

Effect on Plant Fibre of the Natural Dye with Mordant's Combination

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Abstract

The colour fastness properties of the flower of *Calendula officinalis* dyed on silk were studied using combination of mordants such as Myrobalan: nickel sulphate, Myrobalan: aluminium sulphate, Myrobalan: potassium dichromate, Myrobalan: ferrous sulphate, Myrobalan: stannous chloride in the ratio of 1:3, 1:1, 3:1. The washing, rubbing, light and perspiration fastness of the dyed samples was also evaluated, giving fair to excellent fastness grades.

Keywords: Natural dye, *Calendula officinalis*, Mordant, colour fastness.

Introduction

India 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 relatives¹. 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. One such product from nature is the dye. Natural dyes have the ability to produce wide range of tints and shades, with the same dye material². But with the invention of synthetic dyes in 1856, the prominence of natural dyes slacked because the synthetic dyes had some advantages over natural dyes like colour fastness, good reproducibility of shades, brilliance of colour and easy to use³. These synthetic dye stuffs produced hazardous by-products some of which possess carcinogenic intermediates and hence a ban has been imposed by Germany and some other European countries on the use of benzidine dyes in textile garments exported into their countries. Hence due to the current eco-consciousness, the researcher's attention has been shifted to the use of natural dyes for dyeing textile materials⁴. The

present study has been undertaken so as to revive the age-old art of dyeing with natural dyes. In the present work, the flower of *Calendula officinalis* dye was used to dye silk at optimized dyeing conditions, using combination of mordants and evaluate the resultant colour fastness of the dyed samples to washing, rubbing, perspiration and light^{5, 6}.

Materials and Methods

Materials

Bleached plain weave silk fabric obtained from Gandhi Ashram, Dehradun, was used for the study. Analytical reagents (AR) grade ferrous sulphate, aluminium sulphate, nickel sulphate, potassium dichromate, stannous chloride, commercial grade acetic acid, common salt, sodium carbonate were used. A natural mordant Myrobalan⁷ (*Terminalia chebula*) powder was used for the study. The ethanol extract of the flower of *Calendula officinalis* was used to get brown colour for dyeing of fabrics. Depending upon the mordant used, the colour obtained on textiles from the flower of *Calendula officinalis* extract may give different shades. The Myrobalan (harda) powder was soaked in water (1:10 volume) for overnight (12h) at room temperature to obtain the swelled Myrobalan gel. It was then mixed with a known volume of water and heated at 80°C for 30min. The resulting solution is cooled and filtered. The filtrate was used as final mordant solution for mordanting^{8, 9}. The present study was undertaken to dye silk yarn with the flower of *Calendula officinalis* dye. A known quantity of flower were dried, powdered and soaked in warm water overnight. The extract was obtained by boiling it in the same water and allowed to cool, finally filtered and used for dyeing. The dyeing was carried out at optimized dyeing conditions namely; dye extraction time 60min, material to liquor ratio 1:20, dyeing Time 50 min. The mordant combinations viz. Myrobalan: nickel sulphate, Myrobalan: aluminium sulphate, Myrobalan: potassium dichromate, Myrobalan: ferrous sulphate, Myrobalan: stannous chloride were used in the ratio of 1:3, 1:1 and 3:1. The total amount of two mordants used in each combination was 5% on the weight of the fabric i.e. 5 gm of the mordant / 100 gm of the fabric. Each of the five mordant combinations in three different ratios mentioned above were used with all the three mordanting methods namely premordanting, simultaneous mordanting and post-mordanting for dyeing. After dyeing, the solution was allowed to cool, removed from dye bath, rinsed under running water to remove excess dye particles and shade dried.

Evaluation of colour fastness^{10, 11}

Colour fastness to washing of the dyed fabric samples was determined as per IS: 764-1984 method 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-A03 (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 ISO: 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 method¹². The sample was exposed to UV light in a Shirley MBTF Microsal fade-O-meter (having 500 watt Philips mercury bulb tungsten filament lamp simulating day light) along with the eight blue wool standards (BS 1006: 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 ISO:971-1983 composite specimen was prepared by placing the test specimen between two adjacent pieces of silk fabric and stitched all among four sides¹³. 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 $37\pm 2^\circ\text{C}$. 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

Mordant combination-Myrobalan: Nickel Sulphate

The evaluation of colour fastness to light, washing, rubbing and perspiration of flower of *Calendula officinalis* dyed silk samples treated with Myrobalan: Nickel Sulphate combination in aqueous medium is presented in Table-1. All the treated samples subjected to light showed fairly good (3-4) light fastness for all ratio mordant combinations. The washing fastness grades ranged (4) for all of the treated samples and there was no colour staining. The colour change to dry and wet rubbing for all the treated samples was excellent (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 colour staining (5) for all the treated samples in both acidic and alkaline media.

Table 1: Fastness Grades of flower of *Calendula officinalis* Dye Dyed on silk at Optimum Dyeing Conditions (Wavelength 420 nm. Dye Extraction Time 60min, Material to liquor ratio 1:20, Dyeing Time 50 min.) Using Mb:NS Mordant Combination.

Mordanting Method	Mordant Proportions	Light Fastness Grades	Washing Fastness Grades		Rubbing Fastness Grades			Perspiration Fastness			
			CC	CS	Grades		Acidic		Alkaline		
					Dry CC	CS	Wet CC	CS	CC	CS	CC
Pre Mordanting	1:3	3-4	4	5	5	5	5	5	4	5	4
	1:1	3-4	4	5	5	5	5	5	4	5	4
	3:1	3-4	4	5	5	5	5	5	4	5	5
Simultaneous Mordanting	1:3	3-4	4	5	5	4-5	5	5	5	5	5
	1:1	3-4	4	5	5	5	5	5	5	5	5
	3:1	3-4	4	5	5	5	5	5	5	5	4-5
Post Mordanting	1:3	3-4	4	5	5	4	5	5	5	5	5
	1:1	3-4	4	5	5	4	5	5	5	5	4-5
	3:1	3-4	4	5	5	4	5	5	5	5	5

Mb:NS – Myrobalan : Nickel sulphate, CC – Colour change, CS – Colour Staining

Mordant combination-Myrobalan: Aluminium Sulphate

The evaluation of colour fastness to light, washing, rubbing and perspiration of flower of *Calendula officinalis* dyed silk samples treated with Myrobalan: Aluminium Sulphate combination in aqueous medium is presented in Table-2. All the treated samples subjected to light showed fairly good (3-4) light fastness for all ratio mordant combinations. The treated samples for pre mordanting showed fair (3 to 2-3) washing fastness grades, but they ranged between excellent to good (4-5 to 4) for all of the treated samples for simultaneous and post mordanting. There was no colour staining. The colour change to dry and wet rubbing for all the treated samples was excellent (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 4-5 to 4, except for 1:3 mordant proportion in pre-mordanting method, where it was fair (3), for all samples in both acidic and alkaline media. There was no colour staining (5) for all the treated samples in both acidic and alkaline media.

Table 2: Fastness Grades of flower of *Calendula officinalis* Dye Dyed on silk at Optimum Dyeing Conditions (Wavelength 420 nm. Dye Extraction Time 60min, Material to liquor ratio 1:20, Dyeing Time 50 min.) Using Mb:AS Mordant Combination.

Mordanting Method	Mordant Proportions	Light Fastness Grades	Washing Fastness Grades		Rubbing Fastness Grades			Perspiration Fastness			
			CC	CS	Dry		Wet	Acidic		Alkaline	
					CC	CS		CC	CS	CC	CS
Pre Mordanting	1:3	3-4	3	5	4-5	5	3	5	3	5	3
	1:1	3-4	2-3	5	5	4-5	5	5	4-5	5	4
	3:1	3-4	2-3	5	5	4-5	5	5	4-5	5	4
Simultaneous Mordanting	1:3	3-4	4-5	5	5	5	5	5	4	5	4
	1:1	3-4	4	5	5	5	5	5	4	5	4
	3:1	3-4	4	5	5	5	5	5	4	5	4
Post Mordanting	1:3	4	4	5	5	4-5	5	5	4	5	4-5
	1:1	4	4-5	5	5	4/5	5	5	4-5	5	4-5

Mb:AS-Myrobalan: Aluminium sulphate, CC-Colour change, CS-Colour Staining

Mordant combination-Myrobalan: Potassium dichromate

The evaluation of colour fastness to light, washing, rubbing and perspiration of flower of *Calendula officinalis* dyed silk samples treated with Potash dichromate: Copper sulphate combination in aqueous medium is presented in Table-3. The treated samples subjected to light showed fairly good (3-4) light fastness for all ratio mordant combinations. The washing fastness grades showed fairly good (3-4) for all the treated samples except for 1:3 mordant proportion in premordanting method, where it was fair (2 -3). The colour change to dry and wet rubbing for all the treated samples was excellent (5). The colour staining ranged between no staining to negligible staining (4-5) in dry and wet rubbing except for pre-mordanting method where it showed fair (3). Most of the treated samples showed excellent fastness grade to colour change, except for 1:3 mordant proportion in pre mordanting methods, where it was good (3). There was no colour staining (5) for all treated samples in both acidic and alkaline media.

Table 3: Fastness Grades of flower of *Calendula officinalis* Dye Dyed on silk at Optimum Dyeing Conditions (Wavelength 420 nm. Dye Extraction Time 60min,

Material to liquor ratio 1:20, Dyeing Time 50 min.) Using Mb:PD Mordant Combination

Mordanting Method	Mordant Proportions	Light Fastness Grades	Washing Fastness Grades		Rubbing Fastness Grades			Perspiration Fastness			
			CC	CS	Dry		Wet	Acidic		Alkaline	
					CC	CS		CS	CC	CS	CC
Pre Mordanting	1:3	3-4	2-3	5	5	3	5	5	3	5	3
	1:1	3-4	3	5	5	3	5	5	4	5	4
	3:1	3-4	3	5	5	3	5	5	4	5	4
Simultaneous Mordanting	1:3	3-4	3	5	5	4-5	5	5	4	5	4
	1:1	3-4	3	5	5	4-5	5	5	4	5	4
Post Mordanting	3:1	3-4	3	5	5	4-5	5	5	4-5	5	4
	1:3	3-4	4-5	5	5	4-5	5	5	4-5	5	4-5
	1:1	3-4	4	5	5	4-5	5	5	4-5	5	4-5
	3:1	3-4	4-5	5	5	4-5	5	5	4-5	5	4-5

Mb: PD – Myrobalan: Potassium dichromate, CC – Colour change, CS – Colour Staining

Mordant combination-Myrobalan: Ferrous Sulphate

The evaluation of colour fastness to light, washing, rubbing and perspiration of flower of *Calendula officinalis* dyed silk samples treated with Myrobalan: Ferrous Sulphate combination in aqueous medium is presented in Table-4. The treated samples subjected to light showed fairly good (4-3-4) light fastness for all ratio mordant combinations. The washing fastness grades ranged between excellent to good (5-4) for all the treated samples. The colour change to dry and wet rubbing for all the treated samples was excellent (5). The colour staining in dry rubbing showed fair (4). Most of the treated samples showed excellent fastness grade to colour change, except for 1:3 mordant proportion in simultaneous mordanting method where it was good (4), for all samples in both acidic and alkaline media. There was no colour staining (5) for all the treated samples in both acidic and alkaline media.

Table 4: Fastness Grades of flower of *Calendula officinalis* Dye Dyed on silk at Optimum Dyeing Conditions (Wavelength 420 nm. Dye Extraction Time 60min, Material to liquor ratio 1:20, Dyeing Time 50 min.) Using Mb: FS Mordant Combination.

Mordanting Method	Mordant Proportions	Light Fastness Grades	Washing Fastness Grades		Rubbing Fastness Grades			Perspiration Fastness			
			CC	CS	Dry		Wet	Acidic		Alkaline	
					CC	CS		CS	CC	CS	CC
Pre Mordanting	1:3	3-4	5	5	5	4	5	5	4	5	4
	1:1	3-4	5	5	5	4	5	5	4	5	4
	3:1	3-4	5	5	5	4	5	5	4	5	4
Simultaneous Mordanting	1:3	3-4	5	5	5	4	5	5	4	5	4
	1:1	4	5	5	5	4	5	5	4-5	5	4-5
Post Mordanting	3:1	4	5	5	5	4	5	5	4	5	4
	1:3	3-4	5	5	5	4	5	5	4	5	4
	1:1	3-4	5	5	5	4	5	5	4-5	5	4
	3:1	3-4	5	5	5	4	5	5	4	5	4

Mb: FS – Myrobalan: Ferrous Sulphate, CC – Colour change, CS – Colour Staining

Mordant combination-Myrobalan: Stannous Chloride

The evaluation of colour fastness to light, washing, rubbing and perspiration of flower

of *Calendula officinalis* dyed silk samples treated with Myrobalan: Stannous Chloride combination in aqueous medium is presented in Table-5. The treated samples subjected to light showed fairly good (4 to 3-4) light fastness for all ratio mordant combinations. The washing fastness grades ranged between excellent to good (4-5 to 4) for all of the treated samples and there was no colour staining. The colour change to dry and wet rubbing for all the treated samples was excellent (5). The colour staining ranged between negligible to slight staining (4-5) in dry rubbing. The perspiration fastness grades ranged between 4 and 5 for all samples in both acidic and alkaline media. There was no colour staining (5) for all the treated samples in both acidic and alkaline media.

Table 5: Fastness Grades of flower of *Calendula officinalis* Dye Dyed on silk at Optimum Dyeing Conditions (Wavelength 420 nm. Dye Extraction Time 60min, Material to liquor ratio 1:20, Dyeing Time 50 min.) Using Mb: SC Mordant Combination

Mordanting Method	Mordant Proportions	Light Fastness Grades	Washing Fastness Grades		Rubbing Fastness Grades			Perspiration Fastness			
			CC	CS	Dry		Wet	Acidic		Alkaline	
					CC	CS		CC	CS	CC	CS
Pre Mordanting	1:3	4	4-5	5	5	4-5	5	5	4-5	5	4-5
	1:1	4	4-5	5	5	3-4	5	5	4-5	5	4-5
	3:1	4	4-5	5	5	3-4	5	5	4-5	5	4-5
Simultaneous Mordanting	1:3	3-4	5	5	5	5	5	5	4-5	5	4-5
	1:1	3-4	4-5	5	5	5	5	5	4-5	5	5
Post Mordanting	3:1	3-4	4	5	5	5	5	5	4-5	5	5
	1:3	3-4	4	5	5	4-5	5	5	4-5	5	5
	1:1	3-4	5	5	5	4-5	5	5	4-5	5	5
	3:1	3-4	4-5	5	5	4-5	5	5	4-5	5	5

Mb: SC – Myrobalan: Stannous Chloride, CC – Colour change, CS – Colour Staining

Conclusion

It was found from the study that flower of *Calendula officinalis* dye can be successfully used for dyeing of silk to obtain a wide range of soft and light colours by using combination of mordants. With regards to colour fastness, test samples exhibited excellent fastness to washing (except for pre-mordanting using Myrobalan: Potassium dichromate combination); excellent fastness to rubbing (except for premordanting using Myrobalan: Potassium dichromate combination); good to excellent fastness to perspiration in both acidic and alkaline media and fairly good fastness to light.

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