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Extraction of natural dyes from *Spathodea campanulata* and its application on silk fabrics and cotton

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ABSTRACT

The natural dye was obtained from different solvent extracts of Spathodea campanulata flowers. The dyeing and colour fastness potential of the extracts were evaluated on silk fabrics and 100% cotton using combination of mordants (harda/metallic salts) such as 0.15% alum, 0.08% $CuSO_4$, 0.04% $FeSO_4$, 0.06% $K_2Cr_2O_7$, Myrobolan, 0.08% $SnCl_2$, Myrobolan: $K_2Cr_2O_7$ (1:1), Myrobolan : $SnCl_2$ (1:1). Washing and rubbing with detergent for 30minutes at 60^oC has showed negligible effect on dye fastness. The UV, sunlight and heat fastness of the dyed samples were also evaluated. The results revealed fair to excellent fastness grades. Among the various extracts studied, the extract obtained from 0.1N NaOH along with Myrobolan: $SnCl_2$ (1:1) as mordant produced the better results. The stability studies of the extract was done before dyeing and showed appreciable results.

Keywords: Dye, cotton, silk fabrics, Myrobolan, $SnCl_2$.

INTRODUCTION

Prehistoric dyeing technique like applying crushed pigments to cloth or fixing plant exudates to fabric were most practiced until 19th century. The replacement of natural dyes could happen until the introduction of synthetic dyes due to feasible coloring property of natural dyes. In addition of availability of wide range of colors, greater reproducibility and enhanced value of dyeing could be achieved at lower specific cost. In recent times, awareness as been created and ruthless eco friendly standards are imposed by many countries in response to noxious reactions associated with the synthetic dyes [1]. The methods are made more sophisticated by time and techniques are developed using the natural dyes from berries, crushed fruits and other plants, which are boiled with the fabric to give water resistant and light colors. The use of natural dyes however can offer not only a rich and diverse source of dyestuff, but also provide an alternative and sustainable income to farmers through harvest and sale of these plants. The source for the natural dye can be made easily available from tree waste and grown in gardens [2]. There is been lots of demand since a decade in using the natural dye for coloration [3]. Reports have been published on use of natural dyes over synthetic dyes on silk [4, 5] and cotton [6, 7]. There is greater need in the present day to revitalize the sacrament of natural dye [8] and dyeing techniques as an alternative for the use of unsafe synthetic dyes. Hence the present study was undertaken with the objectives of extraction and application of eco-friendly natural dyes from fresh and dry flowers of *Spathodea campanulata* on silk fabrics and cotton. The effect of UV, sunlight and heat fastness on the fadedness of color was studied. The present study also deals with the effect of mordanting and dyeing properties of cotton and silk fabrics.

MATERIALS AND METHODS

Plant material:
Dark red fresh flowers of *Spathodea campanulata* were collected from Rishi herbal technologies, Bangalore.

Substrates:
Desized, scoured and bleached with 0.5 % hydrogen peroxide, plain 100% cotton fabric and loom state silk procured form the local market were used for the study.

Chemicals:
Analytical grade stannous chloride, copper sulphate, ferrous sulphate, potassium-di-chromate, Sodium hydroxide, HCl, ethanol, alum and natural mordant Myrobolan.

Solvents used for Extraction:
Water, Alkaline water (0.1 N NaOH), Acidified water (0.1N HCl) and Ethanol.

Extraction and application Method:
Application of dyes obtained from fresh flowers of *Spathodea campanulata* on silk fabrics and cotton were evaluated in four stages i.e., Pre-treatment/Scouring, Extraction of dyes, fixing dye with the fiber and dyeing.

Scouring of Silk fabrics and cotton:
Washing solution containing 0.5g/L sodium carbonate and 2g/L nonionic detergent were used to scour silk and cotton fabrics at 50°C for 20min, keeping the material to liquor ratio at 1:40. The scoured material was thoroughly washed with tap water and dried at room temperature. The scoured material was soaked in clean water for 30 min prior to dyeing or mordanting.

Extraction of dyes
Using soxlet extraction process, 10 g of fresh flowers were extracted using 100ml of different solvent system like alkaline water (0.1N NaOH), acidified water (0.1N HCl), distilled water and ethanol. Extraction was performed at temperature 80-85°C for 1hour and three cycles for first three solvents and for ethanol it was maintained at 45-50°C 1 hour and three cycles. All the color was extracted from the flowers by the end of third cycle. The extracts obtained were dried in vacuum (212psi) at 55°c and the powder obtained was stored for further use. The dye extracted from alkaline water was dark red and retained its color after drying process where as the other extracts color was faded. Hence, the extract obtained from alkaline water was used for dyeing process.

Mordanting & Dyeing Procedure:
The extract from alkaline water of *Spathodea campanulata* flower gave red color and the same was used for dyeing fabrics. The concentration and the type of mordant used gave different shades of the color. The myrobolan (harda) powder was soaked in water (1:10 volume) for overnight (12h) at room temperature to obtain the swelled myrobolan gel. It was then mixed with a known volume of water and heated at 80^0C for 30 min. The resulting solution is cooled and filtered. The filtrate was used as final mordant solution for mordanting [9].

Then de-starching of cloth was done by treating it with 10% NaOH for 15minutes and then washed with cold distilled water. This cloth was then transferred into different mordants (3%), 0.15% alum, 0.08% $CuSO_4$, 0.04% $FeSO_4$, 0.06% $K_2Cr_2O_7$, Myrobolan, 0.08% $SnCl_2$, Myrobolan: $K_2Cr_2O_7$ (1:1), Myrobolan : $SnCl_2$ (1:1) for 30 minutes followed by treatment in the dye bath for one hour.

Dyeing procedure was also done with fabric without mordanting. Then the cloth was treated with tepol (color fixative) and dried in sunlight. Cloth pieces were individually soaked with the mixture of extract-mordant solution. After 30 minutes of soaking the cloth was dried in sunlight for 2 hours. The sun dried cloth was further evaluated for its colour, lightness and wash fastness. Wash fastness was tested by washing with soap water (10% w/v) and heat resistance was tested by keeping the cloth at various temperatures viz., 50, 60, 70°C for 30 minutes in the oven without water.

Surface Colour Strength Determination (K/S value)
Using the formula (Kubelka munk equation) and a computer supported with Macbeth 2020 plus reflectance spectrophotometer the K/S value of the undyed, dyed cotton and silk fabrics was determined [10].

$$K/S = \frac{(1- R\lambda_{max})^2}{2R\lambda_{max}} = \alpha C_d$$

Where K is the coefficient of absorption; S the coefficient of scattering; C_d, the concentration of the due and $R\lambda_{max}$ the surface reflectance value of the sample at a particular wavelength, where maximum absorption occurs for a particular colour component.

UV Fastness
Colour fastness to exposure to light was determined. The sample was exposed to UV light using a transilluminator against the fading of blue wool standards.

RESULTS AND DISCUSSION

All the dyed fabrics were evaluated for their surface color strength (K/S values) reported in table 1 & table 2 and the results of assessment of colour fastness behavior to light, washing and heat resistance are given in table 3. The treated samples subjected to light showed appreciable results with all different types of mordants of optimum concentration. No colour staining was observed with the washing fastness and it ranged between 4- 5. Among differently mordanted, silk dyed with 0.1 N NaOH extract with Myrobolan : $SnCl_2$ (1:1) by simultaneous mordanting technique, renders the fabric relatively higher K/S value as compared to other mordanting system.

The use of 0.08% $SnCl_2$ by simultaneous mordanting technique followed by further dyeing with comparable dose of 0.1 N NaOH extract flower of *Spathodea campanulata* colour shows the K/S value of 2.63/2.49 for Light fastness and UV fastness respectively and thus is considered as next good performer and 0.08% of copper sulphate and 0.1 N NaOH extract flower of *Spathodea campanulata* colour shows the K/S value of 2.51/2.45 for light fastness and UV fastness respectively was the next good treatment. It is been reported that wash fastness of the dye is influenced by the rate of diffusion of the dye and state of the dye inside the fiber [11].

Table 1: Surface colour strength with light and washing behavior (K/S values) of dyed cotton and silk fabrics.

Fabric	Mordant	K/S(λ =420 nm)	
		Light	Washing
Silk	Control (No Mordant)	1.94	1.98
	0.15% alum	2.39	2.42
	0.08% $CuSO_4$	2.54	2.51
	0.04% $FeSO_4$	2.43	2.42
	0.06% $K_2Cr_2O_7$	2.21	2.26
	Myrobolan	2.36	2.38
	0.08% $SnCl_2$	2.63	2.49
	Myrobolan: $K_2Cr_2O_7$ (1:1)	2.28	2.30
	Myrobolan : $SnCl_2$ (1:1)	**2.89**	**2.91**
Cotton	Control (No Mordant)	1.39	1.40
	0.15% alum	2.35	2.35
	0.08% $CuSO_4$	2.55	2.48
	0.04% $FeSO_4$	2.47	2.25
	0.06% $K_2Cr_2O_7$	2.19	2.16
	Myrobolan	2.30	2.29
	0.08% $SnCl_2$	2.61	2.65
	Myrobolan: $K_2Cr_2O_7$ (1:1)	2.24	2.26
	Myrobolan : $SnCl_2$ (1:1)	**2.74**	**2.82**

Mordants play very important role in imparting color to the fabric. The mordants used in combination in different ratios gives varying shades [12]. Similarly for cotton fabric, it is observed that among differently mordanted bleached cotton subsequently dyed with 0.1N NaOH extract flower of *Spathodea campanulata* with Myrobolan : $SnCl_2$ (1:1) by simultaneous mordanting technique, renders the fabric relatively higher K/S value 2.74/2.82 for light fastness and UV fastness respectively as compared to other mordanting system. The use of 0.08% $SnCl_2$ by simultaneous mordanting technique followed by further dyeing with comparable dose of 0.1N NaOH extract flower

of *Spathodea campanulata* colour shows the K/S value of 2.61/2.65 for light fastness and UV fastness respectively and thus is considered as next good performer and 0.08% of copper sulphate and 0.1 N NaOH extract flower of *Spathodea campanulata* colour shows the K/S value of 2.55/2.56 for light fastness and UV fastness respectively is the next good performer (Fig.6). Among all the mordants used, the increase in K/S value is found to be the highest for Myrobolan: $SnCl_2$ (1:1) combination mordan. This is more predominant on silk than on cotton. Complexing the fiber with mordant has the effect of insolubilizing the dye, making it color fast [13]. The fabrics dyed with *Spathodea campanulata* flower extract exhibited good fastness properties.

Table 2: Surface colour strength with heat resistance behavior (K/S values) of dyed cotton and silk fabrics.

Fabric	Mordant	K/S(λ =420 nm)		
		50^0c	**60^0c**	**70^0c**
Silk	Control (No Mordant)	2.58	2.57	2.54
	0.15% alum	2.45	2.44	2.40
	0.08% $CuSO_4$	2.38	2.36	2.36
	0.04% $FeSO_4$	2.52	2.50	2.45
	0.06% $K_2Cr_2O_7$	2.26	2.25	2.20
	Myrobolan	2.21	2.21	2.16
	0.08% $SnCl_2$	2.49	2.47	2.45
	Myrobolan: $K_2Cr_2O_7$ (1:1)	2.40	2.38	2.35
	Myrobolan : $SnCl_2$	**2.89**	**2.85**	**2.80**
Cotton	Control (No Mordant)	1.66	1.63	1.60
	0.15% alum	2.38	2.36	2.32
	0.08% $CuSO_4$	2.65	2.64	2.63
	0.04% $FeSO_4$	2.74	2.72	2.70
	0.06% $K_2Cr_2O_7$	2.32	2.31	2.28
	Myrobolan	2.45	2.42	2.38
	0.08% $SnCl_2$	2.26	2.25	2.24
	Myrobolan: $K_2Cr_2O_7$ (1:1)	2.64	2.63	2.60
	Myrobolan : $SnCl_2$	**2.84**	**2.84**	**2.81**

Table 3: Colourfastness of dyed cotton and silk fabrics with selective mordants.

Fabric	Mordant	Light	Washing		Heat resistance		
			CC	CS	50^0c	60^0c	70^0c
Silk	Control (No Mordant)	3	3-4	4	4	4	4
	0.15% alum	4	5	4	4	4	4
	0.08% $CuSO_4$	4	5	4	4	4	4
	0.04% $FeSO_4$	4	4	4	4	4	4
	0.06% $K_2Cr_2O_7$	4	4	4	4	4	4
	Myrobolan	3	4	4	4	4	4
	0.08% $SnCl_2$	4	4	4	4	4	4
	Myrobolan: $K_2Cr_2O_7$ (1:1)	4	4	4	4	4	4
	Myrobolan : $SnCl_2$	**4**	**5**	**4-5**	**4**	**4**	**4**
Cotton	Control (No Mordant)	3-4	3	4	4	4	4
	0.15% alum	4	4	4	4	4	4
	0.08% $CuSO_4$	4	4	4	4	4	4
	0.04% $FeSO_4$	4	4	4	4	4	4
	0.06% $K_2Cr_2O_7$	4	4-5	4	4	4	4
	Myrobolan	4	5	4	4	4	4
	0.08% $SnCl_2$	4	4	4	4	4	4
	Myrobolan: $K_2Cr_2O_7$ (1:1)	4	5	4	4	4	4
	Myrobolan : $SnCl_2$	**4**	**5**	**4-5**	**5**	**5**	**5**

CC – Colour change, CS – Colour Staining

The increase in colour strength K/S values with selective mordants on both silk and cotton fabrics were in the following order: Myrobolan: $SnCl_2$, 0.08% $SnCl_2$, 0.08% $CuSO_4$, 0.04% $FeSO_4$, 0.15% alum, Myrobolan, Myrobolan: $K_2Cr_2O_7$, 0.06% $K_2Cr_2O_7$. Considering the dyeing results, the sequential mordanting systems using Myrobolan: $SnCl_2$ (1:1) + alkaline water (0.1N NaOH) extract of flower of *Spathodea campanulata*, 0.08% $SnCl_2$+ alkaline water (0.1N NaOH) extract of flower of *Spathodea campanulata* and 0.08% $CuSO_4$+ alkaline water (0.1N NaOH) extract of flower of *Spathodea campanulata* were found to be more prospective, rendering a higher degree of increase in surface colour strength. Hence these three systems of mordanting technique could be applied in the dyeing process for both silk and cotton fabrics in the future applications.

CONCLUSION

The dyeing of silk and cotton can be achieved using the flower extracts of *Spathodea campanulata* by using natural and metallic mordants. The dyeing of flower extract exhibited excellent fastness to washing/rubbing and fairly good fastness to light. Among the various fibre-mordanting systems, the use of Myrobolan: $SnCl_2$ with alkaline water extract of flower of *Spathodea campanulata* showed maximum K/S values as compared to other selective mordanting systems.

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