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Eco-dyeing of silk with Dhavdi flowers and its fastness evaluation

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Abstract :The present study is based on the process development for colouring of silk yarns with Dhavdi flowers using selected mordants to study its effects on colourfastness properties and optimization of different extraction conditions, duration and concentration for better dye and its shades. The results revealed that aqueous medium was suitable for extraction of dye from pink petals of Dhavdi flowers for one hour. The shades of midbuff, sand stone ,coffee and olive green brown were obtained by taking 4 g dried powder of dhavdi dye for dyeing 1 gram of silk material for 45 minutes. All the four mordants were found suitable for application on silk, 10 % of alum, 3 % of chrome, 2 % of copper sulphate and 3% ferrous sulphate were found to produce best shades on silk material. Post mordanting had resulted in darker shades in all the mordant. Simultaneous mordanting method was found best in case of chrome mordant. Excellent to outstanding fastness to sunlight was found in all mordanted samples. There was absolutely no staining in washed samples. Colour change was not found in the samples subjected to crocking in both dry and wet conditions except in case of ferrous mordanted samples,. The colour was stable as shown by their higher resistance towards acidic and alkaline perspiration. Hence, the dye obtained from Dhavdi flowers proved to be extremely good and can be recommended for dyeing silk fabric.

Keywords : Eco dyeing, Silk, Dhavdi flowers, Fastness

INTRODUCTION

Colour, such a vital and vibrant ingredient of our existence that it is difficult to imagine what life would be like without it. Colour makes things beautiful and improves appearance. Colour in ancient times was considered a spiritual necessity of equal importance to the physical needs of food. At the time of development of spinning, knitting and weaving, the art of dyeing also makes an important contribution to fabric decoration. Dyeing is one of the process of decorating textiles with different dyestuffs and the colour harmonies obtained by a combination of various dyeing methods (Gogoi *et al.*,1997). Natural dyeing materials colouring matters are widely distributed in India, both in rural and urban areas. India had been the largest exporter of natural dyes, such as Indigo, Lac, Safflower, cutch, madder etc. and also beautifully dyed and printed cotton, silk and woolen carpets in artistic designs (Chavan,1995).

The age old tradition of dyeing with fruits, flowers, leaves, barks, berries, stalks and roots of different plants and some of them were also of mineral and animal origin e.g. dye extracted from insect, lack and cochineal etc. Since these dyes are obtained from the natural products, these are called as "Natural Dyes". Natural Dyes are dyes and pigments that are obtained from animal or vegetable matter without chemical processing (Mehra and Mehra, 1993). The early people boiled plant materials to extract the colour and simmered their yarns in the resulting liquid and when the colour faded, they repeated the process.Many firms in the developed nations phased out objectionable products and processes through innovative approaches. But this was just not enough and we had to get back to the square one i.e. from where our forefathers had started. There was the revival of natural dyes for colouring textile.

Natural dyes have several advantages over synthetic dyes from the point of view of health, safety and ecology. They have been known and used for thousands of years without any adverse effect on human health has not been reported. Their operations involved are purely physical such as grinding, vacuum or spray drying and water or solvent extractions. Thus, the processing of natural dyes do not pose the hazardous environmental problems encountered in the manufacture of synthetic dyes, particularly from the point of view of the generation of toxic wastes (Dedhia, 1998).

India with its rich biodiversity offers a variety of sources from which natural colour could be extracted. Naming just a few as Gudhal flowers, Harade, Naspaal, Harsingaar flowers, Kaiphal, Onion rinds, Sinduri, Safflower, Kesula flowers, Marigold, Dhavdi flowers etc. have been worked on for developing various dyeing techniques to achieve better dyes results. *Woodfordia fruiticosa* commonly known as Dhavi or Dhavdi, it is commonly found in forests, on the walls of old buildings and wastelands particularly along the Aravali range and the east of it. In Rajasthan, it is widespread in Todgarh (Ajmer), Anand

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Table 1. Optimization of mordanting method for all the selected mordants.

S. No.	Mordanting method	Mordant	Concentration g/100g yarn	Percent absorption
1	Pre mordanting	Alum	10*	19.64
		Chrome	1	32.65
		Copper Sulphate	3	19.52
		Ferrous sulphate	3*	18.67
2	Sim-mordanting	Alum	10	28.93
		Chrome	3*	27.02
		Copper Sulphate	2	14.19
		Ferrous sulphate	1	33.50
3	Post mordanting	Alum	15	23.88
		Chrome	2	39.72
		Copper Sulphate	2*	42.33
		Ferrous sulphate	2	33.39

* Indicates the optimized value

Sagar forest (Basnwara) Kota, Pali, Sirohi and Jhadol in Udaipur. It is lithophytes in nature and grows from the rock crevices. Its branches are long, spreading bark, leaves sub-opposite; flowers are numerous in short 2 to 15 flowered cymes. It has a high medicine value. Its plant yields a gum soluble in water, which is used in textile printing. The leaves supply 15 – 20% of tannin, bark gives 20 – 27% tannin. The flowers are used in India as dye.

MATERIALS AND METHODS

Dyeing of silk: Silk yarn was dyed using the dye extract from petals of *Woodfordia fruiticosa* following the optimized conditions of dyeing. Chemical such as sodium carbonate, sodium hydroxide, sodium bicarbonate, acetic acid, sodium chloride and urea etc. of L.R. grade were selected for carrying out the research experiments.

Preparation of yarn for dyeing

Silk: Silk contains sericin which interferes with the luster and dye absorption of silk fibres. Hence, removal of sericin gum was a preliminary treatment before dyeing. The raw silk yarn was taken in the form of hank and boiled in the non ionic detergent (Lissapol N) at the ratio of 2 g per liter of water for 45 minutes. The yarn was then thoroughly rinsed to remove traces of detergent and dried in shade.

Optimization of dyeing conditions

i) Selection of wavelength: The percent dye absorption by the silk yarn was calculated by recording the optical density of the dye liquor before and after dyeing. The dye liquor was exposed to visual light of specific wave length using spectrophotometer. The beam of light transmitted by the sample was detected and displayed as optical density. The hue given by the light was recorded for Dhavdi flowers dyes, dilution factor of 1:10 and wavelength of 400 nm. was found optimum.

ii) Optimization of dye concentration: To optimize the suitable dye concentration, 1g to 10 g of dye material was boiled in 100 ml of water to produce dye liquor. 1g of silk yarn was then placed in the dye liquor and dyed for 30 to 45 minutes. The optical density before and after was noted and the percentage of dye absorption was calculated.

iii) Optimization of dye extraction method: Three methods of dye extraction namely – Acidic, Alkaline and Aqueous were employed for extraction of dye and all the three methods of mordanting were used namely pre – mordanting, simultaneous mordanting and post mordanting methods.

a. Pre mordanting method: In this method, the silk yarn was mordanted and then dyed. An aqueous mordant solution was prepared by dissolving required amount of mordant in water. The amount of mordant to he used was calculated based on the weight of silk yarn expressed in terms of percentage as given below :

$$\text{Amount of mordant} = \frac{\text{Wt. of material in g} \times \text{\% of mordant}}{100}$$

The silk yarn was boiled in this solution for 30 minutes and was made ready for dyeing.

b. Simultaneous mordanting: In this method the mordant and the dye were applied simultaneously in the same bath. The silk yarn was placed in the extracted dye bath and dyeing was carried out for 15 minutes. The required amount of mordant was added to the dye bath by lifting silk yarn and stirring. The yarn was then boiled in this solution for 30 minutes. The optical density of the dye – liquor was recorded before and after dyeing. The dyed silk yarn was removed from the dye bath, washed, rinsed and dried in shade.

c. Post mordanting: the yarn was first dyed and then mordanted. An aqueous solution with suitable mordant was prepared. The silk yarn already dyed in dye liquor was boiled in this mordanting liquor for 30 minutes. It was then washed, rinsed and dried.

Dyeing: The silk yarns to be dyed were weighed. The extracted dye liquor was taken as per the M.L. ratio of 1: 50. The optical density was recorded. The yarn was placed

Table 2. Fastness grade of the Dhavdi flowers dye material on silk at optimum dyeing condition for different mordants.

For Alum Mordant:

Mordanting method	Mordant Conc. (%)	Dye absorption (%)	Fastness properties													
			Sun light	washing			dry	crocking			acidic perspiration			Alkaline perspiration		
				cc	cs			wet			cc	cs		cc	cs	
					s	c	cc	cs	cc	cs		s	c		s	c
control	------	14.56	6-7	4	4	4	4-5	4-5	4	4	3-4	3-4	3-4	4	4	3-4
Pre	5	17.78	Very dark shades	4-5	4-5	4	4-5	4-5	4-5	4	5	4-5	4-5	5	4-5	4-5
Pre	10	19.64		4	4-5	4	4-5	4-5	4-5	4-5	4	4	4	4-5	4-5	4-5
Pre	15	18.91		4-5	4-5	4-5	4-5	4	4	4	4	4	4	5	4-5	4-5
Sim	5	26.49	Shades became dark	4	4	3-4	4-5	4-5	4	4	4-5	4	4-5	4	4	4
Sim	10	28.93		4	3-4	3-4	4-5	4	4-5	4-5	4	4	4	4	3-4	3-4
Sim	15	27.11		4	4	3-4	4-5	4	4-5	4	5	4-5	4-5	5	5	5
Post	5	19.40	8	4	4	4	4-5	4-5	4	3-4	5	4-5	4-5	5	5	4-5
Post	10	23.47	8	4	4-5	4-5	4	4	4	3-4	5	5	5	5	4-5	4-5
Post	15	23.88	7	5	5	4	4	4	3-4	3-4	5	4-5	4	4	3-4	3-4

For Chrome Mordant:

Mordanting method	Mor. Con. (%)	Dye absor. (%)	Fastness properties													
			Sun light	washing			dry	crocking			acidic perspiration			Alkaline perspiration		
				cc	cs			wet			cc	cs		cc	cs	
					s	c	cc	cs	cc	cs		s	c		s	c
control	------	14.56	6-7	4	4	4	4-5	4-5	4	4	3-4	3-4	3-4	4	4	3-4
Pre	1	32.65	7-8	3-4	4	3-4	4	4	4	3-4	5	4-5	4-5	4	4	4
Pre	2	30.91	7	4	4	4	4-5	4	4	3-4	5	5	5	4-5	4	4
Pre	3	28.43	7	3-4	3-4	4	4	4	3-4	3-4	5	5	4-5	4	4	4
Sim	1	21.08	6-7	5	4	4	4-5	3-4	3-4	3-4	4	4	4	4	4	4
Sim	2	18.63	7	5	4	4-5	4	3-4	4	3	4	4	4	5	4-5	4-5
Sim	3	27.02	7	5	4	4	4-5	4	4	4	4-5	4-5	4-5	4-5	4	4-5
Post	1	37.51	7	4-5	4	4	3-4	3	4	3	4	4	3-4	4-5	4-5	4-5
Post	2	39.72	6-7	4-5	4-5	4-5	3-4	3	3-4	3	5	4-5	4-5	5	4-5	4-5
Post	3	35.85	8	5	4-5	4-5	4	3-4	3-4	3	5	4-5	4-5	4-5	4-5	4

Table 2. Cont.

For Copper Sulphate Mordant:

Mordanting method	Mordant Con.	Dye absorption	Fastness properties													
			Sun light	Washing			Crocking				Acidic perspiration			Alkaline perspiration		
				cc	cs		dry		wet		cc	cs		cc	cs	
					s	c	cc	cs	cc	cs		s	c		s	c
control	------	14.56	6-7	4	4	4	4-5	4-5	4	4	3-4	3-4	3-4	4	4	3-4
Pre	1	17.88	8	5	4-5	4-5	4	4	4	3-4	5	4	4-5	4-5	4	4
Pre	2	13.05	7	4	4-5	4-5	4-5	4	4	3-4	4-5	4	4	4-5	4-5	4-5
Pre	3	19.52	8	4	4	4	4	4	3-4	3-4	5	5	5	5	5	5
Sim	1	11.18	8	4	4-5	4	4-5	3-4	3-4	3-4	4-5	4-5	4	4-5	4	4
Sim	2	14.19	7	4-5	4-5	4	4	3-4	4	3	4-5	4-5	4	5	4	4
Sim	3	12.66	7	4-5	4-5	4	4	3-4	4	3	5	4-5	4-5	5	4-5	4-5
Post	1	37.98	7	4	3-4	3-4	4-5	3-4	3-4	3	4-5	4	4	4	4	4
Post	2	42.33	8	4-5	4	4-5	4-5	4	4	3	4-5	4-5	4-5	5	4	4-5
Post	3	41.73	7	4	3-4	3-4	4	3-4	3-4	3	5	4	4-5	4-5	4-5	4-5

For Ferrous Sulphate Mordant:

Mordanting method	Mordant Con.	Dye absorption	Fastness properties													
			Sun light	Washing			Crocking				Acidic perspiration			Alkaline perspiration		
				cc	cs		dry		wet		cc	cs		cc	cs	
					s	c	cc	cs	cc	cs		s	c		s	c
control	------	14.56	6-7	4	4	4	4-5	4-5	4	4	3-4	3-4	3-4	4	4	3-4
Pre	1	13.16	8	3-4	3	3-4	3	3	3	2	4	3-4	4	4	3-4	4
Pre	2	17.19	7	4	4	4	3-4	3	3-4	3	4-5	4	3	4-5	3	3
Pre	3	18.67	7	4-5	4-5	4-5	3-4	3	3	3	4-5	4-5	4-5	5	4-5	4-5
Sim	1	33.50	8	4	3	3-4	3-4	3	3	3	4	4	4	4	3-4	3-4
Sim	2	22.34	8	4-5	3	3-4	3-4	3-4	3-4	3	4	4	4	4-5	4-5	4-5
Sim	3	26.47	8	4	3-4	3-4	4	3	3	3	4-5	4	4	4-5	4	4
Post	1	31.26	7	3-4	3-4	3-4	3	3	2	3	5	4-5	4-5	4	4	4
Post	2	33.39	7	4	4	4	3-4	3	3	3	4-5	4	4	4-5	4-5	4
Post	3	28.17	8	5	4-5	4-5	3	3	3	3	5	4	4-5	5	4-5	4-5

Note : Dye Percentage: 4g, Dyeing Time: 45min, Dye Extraction Time: 60 Min, Extraction Medium: Acidic, Mordanting Time: 30 Min.

in the dye liquor and dyed for 45 minutes with occasional stirring. After dyeing the yarn was removed and the optical density of the remaining dye liquor was noted. The dyed silk yarn was then washed, rinsed and dried in shade. The percentage of dye absorption of the yarn was estimated by using the following formula:

$$\% \text{ of dye absorption} = \frac{\text{O.D. of dye liquor before dyeing} - \text{O.D. of the dye liquor after dyeing}}{\text{O.D. of dye liquor before dyeing}} \times 100$$

Tests for colourfastness: The dyed silk yarn was evaluated for colourfastness to washing, sunlight, crocking and perspiration by following standard procedures laid down by Bureau of Indian standards No's – IS : 3361 – 1979, IS : 686 – 1957, IS : 766 – 1956, IS : 971 – 1956 respectively.

RESULTS AND DISCUSSION

Three methods of extraction have been followed for dye extraction from Dhavdi flowers. The concentration of the dye has been estimated by recording the optical density using sprecto photometer. The Aqueous (pH 5.6) method recorded O. D. of 1.90, while acidic and alkaline methods recorded 1.54 and 1.11 optical density, respectively. Since the aqueous method of dye extraction yielded high dye, it was optimized for dye extraction. To optimize the dyeing time for silk yarn, the dye extraction time was carried out for 30, 45, 60 and 90 minutes and the results obtained were 1.01, 1.72, 1.76 and 1.91 optical density. The dye concentration has increased with the increase in the duration of extraction at high temperature up to 90 minutes. When silk samples were dyed in these extracts there was not any difference in the shades obtained with the dye extracts of 60 and 90 minutes. Hence, 60 min was selected for dye extraction. The dyeing was carried out for 30 min and 45 min to optimize the dyeing time for silk yarn. Dye absorption and the depth of shade was found brighter at 45 min (12.31%) therefore it was selected. Fig. 1. Depicts the percent absorption values observed at different dye material concentrations. The percent absorption was found maximum at 4 g of dye stuff per g of silk yarn and thereafter it showed declining trends hence selected.

Mordants such as alum, chrome, copper sulphate and ferrous sulphate were used to mordant silk for dye fixation. In order to select the three best mordant concertration, 1 to 5g of mordants were used in case of chrome, copper and ferrous sulphate whereas 5, 10, 15, and 20% mordant concentration was selected, for Alum mordant. Fig. 2 shows the absorption levels with various concentrations of mordants. The mordant concentration was optimized by way of percentage of absorption and visual assessment of the dyed silk yarn. In case of Alum 5, 10 and 15 % of mordant produced good shades hence selected. In case of chrome and ferrous sulphate and copper sulphate mordant, 1, 2 and 3 % mordant conc.

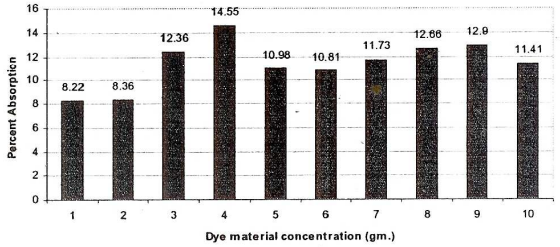

Fig. 1. *Optimization of Dye material concentration.*

were selected as the visual appearance and percent dye absorption values matched. The tones obtained with Alum mordant were mid-buff. The shades resemble sand stone with copper sulphate mordant and quite dark shades of coffee were produced with chrome. Ferrous mordanted sample had added grey hue to the shades in simultaneous mordanting method. Olive green shades resulted in pre and simultaneous mordanting method. In case of chrome mordant the percent absorption as well as shades were found darker and faster in Simultaneous mordanting method. In rest of the mordants, the pre mordanting method had produced more even and good shades in case of alum and Ferrous mordant. The effect of mordanting method on Alum and ferrous mordanted sample was very obvious. Pre mordanting followed by simultaneous and post mordanting produced light to darker shades. (Table 3). The shades of mid buff, sand stone, coffee and olive green were obtained with Dhavdi flower as dye on silk .These shades were found to be very fast. In all the samples the rating were found to be very good to excellent even shades became darker and more brightened after subjecting them to washing and sunlight. The fastness rating of control sample has been changed very interestingly towards higher sides resulting in extremely good fastness to all agencies.

Table 2 gives the fastness grades of alum mordanted silks showing outstanding colousfastness to sunlight even shades became darker in pre mordanted samples.. Even washing fastness was found extremely well having rating of 4 to 4-5. There was absolutely no colour change but very slight staining on adjacent samples was noticed. Dhavdi flower dye showed good to very good colour fastness to both dry and wet crocking on silk except ferrous mordanted samples which showed noticeable colour change in both dry and wet crocking. Colour staining was also visible in wet crocking with copper mordanted samples. No colour change and Colour staining was observed in all the samples of Acidic and alkaline perspiration. Mordanting has seemed to increased the fastness of dyed samples to both acidic and alkaline perspiration when compared with control samples. Darker shades were found in post mordanted samples. A range of pleasing mid-buff colours was observed in both pre and simultaneous mordanted samples.

Table 3. Summary of optimum dyeing conditions of Dhavdi flowers dye material on silk.

mordant	Mordan-ting method	Mor. Con (%)	Dye absorption (%)	Fastness properties													
				Sun light	washing			dry crocking		wet crocking		Acidic perspiration			Alkaline perspiration		
					cc	cs		cc	cs	cc	cs	cc	cs		cc	cs	
						c	s						c	s		c	s
control	——	——	14.56	6-7	4	4	4	4-5	4-5	4	4	3-4	3-4	3-4	4	4	3-4
Alum	Pre-	10	19.64	Very dark shades	4	4-5	4	4-5	4-5	4-5	4-5	4	4	4	4-5	4-5	4-5
Chrome	sim	3	27.02	7	5	4	4	4-5	4	4	4	4-5	4-5	4-5	4-5	4	4-5
Cuso4	post	2	42.33	8	4-5	4	4-5	4-5	4	4	3	4-5	4-5	4-5	5	4	4-5
Feso4	pre	3	18.67	7	4-5	4-5	4-5	3-4	3	3	3	4-5	4-5	4-5	5	4-5	4-5

Note : Dye Percentage: 4g, Dyeing Time: 45min, Dye Extraction Time: 60 Min, Extraction Medium: Aqueous, Mordanting Time: 30 Min.

Singh *et al.* (1993) pointed out that while samples mordanted with copper sulphate and chrome had a fair to good light fastness, these samples became dark on exposure to light. This may be attributed to the photo chemical changes in dye molecules which lead to change of hue or bleeding. The fastness grades of chrome mordanted and dyed silks are given in table - 2. It is clear from the table that the lighter shades resulted in pre-mordanted samples. In case of Simultaneous and post mordanted samples, 3% conc. produced deep and dark shades. All the colours are fast on silk. Very good to outstanding sunlight colorfastness was found in all samples except simultaneous (1%) and post (2%) mordanted samples. The wash fastness was fair to good in pre-mordanted samples. However in simultaneous and post mordanted samples the ratings were found from good to very good. The colour staining was slight. The silk samples showed very good resistance to dry crocking in few post mordanted samples with slight staining on adjacent samples. However in wet crocked samples slight colour change with noticeable staining was observed in almost all the samples. Colour staining was also noticeable in post mordanted samples of wet crocking. Very good to excellent fastness to both Acidic and alkaline perspiration was observed in chrome mordanted silks in all the three mordanting methods.

Silk mordanted with copper sulphate and dyed in Dhavdi flowers dye showed very good to outstanding fastness to sunlight as seen from table 2. The data such obtained coincide with the fact that the copper sulphate mordant improves the colour fastness to sunlight. Colour change was not found in the samples after washing, dry crocking and both acidic and alkaline perspiration.

It was observed that copper sulphate gives good wash fastness with medium and higher concentration using all the 3 mordanting method. While premordanting also gave good wash fastness at 1% concentration the other 2 methods gave an average wash fastness at the same concentration (Aeron, 2000).

Slight colour staining was observed in 1% Post mordanted samples subjected to washing. Noticeable staining was found in wet crocked samples especially in

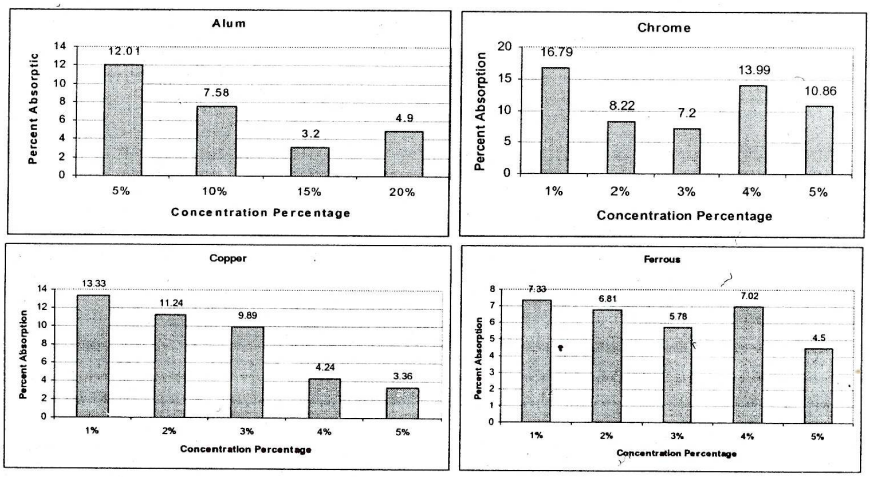

Fig. 2. *Optimization levels with various concentrations of mordants*

Table 4. Cost evaluation for dyeing silk with Dhavdi flowers.

Material	Cost per kg of silk (Rs)
Dye material	40.00
Acetic acid	1.00
Sodium bicarbonate	Nil
Fuel sodium	25.00
Soap & Other chemicals	7.00
Total	73.00

Cost of alum (100 g) per kg silk-Rs. 14.00; cost of chrome (30 g) per kg silk -Rs. 12.70; cost of copper sulphate (20 g) per kg silk - Rs. 5.00, ferrous sulphate (30 g) per kg silk -Rs. 4.00.

simultaneous and post mordanted samples. All silk samples mordanted with copper sulphate showed high resistance to both acidic and alkaline perspiration .There was absolutely no colour change and no colour staining on adjacent samples.

As seen from table 2, ferrous sulphate mordanted silks also showed similar results as with the other mordanted samples. Very good to outstanding fastness to sunlight was found irrespective of the mordanting method and mordant concentration. Dark shades of coffee brown were obtained. 3% conc. produced darker shades irrespective of mordanting method. It was interesting to note that the shades became darker with increase in mordant concentration.

These was absolutely no colour change and no staining in post mordanted silk samples. The fastness to both acidic and alkaline perspiration was found to be remarkable when compared with the ratings of control sample. The unmordanted sample showed only fair to good resistance to alkaline perspiration which has changed to very good to excellent without any colour change and colour staining in most of the samples.

The economic viability of using the natural dye for cottage level dyeing (Table 4) showed that the dyeing process was very economic and can be adopted at village level by rural people also.

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