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A Study on Extraction and Application of Eco-Friendly Natural Dye Extracted from Flowers of Hibiscus Vittifoliusl on Silk Fabric using combination of Mordants and their Antimicrobial Activity

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Abstract

In the present study dye extract was prepared from flower of Hibiscus vittifolius L was used as a source of natural dyes for dyeing of silk samples. It belongs to family malvaceae, commonly known as siru thutthi. The dye has good scope in the commercial dyeing of silk in textile industry. So in present investigation, bleached silk fabrics were dyed with different chemical mordants. The colour fastness properties dyed fabrics washing, light, rubbing fastness and perspiration fastness and K/S –value of dyed silk fabric were determined. From the comparative study of fastness properties and K/S –value of the dyed silk samples, Hibiscus vittifolius L in pre mordanting method with 4% mordant (ferrous sulphate) combination give better results. Antimicrobial activities of the dye extract were also studied.

Keywords: Extraction, Natural dyes, Flowers, *Hibiscus vittifolius* L, Silk, Textiles.

Introduction

India has a rich biodiversity and it is not only one of the world's twelve mega diversity countries, but also one of the eight major centers' of origin and diversification of domesticated taxa. It 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 relatives[1]. 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 materials[2]. Presently there is an excessive use of synthetic dyes, estimated at around 10x106 tons per annum, the production and application of which release vast amount of waste and unfixed colorants causing serious health hazards and disturbing the eco-balance of nature. Nowadays, fortunately there is increasing awareness among people towards natural dyes. Natural dyes are preferred in developed countries, because they are non-allergic, non-carcinogenic and have lower toxicity and better biodegradability than the synthetic dyes[3]. Research has shown that synthetic dyes are suspected to petrochemical release harmful chemicals that are considered to be high pollutant in both water and land which would be allergic, carcinogenic and detrimental to human health. Natural dyes from plants provide important alternative to based dyes and if harvesting is carefully managed without affecting environmental and social benefits. Natural dyes not only release medicinal properties but also improve the aesthetic value of the product and they are unique and eco friendly[4]. Recently, interest in the use of natural dyes has been growing rapidly due to the result of stringent environmental standards imposed by environmental board and pollution control board of many countries in response to toxic and allergic reactions associated with synthetic dyes[5]. Colour fastness is the resistance of a material to change any of its colour characteristic are extent of transfer of its colorants to adjacent which materials in touch generally light fastness wash fastness and rub fastness are considered far textile fibers[6].

Hibicus vittifolius L: Undershrub, up to 1 m high; stem stellate-pubescent, greenish-purple. Leaves alternate Stipule small, deciduous. Flowers 5cm across, golden yellow, with dark purple center, solitary, axillary. Plant Name *Hibiscus vitifolius* L. Synonym *Fioria vitifolia* L. Mattei, Family Malvaceae, Local Name Siru thutthi.

Materials and Methods

Materials: Source: The flowers of *Hibicus vittifolius* L, Mattei was collected from alivalam village, Thanjavur district Figure-1.

Fabric: Degummed silk fabric was used for dyeing.

Mordants: Chemicals used: AR grade metallic salts such as copper sulphate, ferrous sulphate, aluminium sulphate, Potassium dichromate, stannous chloride was used as chemical mordants.

Experimental: Dye Extraction: 200 gram of fresh flowers was weighed and taken in soxhlet apparatus and 500 ml of solvent

(ethanol water) in the ratio 80:20 was added to it. The soxhlet apparatus was heated 70°C for 60 min. After extraction, the extract was filtered and used for dyeing.

Figure-1
***Hibicus vittifolius* L,**

Effect of M: L Ratio: The silk samples were dyed with dye extracts keeping various M: L ratio as 1:10, 1:20, 1:30 and 1:40. It was observed that the dye uptake was good in M: L ratio 1:20.

Dyeing procedure: The silk samples were dyed with dye extract keeping M: L ratio as 1:20 dyeing was carried out at 78°C and continued for 1hour.

Mordanting: The silk samples were treated and with different chemical mordants by the following three methods[7]. i. Pre-mordanting, ii. simultaneous mordanting. iii. post- mordanting

Pre-Mordanting of silk fabrics with metallic salts: Bleached silk fabrics with or without pre-mordanting were further mordanted prior to dyeing using 1%, 3% and 4% of any one of the chemical mordants, such as aluminium sulphate, ferrous sulphate, potassium dichromate, stannous chloride, copper sulphate at 60°C for 30 min with material-to liquor ratio of 1:20. The samples treated with metal salts were dyed with the dye extract.

Simultaneous -Mordanting of silk fabrics with metallic salts: Bleached silk fabrics were treated with both dye extract and metal salts simultaneously, using 1%, 3% and 4% of any one of the chemical mordants, such as aluminium sulphate, ferrous sulphate, potassium dichromate, stannous chloride, copper sulphate at 78°C for 60 min with material-to liquor ratio of 1:20

Post-Mordanting of silk fabrics with metallic salts: The wetted out silk samples were entered into different dye baths containing required amount of dye extract and water. After 10 minutes required amount of sodium carbonate was added. After 20 minutes required amount of sodium chloride was added. The dyeing was carried out for one hour at 78°C. The dyed samples were taken out, squeezed and used for treatment with metal salts process without washing. The dyed silk samples were treated with different metal salts using 1%, 3% and 4% of any one of the chemical mordants, such as aluminium sulphate, ferrous sulphate, potassium dichromate, stannous chloride, copper sulphate at 60°C for 30 min with material-to-liquor ratio of 1:20. In all the above three methods, after the dyeing is over, the dyed samples were repeatedly washed with water and then dried in air.

Color fastness: The colour fastness of the dyed fabrics was tested according to AATCC and IS standards. Color fastness to washing, light, rubbing and perspiration were determined from standard test methods. Wash fastness using Launder O Meter (AATCC – 110106), Light Fastness (AATCC test method 16-1993). Rubbing Fastness IS: 766-1956, and Acidic and Alkaline Perspiration (AATCC test method 15-1997) respectively[8].

Measurement of colour strength: The spectral reflectances of the dyed samples were measured using a Text flash spectrophotometer (Data colour corp). The K/S values were calculated by Kubelka-Munk equation.

$$K/S = (1-R)^2/2R \quad (1)$$

Where, R is the decimal fraction of the light reflectance of the dyed fabric at λmax. K is the absorption coefficient and S is scattering coefficient[9].

Antimicrobial Screening Test: Investigations were carried out to determine the antibacterial activity of the dye solution 50 mg/µl[10]. The antimicrobial (both bacterial and fungal) studies were carried out in duplicates using agar well diffusion method and a control set was along with each test to determine the antimicrobial activity[10].

Pathogenic micro organisms were purchased from trichy medical college pathology departments. The test bacteria *Staphylococcus aureus* ATCC 25923, *Escherichia coli* ATCC 25922, *Pseudomonas aeroginosa* ATCC 27853. Pathogenic bacteria culture in nutrient broth at 37°C for 24 hrs the culture were swapped on nutrient agar plates 100µl of the dye solution loaded in the well after incubation 37°C for 24 hours the zone of inhibition was measured by the taking the average of the zone obtained from the duplicate plates.

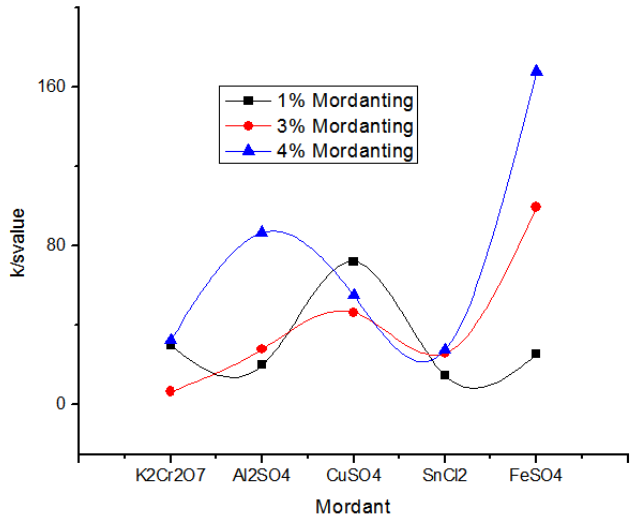
Anti fungal activity of the dye against *Aspergillusniger* and *Candida albicans* have been detected on sterile potato dextrose agar plates. Concentration of the dye in a similar way as described above. After incubation at between 25°C to 3°C for 72 hrs the zone of inhibition was measured[10].

Result and Discussion

Soxhlet Extraction Method: Two hundred gram of fresh flowers was kept in the thimble of soxhlet extractor and 500 ml of solvent (ethanol80:water20) poured in the RB flask and a condenser with high flow rate of water is fitted over it. The soxhlet apparatus was heated 70°C for 60 minutes. After extraction, the extract was filtered and used for dyeing. It was observed that, colour of the dye extract was milky yellow colour as shown in Figure-2.

Figure-2
Hibiscus vitifolius L

Effect of Mordanting: The dye extract was found to be suitable for silk fabric. The silk fabrics were dyed with chemical mordant's. In all the three dyeing method, pre mordanting method gave excellent results. In all the three dyeing, using flowers extract the mordant's ferrous sulphate show gave good results. For dyeing of silk, 1%, 3% and 4% mordant concentrations were used for present study. Among these three concentrations 4% mordant's concentration gave better results shown in Figure–3.



Figure–3
Surface colour strength (K/S values) of dyed silk fabrics using combination of mordant's

Optimization of mordants with K/S value and colour hue changes: Various hues of colour were obtained from pre mordant method in 4% mordants silk with copper sulphate, ferrous sulphate, aluminium sulphate, potassium dichromate, stannous chloride as shown in Table-1. The different mordants not only cause difference in hues of colour and significant changes in K/S values but also changes in L* Values and brightness index value. The effect of mordants on colour values of cotton dyed with flowers of *Hibiscus vittifolius* L is shown in Figure-4.

Table-2 shows L*, a* and b* K/S values and it can be seen that, mordants which show higher value of L* show lighter shades while lower L* value show darker shades for silk. Similarly, negative values of a* and b* represent green and blue respectively. Among the chemical mordants used, the highest colour value (K/S =167.917) was obtained with ferrous sulphate and lowest colour value (K/S =27.715) with stannous chloride.

Fastness properties: It was observed that, dyeing with *Hibiscus vittifolius* L gave good washing, light and rubbing fastness properties. The fastness properties of dyed of silk fabrics are shown in Table-3. Overall, it could be used for commercial purposes and attain acceptable range.

Antimicrobial Activity Studies: In this study, three different bacterial and two fungal pathogens were used to screen the possible antimicrobial activity of dye extract. Dye extract exhibited antibacterial and antifungal activity against all test microorganisms.

Antibacterial and antifungal activity: The Dye extract showed good antibacterial activity against the three bacterial pathogens. Among the three bacterial pathogens, dye extract showed more effective against Escherichia coli, Pseudomonas aeroginosa and Staphylococcus aureus pathogens as shown in Table-4 and Figure-5.

Table-1
Colour produced on silk by different 4% mordants in - pre mordanting method

Mordant's	**Colour obtained**
$K_2Cr_2O_7$	
Al_2SO_4	
$CuSO_4$	
$SnCl_2$	
$FeSO_4$	

Table-2
Different pre mordant method in 4% mordants L*, a*, b* and K/S values for silk dyed with flowers of Hibicus vittifolius L

Mordant's	L*	a*	b*	K/S Value
$K_2Cr_2O_7$	71.332	9.049	41.957	32.505
Al_2SO_4	78.028	1.480	74.442	86.804
$CuSO_4$	67.646	2.560	53.611	55.199
$SnCl_2$	82.435	-1.364	28.105	27.715
$FeSO_4$	36.186	2.203	14.926	167.917

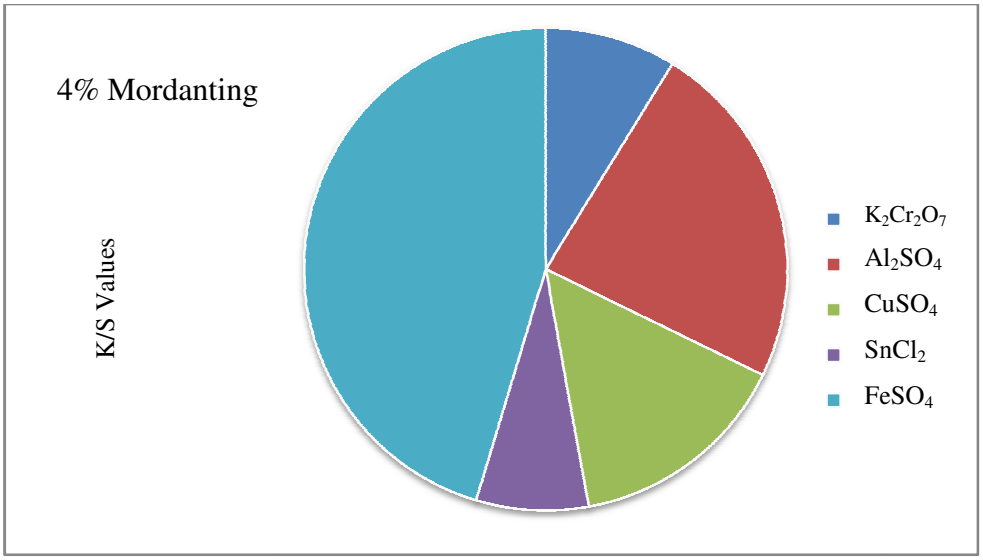


Figure-4
Effect of mordant method in 4% mordants for silk dyed with flowers of *Hibicus vittifolius* L (pre mordant)

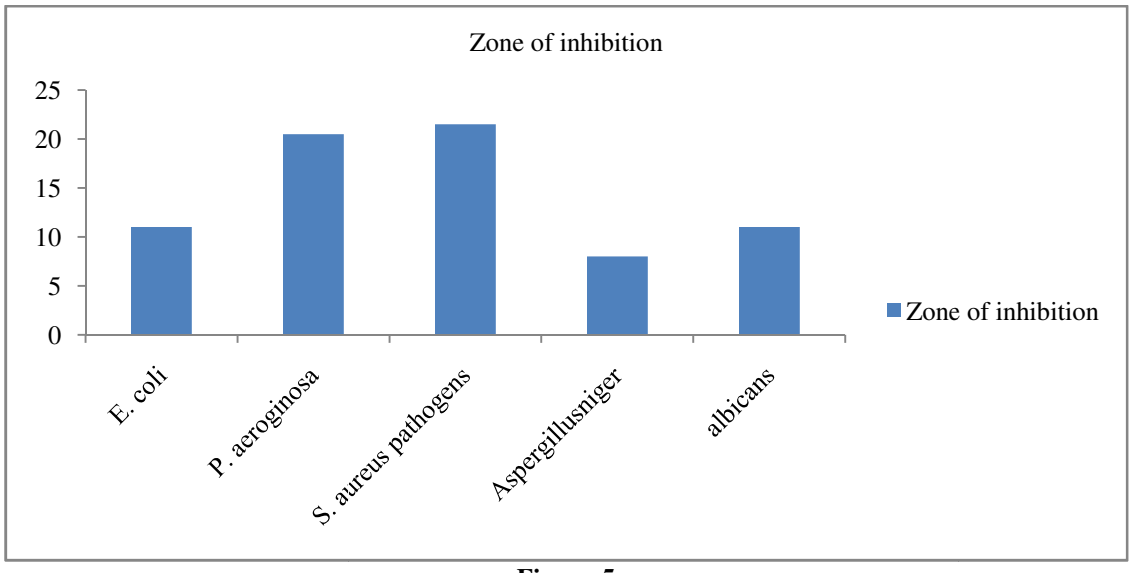


Figure-5
Antimicrobial activities of dye extract from flowers of *Hibiscus vittifolius* L

Table-3
Fastness properties for silk fabric dyed with flowers of *Hibiscus vittifolius* L

Mordant's	Method of mordanting	Mordant concentration (%)	Washing		Rubbing		Sun Light	Perspiration			
								Acidic		Alkaline	
			CC	CS	Dry	Wet		CC	CS	CC	CS
$K_2Cr_2O_7$	Pre-mordanting	1	4	5	4	4	5	3	5	3	5
		3	4	5	4-5	4	4	2	5	4	3
		4	4	5	4	4-5	4	5	5	4	5
	Simultaneous mordanting	1	4	5	4-5	4	4	5	5	4	5
		3	5	5	4-5	4	5	5	5	2	5
		4	4	5	4-5	4	5	5	5	5	5
	Post-mordanting	1	4	5	4	4	4	1	5	4	5
		3	5	5	4-5	4	5	3	5	4	5
		4	5	5	4-5	4	5	4	5	4	5
Al_2SO_4	Pre-mordanting	1	5	1	4-5	4	4	3	4	3	5
		3	2	5	4-5	4	4	3	4	4	5
		4	4	5	4-5	4	3	4	5	4	5
	Simultaneous mordanting	1	1	5	4-5	4	4	4	5	4	5
		3	5	1	4-5	4	4	4	5	4	5
		4	4	5	4-5	4	4	3	4-5	3	5
	Post-mordanting	1	1	5	4-5	4	5	4	5	5	5
		3	4	5	4-5	4	3	1	5	1	5
		4	5	4	4-5	4	4	4	5	4	5
$CuSO_4$	Pre-mordanting	1	4	5	4	3-4	4	4	4-5	4	4-5
		3	4	5	4	3-4	4	4-5	4	4	4-5
		4	4	5	4	3-4	5	4-5	4	4-5	4
	Simultaneous mordanting	1	4	5	4-5	4	5	4-5	4	4-5	4

Mordant's	**Method of mordanting**	**Mordant concentration (%)**	**Washing**		**Rubbing**		**Sun Light**	**Perspiration**			
								Acidic		**Alkaline**	
			CC	**CS**	**Dry**	**Wet**		CC	CS	CC	CS
		3	4	4-5	4-5	4	4	4	5	4	5
		4	4	4	4-5	4	4	4	5	4	4-5
	Post-mordanting	1	3	4	4	3-4	4	4	4-5	4	5
		3	3	4-5	4	3-4	5	4	4-5	4	4-5
		4	4	4-5	4-5	4	5	4	5	4	4-5
$SnCl_2$	Pre-mordanting	1	1	5	4-5	4	5	5	5	5	5
		3	4	1	4	3-4	5	4-5	4	5	5
		4	4	5	3-4	2-3	5	4	4-5	4	4-5
	Simultaneous mordanting	1	3	5	3-4	4	5	4	4	4	3-4
		3	3	5	3	5	5	4-5	4	4-5	4
		4	3	5	4-5	4	5	4	4-5	4	4-5
	Post-mordanting	1	1	5	4-5	4	4	4	4-5	4	4-5
		3	1	5	4	3-4	4	4	5	4	4-5
		4	1	5	4-5	4	5	4	4-5	4	5
$FeSO_4$	Pre-mordanting	1	4	4-5	4-5	4	5	3	4-5	1	4-5
		3	4	4-5	4-5	4	5	2	4-5	2	4-5
		4	**5**	**5**	**4-5**	**4**	**5**	**5**	**4-5**	**5**	**4-5**
	Simultaneous mordanting	1	3	5	4	3-4	5	2	5	2	5
		3	4	4-5	4-5	4	5	3	5	3	5
		4	4	5	4-5	4	5	3	5	3	5
	Post-mordanting	1	3	5	4-5	4	4	1	5	1	5
		3	4	5	4-5	4	4	1	5	1	5
		4	4	5	4-5	4	4	1	5	1	5

Table-4
Antimicrobial activities of dye extract from flowers of *Hibiscus vittifolius* L

Micro organism	Test Culture	Diameter Zone of inhibition (mm)
Bacteria	*Escherichia coli*	11
	Pseudomonas aeroginosa	20.5
	Staphylococcus aureus pathogens	21.5
Fungal	*Aspergillusniger*	8
	Candida albicans	11

Conclusion

The dyeing of silk can achieved using the flower extracts of *Hibiscus vittifolius* L by using chemical mordant's. The washing, light and rubbing fastness of all dyeing with mordant's were quite good and also dye extract has shown good antimicrobial activity. From the comparative study of fastness properties the dyed silk samples *Hibiscus vittifolius* L in pre mordanting method with 4% (ferrous sulphat) mordant combination gives better results. From this result it was concluded that the dye exracted from the Hibiscus vittifoliusL was most suitable for dyeing of silk and it is also used in medicinal textiles.

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