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Research Paper

Natural dye from traditional medicinal source: mulethi

■ ANKITA SHARMA, ARCHANA SINGH AND SUMAYAL ANJUM

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See end of the paper for authors' affiliations

ANKITA SHARMA
Department of Textiles and Clothing, College of Home Science, Chandra Shekhar Azad University of Agriculture and Technology, KANPUR (U.P.) INDIA

■ **ABSTRACT :** With the growing importance and popularity of concepts like green, eco-friendly, sustainability, etc. natural dyes seems to have become an obvious choice for enlightened producers and consumers of textiles. There is a great demand for natural dyed products in foreign countries as well as in India. Cotton fabric dyed with natural dyes in soft and subdued shades with traditional designs creates a fashion statement. In this view the present research work has been taken on standardization of dyeing process for cotton with natural dye extracted from mulethi (*Glycyrrhiza glabra*).

■ **KEY WORDS:** Natural dyeing, Mulethi, Cotton, Mordant

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The importance of colour in textile has been recognized from thousand of years; ancient writing contains frequent references for it. India has had the distinction of being a colour box of the ancient world in the past. Natural dyes are excellent for their soft lustrous shade and aromatic smell, soothing to human eyes and these dyes are also very good source of experimentation on natural fibre such as cotton, linen, silk, wool. Man has tried to get colours from various natural sources such as plant, animal, insects, vegetables etc. and has eventually learnt to use them on various textile materials.

The natural dyes, which are non pollutant, appear as an ideal choice due to their non-toxic nature and easy handling. These natural colourants have several advantages over synthetic dyes from health, safety end ecology point of view. There is increased awareness and demand of natural dyed products in the international market. Taking above all the facts in view, present study has been attempted to optimize various dyeing variables for dyeing of cotton fabric with dye extracted from mulethi.

■ RESEARCH METHODS

Selection of fabric and pre-treatment :

Cotton fabric, procured from local market of Kanpur Nagar was selected for the study. Before dyeing the fabric was soaked overnight in luke warm detergent solution containing 0.5 ml of genteel per 100 ml of water to remove starch. The samples were washed, rinsed and ironed, when slightly wet.

Selection of natural dye :

Dried mulethi (*Glycyrrhiza glabra*) was collected and used for conducting dye experiments. It is basically a herb which is easily available in local market. It is used in many ayurvedic medicine for cold and cough.

Selection of natural mordants :

The three natural mordants *viz.,* harad, bamboo bark and tea leaves were taken in five concentration of 2, 3, 4, 6 and 8 owf. They were soaked separately in 100 ml of water and filtered.

Optimization of variables for dyeing :

A series of experiments were conducted to optimize various variables of dyeing like the medium of dye extraction, concentration of dye material, time for extraction of dye, dyeing time and concentration of mordants. The optical density (O.D.) of the dye solution was recorded both before and after dyeing and per cent absorption was calculated with the help of E.C. Double Beam UV-VIS spectrophotometer using following formula:

$$\text{Per cent absorption} = \frac{\text{O.D. before dyeing} - \text{O.D. after dyeing}}{\text{O.D. before dyeing}}$$

Selection of medium for dye rxtraction :

Dye solutions were prepared by extracting 2g. of dye stuff in 100 ml of each acidic, alkaline and aqueous medium. The dye material was entered to each medium *i.e.* acidic, alkaline and aqueous solution and boiled at 80°C for 60 minutes. After that the solutions were filtered, pre soaked cotton samples of known weight were then added to each beaker and dyeing was carried out for 60 minutes. The samples were then allowed to cool at room temperature, taken out, rinsed in water dried in shade and ironed. The medium which gave the best shade was selected for the study. Based on maximum per cent absorption, concentration of extraction medium was optimized.

Selection of wave length :

The dye extract was subjected to ultra violet and visual light of wave length using, E.C. Double Beam UV-VIS spectrophotometer. The wave length at which maximum per cent absorption was obtained was taken as the suitable wave length.

Optimization of concentration of dye material :

Five dye solutions were prepared by boiling 2 g, 4 g, 6 g, 8 g, 10 g of mulethi (dye material) in 100 ml of water at 80°C for 60 minutes. The solutions were cooled and filtered. One ml dye solution was pipetted out from each beaker and then volume was made up to 100 ml by addition of water. Then the optical density of each of the five solutions was recorded. Two gram of cotton fabrics, pre soaked in water, were dyed in each beaker separately for 60 minutes. While dyeing the temperature was raised to boiling and then allowed to simmer at 80°C for 60 minutes with continuous stirring. Dyed samples were washed and dried in shade and ironed when half wet. Optical density of all the five dye solutions was again recorded using the same procedure as mentioned before. The concentration, which showed the highest per cent absorption was selected as optimum concentration of dye and used for further study.

Optimization of extraction time :

The optimum concentration of dye material was added to five beakers separately containing 100 ml of water. The extraction of the dye was carried out at 80°C for 30 min., 45 min., 60 min., 75 min. and 90 min., respectively. The dye solutions were then cooled and filtered. One ml dye solution was pipetted out from each beaker and then volume was made upto 100 ml by addition of water. The optical density of the solutions were recorded. Two grams of the pre soaked cotton samples were added to each beaker and dyed at 80°C for 60 minutes. The dyed samples were removed from dye liquor, dried in shade and ironed. The optical density of the dye solutions before and after dyeing was recorded. On the basis of per cent absorption, the best time for extraction of dye was optimized.

Optimization of dyeing time :

Dye solution of mulethi with optimized concentration and extraction time was prepared. Presoaked cotton samples of 2 g were dyed in these solution for 30, 45, 60, 75 and 90 minutes, respectively at 80° C. Dyed samples were removed from the dye bath solution, rinsed in tap water, dried in shade and ironed. Optical density was measured before and after dyeing. Dyeing time was optimized on the basis of per cent absorption.

Optimization of concentration of natural mordants :

Harad, Bamboo Bark and Tea leaves were taken as natural mordants with five concentrations of 2g, 3g, 4g, 6g and 8g. Each of the mordants were soaked overnight with their respective concentrations in 100 ml of water and filtered after heating for 15minutes. Dye solution of mulethi was prepared and all the three methods of mordanting *i.e.* pre, simultaneous and post mordantings were done to dye 2g of pre- soaked sample. After dyeing samples were rinsed under tap water, dried in shade and ironed when half wet. Out of the above mentioned concentrations one of the best concentration of each of the mordant was selected for optimizing the mordant concentration on the basis of per cent dye absorption.

Visual evaluation of the samples :

To select the best concentration of each mordant, dyed samples were evaluated visually by a panel of 15 judges. The evaluation was based on the luster, depth of shade obtained, evenness of the dye and overall appearance of the samples. Marks were calculated for each samples and converted into percentage ratings. The sample which got the highest percentage rating was selected as the best.

■ RESEARCH FINDINGS AND DISCUSSION

The results obtained from the present investigation as well as relevant discussion have been summarized under following heads :

Medium for dye extraction :

Among the three medium for dye exrtraction *i.e.*, acidic, aqueous and alkaline medium experimented, mulethi showed best results with acidic medium. Thus acidic medium was selected as extraction medium for dye.

Concentration of extraction medium :

Mulethi was extracted in acidic medium using 0.2 to 1.0 ml HCl/100 ml of water. Cotton fabric was dyed in each extracted solution and per cent absorption was calculated. The results

are reported in Table 1. It is observed from the table that the dye extracted in 1.0 ml HCl concentration gave best shade as compared to other concentrations, thus was selected as optimum.

Table 1 : Optimization of acid concentration (extraction medium) for dye extraction

Acid concentration (ml/100 ml of water)	Per cent absorption
0.2	30.10
0.4	32.76
0.6	32.64
0.8	31.85
1.0	*37.88

*Maximum per cent absorption

Wave length :

Wave length of the extracted dye solution was calculated using double beam UV-VIS spectrophoto metre and it was found that maximum per cent absorption was observed at 660 nm.

Concentration of dye material :

Different concentrations of dye material were taken to optimize the concentration of dye. Cotton samples were dyed for five different dye material concentrations *i.e.* 2 g, 4 g, 6 g, 8 g and 10 g. The per cent absorptions were calculated on the basis of optical density.The results are reported in Fig. 1. This shows that out of five dye concentrations (2, 4, 6, 8 and 10 g) tried, maximum per cent absorption, was obtained with 6g dye material/ 100 ml of water/2 g of cotton sample. Thus it was selected as optimum.

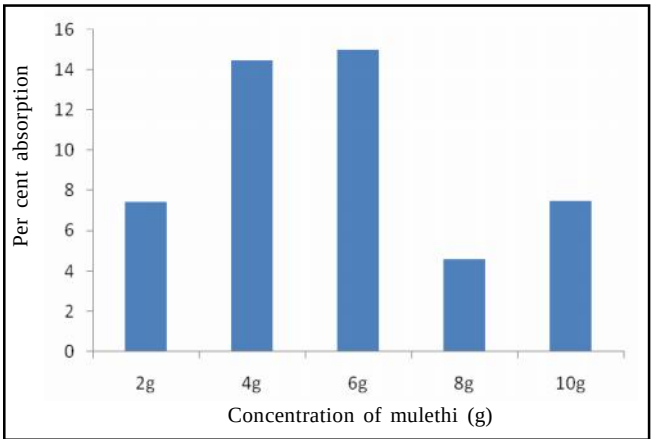

Fig. 1 : Per cent absorption by cotton fabric dyed with different concentration of mulethi

Time for extraction of dye :

The mulethi was subjected to different periods of boiling for extraction of the dye. Cotton samples were dyed with each extracted solution and per cent absorption was calculated for each samples. It is depicted in Table 2 that the percentage absorption of mulethi dye increases from 30 to 60 minutes extraction time and after that marginal decline in per cent absorption was observed from 75 to 90 minutes. Therefore it is clear from the data that per cent absorption was highest for 60 minutes of extraction time and therefore it was selected as optimum.

Table 2: Percentage absorption by cotton fabric dyed with Mulethi for different extraction time

λ-max (nm)	Extraction times (minutes)	**O.D. before dyeing	O.D. after dyeing	Per cent absorption
660	30	0.744	0.618	16.94
	45	1.517	1.310	13.64
	60	0.825	0.523	36.60*
	75	0.906	0.763	15.79
	90	1.679	1.408	16.15

*Maximum per cent absorption ** Optical density

Dyeing time :

Cotton samples were dyed for 30, 45, 60, 75 and 90 minutes with optimized concentration of mulethi *i.e.* 6 g. The results of per cent absorption are given in Fig. 2 which reveals that maximum per cent absorption (39.89) on cotton was observed with 60 min dyeing time, hence, it was selected as optimum.

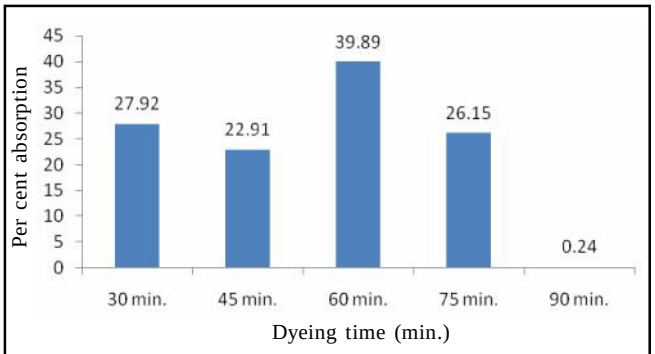

Fig. 2 : Per cent absorption by cotton fabric for different dyeing time

Concentration of mordants :

The cotton samples were dyed with all the three natural mordants (Harad, Bamboo Bark, Tea leaves) in different concentrations, using simultaneous method of mordantings and dyeing. The samples were visually evaluated for selection of the best concentration of each mordants. The results are shown in Table 3 which depicts that 4 g concentration of all three natural mordants showed highest marks.

The use of mordant facilitated overall fair to good colour fastness *i.e.* washing, light, perspiration and crocking as compared to blank sample.

Table 3 : Percentage of marks given to cotton fabric dyed with mulethi using different concentration of natural mordants

Concentration of natural mordants (g)	Percentage of marks obtained		
	Bamboo bark	Harad	Tea leaves
2	64.5	52	60.2
3	72.3	70.5	68.5
4	87.3*	92*	82*
6	69.5	73.6	71.6
8	72.3	74	70.5

*Maximum per cent absorption

Mordanting method :

Cotton samples were dyed with all the three methods of mordanting ie; pre, simultaneous and post mordanting with the selected concentrations of each mordant (bamboo bark, harad, tea leaves). It was observed that all the mordants showed different shades on cotton with three methods of mordanting.

In case of bamboo bark, all three methods of mordanting ie; pre, simultaneous and post mordanting produced different shades such as brown, brownish cream and grayish brown, respectively. Though the colours obtained with blank dye solution (without mordant) was found to be more bright, the use of mordant facilitated overall good colour fastness *i.e.*, washing, light, perspiration and crocking as compared to blank sample.

Samples mordanted with harad showed mustard yellow in pre and simultaneous mordanting and dyeing methods, and very light yellowish brown in post mordanting method. Tea leaves produced grayish brown in pre mordanting,yellowish brown in simultaneous and mud colour in post mordanting. Similar work related to present topic was also done by Mahanta and Tiwari (2005); Gulrajani (2001); Siva (2003) Krishnamurthy *et al.* (2002); Singh *et al.* (2005) and Hussein *et al.* (1997).

Final recipe for dyeing of Cotton sample (2g)

Dye concentration : 6 g
Extraction Medium : Acidic (HCl)
Concentration of HCl : 1.0 ml
Extraction Time : 60 minutes
Dyeing Time : 60 minutes

Mordants' concentration

Bamboo bark : 4 owf
Harad : 4 owf
Tea Leaves : 4 owf

Conclusion :

The present study was planned to look out for safer alternative for dyeing with natural dyes. Mulethi was found to be a good source of natural dye for dyeing of cotton. Experimental results indicated that various shades of yellow and brown can be produced from a single source depending on the types of mordant used. Mixing of one or more natural mordants can be done along with their suitable methods of mordanting to obtain new colours.Therefore mulethi could be a potential source for cellulose textile substrates and also satisfy the demand of green minded consumers.

Authors' affiliations:
ARCHANA SINGH AND SUMAYAL ANJUM, Department of Textiles and Clothing, College of Home Science, C.S.A. University of Agriculture and Technology, KANPUR (U.P.) INDIA

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