solution was prepared by adding 5 gm of powder in 100 ml of water. The extraction was done at 100°C for one hour at 6-7 pH. The hot solution was filtered and a clear solution was obtained which was used for dyeing wool fabric. Same extraction was done for cotton at 8-9 pH.

Mordants used

Three synthetic mordants and one natural mordant were used for mordanting *viz*. Potassium Aluminium sulphate, Stannous Chloride, Ferrous sulphate and Tannic acid.

Mordanting methods used

Pre-mordanting

Fabric first treated with mordant for 30 minutes at 60°C. Wool and cotton fabric dyed by the standard method prescribed for natural dyes. The process of dyeing is carried out at 1:30 MLR (material to liquor ratio) for 40 min at 80°C using Rota-Dyeing Machine. Since we know that synthetic mordants are very toxic and their excessive use can create an environmental issue, therefore, only 1% of mordant used which is a permissible limit.

Post-mordanting

In this method both the fabrics dyed first by the method used above and rinsed in hot and then cold water and finally treated with mordant solution for 30 minutes at 60°C and rinsed.

Simultaneous-mordanting

1 ml of 1% mordant mixed with 30 ml of dye extract and then 1 gm of fabric dyed with it for 40 minutes at 60°C.

Different fastness properties: Dyed fabrics then subjected to different fastness tests *viz.* light, washing and rubbing to optimize the best shade and best method of dyeing.

Test for color fastness to light:

This test carried out as per IS: 2454-1984 method. The dyed samples of 3 cm × 6 cm cloths fixed on a black cardboard in such a way that all samples were half exposed and half covered. This frame then placed inside the fadeometer fitted with mercury bulb tungsten lamp (MBTF). The specimens brought out after 17 hours and color fading assessed against the blue wool standards (BS 1006: BOI: 1978).

Test for color fastness to washing using Launderometer (TC/ UICT/TEQIP):

Color fastnesses to washing of the dyed samples determined as per IS: 764-1984 method. In this test the different dyed fabrics were cut into pieces of 10 cm × 4 cm and placed between two pieces of undyed fabrics (wool and cotton) of same size. All three layers were sewn from all sides. This is said to be the *'sandwich type of arrangement'* of fabrics. The washing was done by 5% non-ionic soap solution (cell det (R)) for 60 minutes using Washing fastness tester (Launderometer) using 50 steel balls. The samples then rinsed in running water and dried in shade and then assessed with the help of grey scale (ISO: 05-A02) and (ISO- 105-A02) for loss of shade and extent of staining.

Test for color fastness to rubbing:

This test was done as per IS: 766-1984 method using Crock meter in which a piece of dyed cotton is rubbed 10 times on a sample with the help of crock meter. Two types of rubbing is done - *wet rubbing* and *dry rubbing*. The staining on the wet and dry piece of fabric was evaluated with the help of grey scale as per ISO-105-A03.

K/S values (Determination of surface color strength) (Dye ability of dyed fabric)

The K/S value of the dyed and undyed wool and

Table 2: Rating of Fastness to washing for cotton and wool (*M. esculenta*).

S. No.	Mordant used	Mord-anting Method	Color change (Wool)	Color change (Cotton)	Staining (cotton)		Staining (Wool)	
					Cotton	Wool	Cotton	Wool
1.	Pot. Alum.	Pre	4/5	4/5	4	5	4/5	4/5
	sulphate	Post	4/5	4/5	5	4/5	4	4/5
		Sim.	4/5	4	4/5	4	4	4/5
2.	Tannic acid	Pre	4	4	4	5	4	4/5
		Post	4/5	4/5	5	4/5	4	4/5
		Sim.	4	4	4/5	4	4/5	4/5
3.	Ferrous	Pre	4	4	5	4	4	4/5
	sulphate	Post	5	4	5	4/5	4	4/5
		Sim.	4	4/5	4/5	4/5	4/5	4/5
4.	Stannous	Pre	4	4/5	5	5	4/5	4/5
	chloride	Post	5	4	5	4/5	4	4/5
		Sim.	4/5	4/5	4/5	4/5	4/5	4

cotton fabrics was determined by measuring surface reflectance of the sample using a computer aided Mcbeth 2020 plus reflectance spectrophotometer using the following Kubelka-Munk equation :

$$K/S = \frac{(1 - R_{\lambda max})^2}{2R_{\lambda max}}$$

Where K is the coefficient of absorption, S is the coefficient of scattering ; $R_{\lambda max}$ is the surface reflectance value of the sample at particular wavelength when maximum absorption occurs for a particular dye / colorant. The K/S value of dyed yarn is directly proportional to the amount of dye present in the material and it has been also found that generally K/S value increases with mordants.

Results and Discussion

The preliminary experiments showed that the bark extract of *Myrica esculenta* yielded a wide range of colors. So a detailed study was conducted to standardize the methods of extraction and application of dyes on cotton and wool yarn. The results obtained are discussed below.

Fastness tests

Color fastness to washing

The samples were assessed on the basis of change in color and staining on the adjacent fabrics with the help of grey scale. The rating is given in table 2 on the basis of which it has been found that in cotton, good to very good (4 to 4/5) fastness grading ranges were obtained with four mordants. In case of wool, a very good (4/5) grading was obtained with Potassium aluminium sulphate, good to very good (4 to 4/5) with Tannic acid and good to excellent (4 to 5) with Stannous Chloride and $FeSO_4$ table 2.

Color fastness to rubbing

On the basis of results it is concluded that for Cotton fabric, negligible to non- staining (4/5 to 5) grading is obtained with about all the mordants in case of dry rubbing,

Table 3: Rating of Fastness to Rubbing for Cotton and Wool.

S. No.	Mordant used	Mord-anting Method	Staining (Cotton)		Staining (Wool)	
			Dry	Wet	Dry	Wet
1.	Pot. Alum. sulphate	Pre	5	4	4	3
		Post	5	4	4/5	4
		Sim.	4/5	3/4	4/5	3
2.	Tannic acid	Pre	5	4	4	3
		Post	5	4/5	4/5	4
		Sim.	4/5	4	4	¾
3.	Ferrous sulphate	Pre	4/5	3/4	4	¾
		Post	4/5	3/4	4/5	¾
		Sim.	5	4	4/5	¾
4.	Stannous chloride	Pre	4/5	3/4	4	¾
		Post	4/5	4	4	¾
		Sim.	5	3/4	5	4

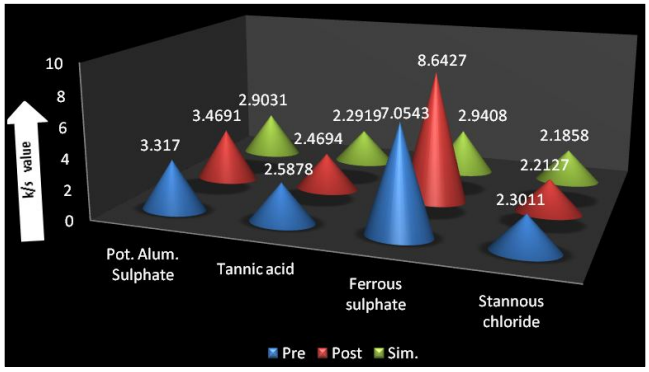


Fig. 4: Graphical representation of K/S values of dyed wool samples.

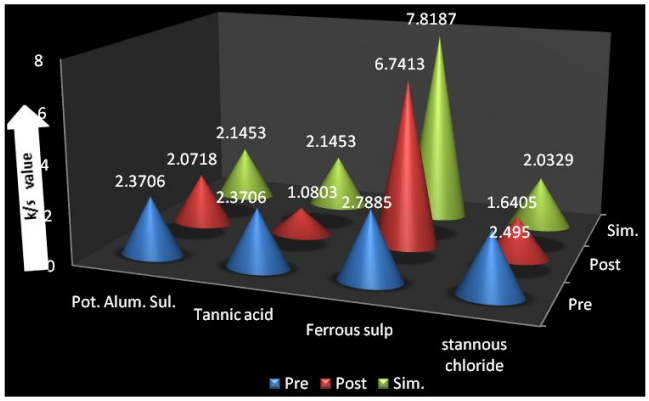


Fig 3: Graphical representation of K/S Values of dyed cotton samples.

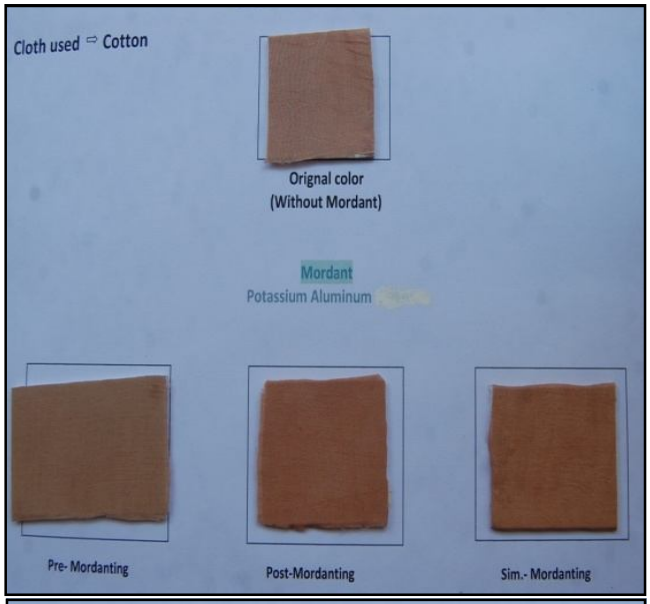
in case of wet rubbing, noticeable to slight (3/4 to 4) grading with Potassium Aluminium Sulphate $FeSO_4$ Stannous Chloride and negligible to non- staining (4/5 to 5) grading with Tannic Acid is obtained. For Wool in case of dry rubbing all the four mordants give a slight to negligible (4 to 4/5) grading. In case of wet rubbing noticeable to slight (3 to 4) grading is obtained with Potassium Aluminium Sulphate, Tannic Acid and Stannous while noticeable (3/4) grading is obtained with $FeSO_4$ table 3.

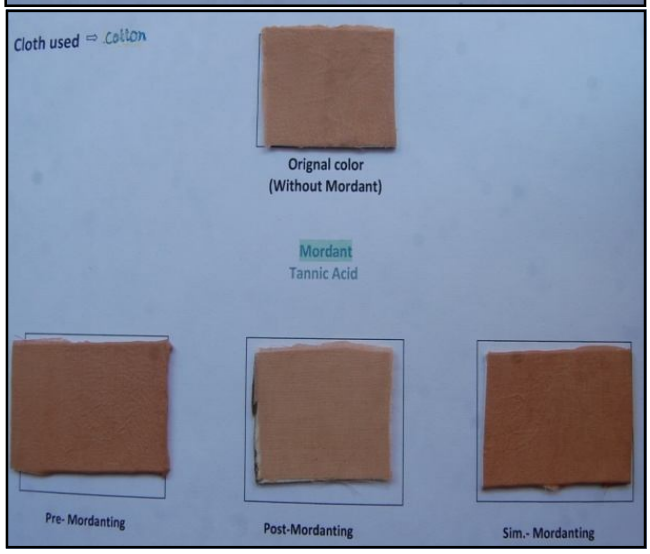
Color fastness to light

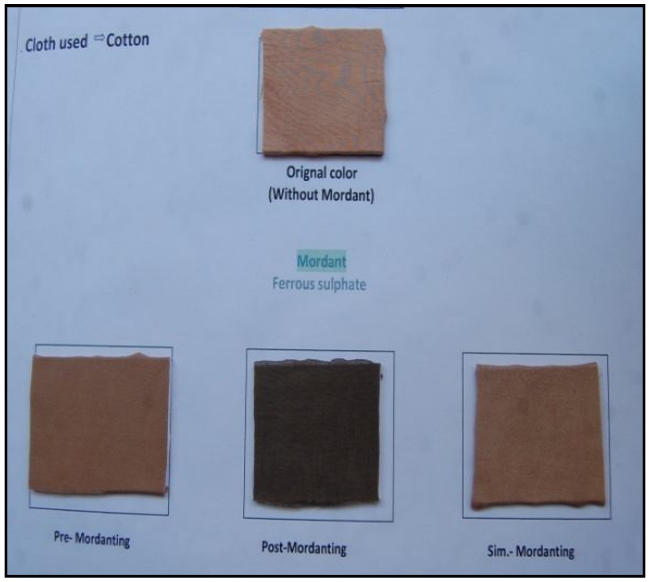
Light is also a very important factor which affects the color. Generally, color gets faded in sunlight, so this test gives very important information as to whether the color applied on fabric is stable to light or not. To perform this test dyed samples were placed in light for more than 15 hours continuously. Results obtained are very good which are given below in the Table 4 which reveals that for both cotton and wool good to very good (5 to 6) results are obtained with Potassium Aluminium Sulphate, Tannic Acid and Stannous Chloride whereas good to excellent

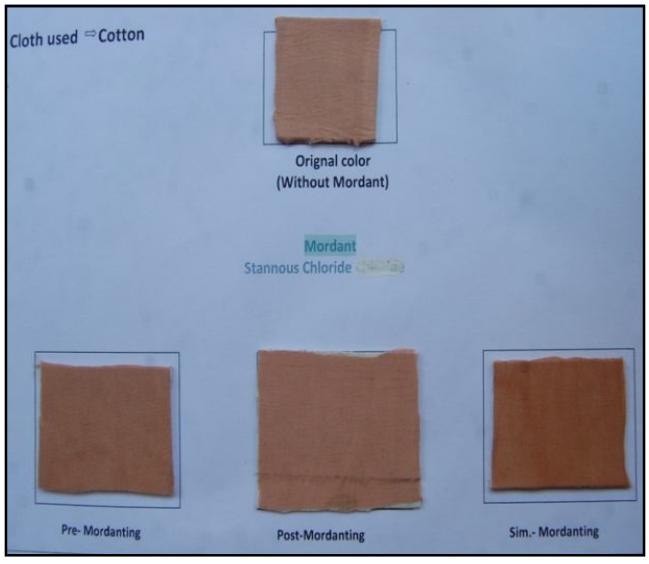
Table 4: Rating of Fastness to Light for Cotton and Wool.

S. No.	Mordant used	Mordanting method	Rating values	
			Cotton	Wool
1.	Pot. Alum. sulphate	Pre	5	6
		Post	5/6	5/6
		Sim.	5/6	5/6
2.	Tannic acid	Pre	6	6
		Post	5/6	6/7
		Sim.	5	5/6
3.	Ferrous sulphate	Pre	5/6	6
		Post	7	7
		Sim.	6/7	6
4.	Stannous chloride	Pre	5/6	6
		Post	5/6	6/7
		Sim.	5/6	6









(5/6 to 7) in case of $FeSO_4$.

***Dye-ability test* (*K/S values*)**

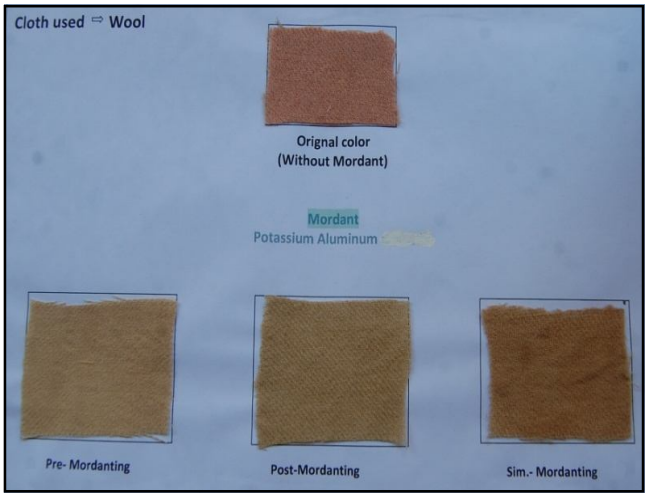
The color strength of dyed fabric was assessed by using computer color matching machine. K/S values of all the dyed samples were determined which are given below table 5. It is seen that maximum color is absorbed in case of Ferrous Sulphate in Sim. mordanting method whereas minimum absorption is obtained with Tannic acid in Post mordanting.

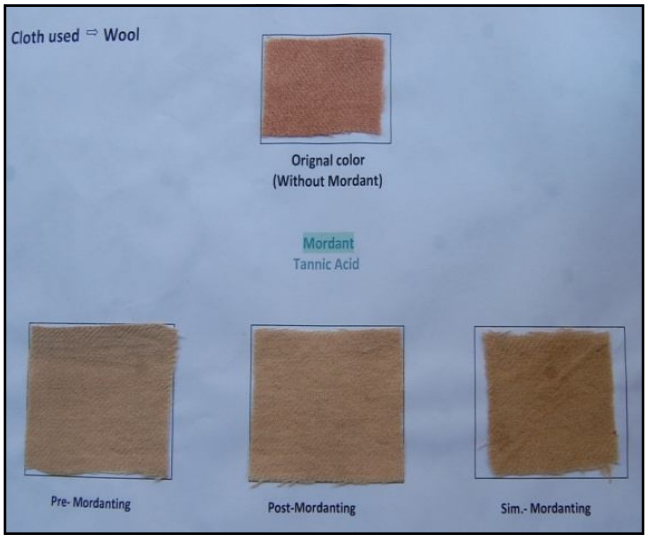
Table 5: K/S Values of dyed cotton and wool samples.

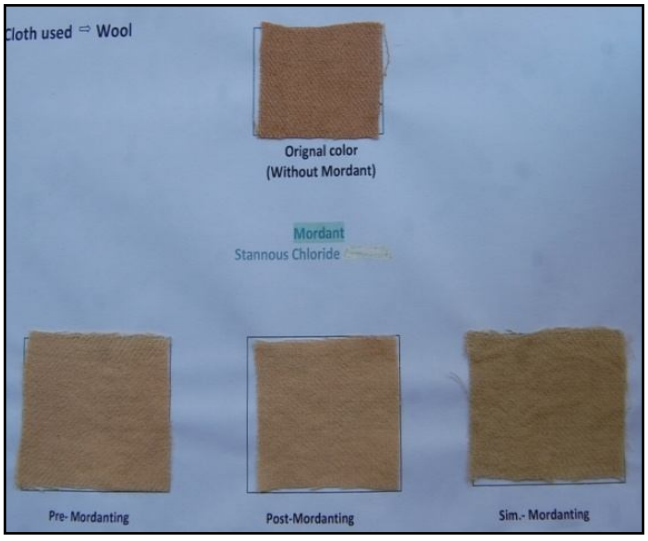
S. No.	Mordant Used	Mordanting method	K/S Values Cotton	K/S Values Wool
1.	Pot. Alum. Sulphate	Pre	2.3706	3.3170
		Post	2.0718	3.4691
		Sim.	2.1453	2.9031
2.	Tannic acid	Pre	2.3706	2.5878
		Post	1.0803	2.4694
		Sim.	2.1453	2.2919
3.	Ferrous sulphate	Pre	2.7885	7.0543
		Post	6.7413	8.6427
		Sim.	7.8187	2.9408
4.	Stannous chloride	Pre	2.495	2.3011
		Post	1.6405	2.2127
		Sim.	2.0329	2.1858

Conclusion

Myrica esculenta is a plant which is locally known as Kaphal or Kafal. It is a wild tree which is found in Garhwal Himalaya at an elevation of 1400-2000 m. The bark of the plant is rich in colour- imparting compounds. Thus, the selected plant-part offers a good source of natural dyes and can be comfortably employed for dyeing cotton and woolen samples. These are quite fast towards light and washing and mordanting the dyed cotton and woolen samples enhances the shade and improves the washing fastness. These results can help in the best utilization of natural resources.







Photographs of dyed samples

Abbreviations

Sim. Simultaneous, Pot. Potassium, Alu. Aluminum, Sul. Sulphate

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