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Extraction and dyeing conditions of natural dye from flowers of *Plumeria rubra* L. on textiles and fastness properties

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Eco-friendly dye was extracted from the flowers of *Plumeria rubra.* This dye was applied on cotton and silk fabrics with 2%, 5% and 10% o.w.f. dye concentrations in the presence and absence of alum, tannic acid and tartaric acid mordants using the exhaustion method. The color strength, CIE lab values and fastness properties of the dyed samples were assessed. The mordants were found to influence the colour strength and fastness properties of dyed cotton and silk fabrics. Dyed samples were tested for antimicrobial activity and found to possess antibacterial activity. The flowers of *Plumeria rubra* offer a potential source of natural dye for cottage dyeing industry.

Keywords: *Plumeria rubra*, Quercetin, Microbes, Cotton, Silk, Dyeing

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In recent times, the textile dye industry has been forced to stop the production of potentially dangerous dyes and subsequently reduce the toxic effluents. Natural dyes derived from flora are safe because of their non-toxic, biodegradable and non-carcinogenic characteristics. They do not cause pollution and waste water problems and represent a more environmentally friendly alternative[1-5]. A large number of plant and animal/insect sources have been identified for extraction of color to be used in the textile industry[6,7]. In India, some 500 varieties of plants that can yield natural dyes are available[8]. *Plumeria rubra* L. is a small/medium sized, deciduous tree belonging to the Apocynaceae family, commonly known as "Temple Tree" or "*Champa*" in India. It is an ornamental tree and is commonly grown in gardens. It is found in abundance in Central and South America, although it is grown worldwide, and is prominently found in Hawaii and the Pacific Islands. The flowers are also used in pectoral syrups. The flavonoid present in flowers is Quercetin[9] (Fig. 1). The present work aims at studying the dyeing potential of *Plumeria rubra* flowers extract on silk and cotton and its antimicrobial activity.

Material and methods

Raw materials

Fresh flowers of *Plumeria rubra* (2 Kg) were collected from Lalbagh Botanical Gardens, Bangalore, India, during the summer season. The flowers were washed well with tap water and dried at room temperature. The dried flowers were cut into small pieces and then ground to a fine powder.

Dye extraction

Fresh *Plumeria rubra* flowers were dried at room temperature till complete dry and then crushed using a mixer grinder and then used as the raw material for dye extraction. Optimization of extraction was performed at different temperatures (30, 50, 80, 90 and 100 °C) for different *p*H (5, 7, 8, 9 and 10) and for different durations (45, 60, 75, 90 and 120 min). The extracted dye was filtered and cooled to room temperature.

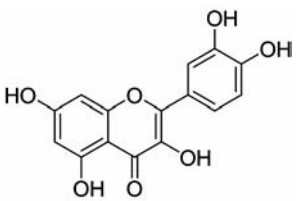

Fig. 1—Quercetin

*Corresponding author

Method of mordanting

Pre- and post-mordanting methods using 2, 5 and 10% (owf) solutions each of potassium aluminum sulphate, tannic acid and tartaric acid were employed at M:L ratio 1:20 and mordanting was carried out for 30 min at 60 °C. In the case of simultaneous mordanting, mordant was added during dyeing along with the dye bath. The fabrics were then washed and dried.

Dyeing of cotton and silk fabrics

Plain, woven, degummed mulberry silk fabric weighing 40 gm/m^2 with a yarn density of 132 ends/inch and 116 picks/inch and plain woven, scoured cotton fabric weighing 110 gm/m^2 with a yarn density of 158 ends/inch and 106 picks/inch were used for dyeing.

Colour strength measurement

Colour strength value was measured using reflectance measurement. Colour values were evaluated by means of K/S and CIELAB colour difference values with illuminant $D_{65}/10^0$ observer on Gretag Macbeth Color Eye 7000 A Reflectance Spectro photometer. Four measurements were made for each sample and the variation in percentage reflectance values over a range of 350-750 nm was recorded. The K/S values were assessed using the Kubelka-Munk equation[10,12]:

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

Where, R is the observed reflectance, K, the absorption coefficient and S, the light scattering coefficient.

Colour fastness properties

The light, washing, rubbing and perspiration fastnesses of the dyed samples were determined according to ISO 105 B02, ISO 105 C02, ISO-X12 and ISO 105 E04 standards, respectively.

Antimicrobial activity

Cultures of the following microorganisms were used in the study: *Escherichia coli, Basillus subtilis, Klebsiella pneumoniae, Pseudomonas aeruginosa, Kerria lacca, Erwina carotovora, Candida albicans* and *Staphyloccus aureus.*

Antimicrobial screening test

Nutrient agar medium (gm/L: peptone 5.0; beef extract 1.5; yeast extract 1.5; NaCL 5.0: agar 20; *p*H 7.5) was prepared and autoclaved at 121°C for 20 min. Sterilized petriplates were prepared with an equal thickness of nutrient agar. Test organisms were grown overnight at 37 ˚C, 120 rpm in 10 mL nutrient broth. This broth was used for seeding the agar plates[13-15].

Results and discussion

Optimization of dye extraction

Fig. 2 shows the UV-Vis absorbance curves of dye at *p*H varying from 3 to 9. The maximum absorbance peak (λ_{max}) was detected at 400 nm. The shapes of the UV-Vis absorbance curves of dye liquor are different in between *p*H 3 and *p*H 9. The absorbance in visible range 350-500 nm was obliviously higher and the color of the dye liquor distinctively became deeper and darker with increasing *p*H. From several sets of experiments, it was observed that the yield of the dye extract was better at *p*H 9 (Fig. 3), duration 90 min at 90 °C temperature.

Effect of dyeing conditions

Fig. 4 shows that the *p*H of the dye bath has a considerable effect on the dye ability of silk fabric. As the *p*H increases, the dye ability decreases; the effect of the dye bath *p*H can be attributed to the

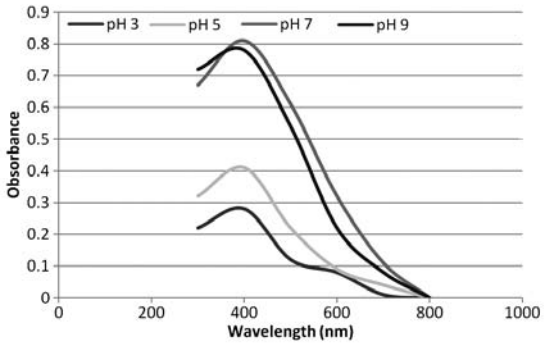

Fig. 2—UV-Vis absorbance curves of the dye

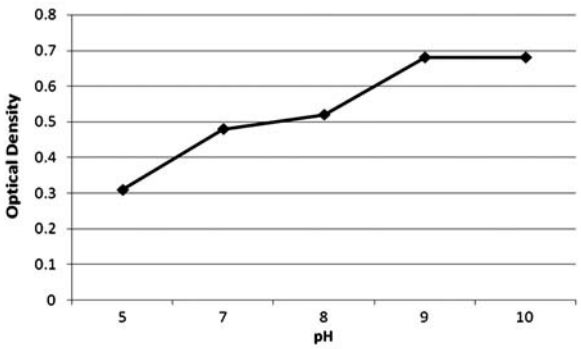

Fig. 3—Effect of *p*H on dye extraction

correlation between dye and silk fibers. Since the dye used is water soluble containing anionic groups, it would interact ionically with the protonated terminal amino groups of silk fibers at acidic *p*H via ion-exchange reaction[16].

Evaluation of colour co-ordinates of dyed samples

The K/S values increased with the increasing concentration of mordants in both silk and cotton samples. Pre-mordanting with alum and post mordanting with tartaric acid gave improved K/S values in case of cotton as compared to other samples. However in case of silk all the mordants showed an improvement in K/S values compared to unmordanted sample. No significant difference was observed among the mordanted samples. It was also evident that a wide variety of shades ranging from yellowish brown to dark brown could be obtained from the dye when mordanted with different mordants as observed from the colour co-ordinates a* and b*. The colour co-ordinates along with K/S values of cotton and silk fabrics dyed with flower extract of *Plumeria rubra* are shown in Tables 1 & 2. It is observed that all the colour coordinates are positive with respect to red-green (a*), and yellow-blue (b*) and therefore, all of them lie in the yellow-red quadrant of the colour space diagram. The colour fastness properties of all the dyed samples were found to be satisfactory. The fastness properties of cotton and silk fabrics dyed with the flower extract are presented in Tables 3 & 4, respectively.

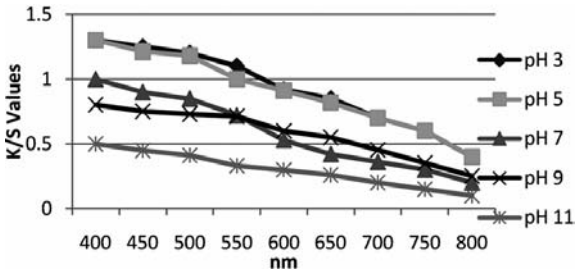

Fig. 4—Effect of dye bath *p*H on color strength of dyed silk fabrics

Table 1—K/S and color co-ordinates of cotton samples dyed with *Plumeria rubra* flower extract

Sl No	Mordant	Method	% (owf)	K/S	L	a*	b*	C	h
Control		---	---	1.01	83.28	3.72	7.69	8.54	64.19
01	A	Pre	2	2.42	78.82	3.33	3.55	4.86	46.81
02			5	3.27	74.88	4.23	15.01	15.64	74.09
03			10	4.02	70.62	4.35	16.4	16.97	75.14
04		Sim	2	2.38	79.17	3.36	7.56	8.24	65.93
05			5	2.42	78.83	3.12	7.12	8.35	67.82
06			10	2.52	78.11	2.89	8.02	8.52	70.17
07		Post	2	2.30	81.86	3.44	6.36	7.25	61.32
08			5	2.65	76.45	3.41	5.99	6.95	60.32
09			10	2.97	75.36	3.36	5.79	6.71	59.92
10	B	Pre	2	2.44	78.52	2.56	6.97	7.43	69.83
11			5	2.65	76.44	3.29	9.14	9.71	70.19
12			10	3.91	71.99	3.33	8.51	9.14	68.64
13		Sim	2	2.67	76.15	3.59	9.11	9.72	68.54
14			5	2.95	75.49	3.48	11.52	12.56	75.92
15			10	3.27	74.88	3.45	13.79	14.22	64.52
16		Post	2	2.54	78.06	3.68	7.77	8.53	64.50
17			5	2.63	77.91	3.68	6.59	7.55	60.82
18			10	3.74	72.56	3.83	8.34	9.18	65.33
19	C	Pre	2	2.64	76.55	3.41	8.37	9.04	67.81
20			5	2.98	75.25	3.72	9.35	10.02	68.19
21			10	3.00	75.04	3.59	8.91	9.61	68.04
22		Sim	2	2.63	76.65	3.31	11.88	12.34	74.42
23			5	2.71	76.13	3.46	12.83	13.29	74.93
24			10	2.78	75.54	3.27	15.98	16.31	78.43
25		Post	2	3.37	73.70	3.79	6.89	7.87	61.19
26			5	3.42	72.85	3.59	6.51	7.44	61.07
27			10	4.09	70.55	3.69	6.10	7.13	58.84

A: alum; B: tannic acid; C: tartaric acid, Pre: pre-mordanting; Sim: simultaneous mordanting and Post: post mordanting

owf: on the weight of fabric, L: Lightness, a*: + red & - green, b*: + yellow & - blue, C- chroma, h- hue angle.

Table 2—K/S and color co-ordinates of silk samples dyed with *Plumeria rubra* flower extract

Sl No	Mordant	Method	% (owf)	K/S	L	a*	b*	C	h
Control		---	---	3.51	76.65	4.32	14.74	15.34	73.48
01	A	Pre	2	6.84	63.23	4.24	18.45	18.93	76.94
02			5	6.60	63.92	4.46	18.53	19.19	77.19
03			10	6.60	63.91	4.60	19.38	19.81	77.89
04		Sim	2	6.60	63.90	5.03	13.83	14.76	69.90
05			5	6.84	63.23	5.35	13.98	14.53	69.08
06			10	6.90	62.94	5.54	14.49	14.22	68.86
07		Post	2	6.38	64.20	4.45	14.34	15.00	72.62
08			5	6.60	63.89	4.53	15.13	15.89	73.25
09			10	6.94	62.86	4.55	15.31	16.08	73.53
10	B	Pre	2	4.15	73.94	2.80	13.83	14.10	78.58
11			5	5.13	66.15	3.38	15.98	16.21	78.10
12			10	6.28	64.52	4.13	18.14	18.62	76.99
13		Sim	2	6.60	63.91	5.11	13.60	14.56	69.34
14			5	6.72	63.49	5.21	13.66	14.55	68.73
15			10	6.96	62.73	5.33	13.27	14.23	68.02
16		Post	2	6.72	63.48	4.43	14.93	15.61	73.33
17			5	6.96	62.74	4.74	15.41	16.17	73.00
18			10	7.09	61.51	4.97	15.80	16.52	72.56
19	C	Pre	2	7.22	61.37	4.10	18.19	18.50	77.07
20			5	7.22	61.35	4.09	18.84	19.33	77.88
21			10	7.41	61.01	4.08	18.96	19.43	77.83
22		Sim	2	6.96	62.75	4.96	14.73	15.58	71.46
23			5	6.96	62.74	5.39	14.52	15.54	69.99
24			10	6.96	62.75	5.33	14.60	15.56	67.00
25		Post	2	6.26	66.05	4.74	15.08	15.83	72.61
26			5	6.49	64.15	4.56	15.07	15.72	73.00
27			10	7.32	61.16	4.31	14.86	15.61	74.19

A: alum; B: tannic acid; C: tartaric acid, Pre: pre-mordanting; Sim: simultaneous mordanting and Post: post mordanting
owf: on the weight of fabric, L: lightness, a*: + red & - green, b*: + yellow & - blue, C- chroma, h- hue angle.

Table 3—Fastness properties of samples dyed with cotton

Sl No	Mordants	Method	% (owf)	Light fastness	Wash fastness		Perspiration fastness				Rubbing fastness	
							Acidic		Alkali		Dry	Wet
					CC	CS	CC	CS	CC	CS		
Control		---	---	2	4	5	3-4	5	3-4	5	5	4
01	A	Pre	2	3	5	5	3	5	4	5	5	4-5
02			5	3	4	5	3	5	4	5	5	4-5
03			10	4	4	5	3-4	5	4	5	5	4
04		Sim	2	2	4	5	4-5	5	4-5	5	5	4-5
05			5	2	4	5	4-5	5	4-5	5	5	4-5
06			10	3	4	5	4-5	5	4-5	5	5	4
07		Post	2	3	4-5	5	4-5	5	4	5	5	4-5
08			5	3	4-5	5	4-5	5	4-5	5	5	4-5
09			10	4	4-5	5	5	5	5	5	5	4
10	B	Pre	2	3	4	5	4-5	5	4-5	5	5	4-5
11			5	3	4	5	4-5	5	4-5	5	5	4-5
12			10	4	4	5	4-5	5	4-5	5	5	4
13		Sim	2	2	4	5	4	5	4	5	5	4-5
14			5	2	4	5	4	5	4-5	5	5	4-5
15			10	2	4	5	4	5	4-5	5	5	4
16		Post	2	3	4	5	5	5	5	5	5	4
17			5	3	4	5	5	5	5	5	5	4-5
18			10	3	4-5	5	5	5	5	5	5	4-5

(*Contd.*)

Table 3—Fastness properties of samples dyed with cotton (*Contd*.)

Sl No	Mordants	Method	% (owf)	Light fastness	Wash fastness		Perspiration fastness				Rubbing fastness	
							Acidic		Alkali		Dry	Wet
					CC	CS	CC	CS	CC	CS		
19			2	3	4	5	5	5	5	5	5	4-5
20			5	3	4	5	5	5	5	5	5	4-5
21			10	4	4	5	5	5	5	5	5	4-5
22		Sim	2	2	3	5	4	5	4	5	5	4
23			5	2	3	5	4	5	4	5	5	4
24			10	4	3	5	4	5	4	5	5	4
25		Post	2	2	4	5	5	5	5	5	5	4-5
26			5	4	4	5	5	5	5	5	5	4-5
27			10	4	4-5	5	5	5	5	5	5	4-5

A: alum; B: tannic acid; C: tartaric acid, Pre: pre-mordanting; Sim: simultaneous mordanting and Post: post mordanting

CC: Colour change, CS: Colour staining

Table 4—Fastness properties of samples dyed with silk

Sl No	Mordants	Method	%(owf)	Light fastness	Wash fastness		Perspiration fastness				Rubbing fastness	
							Acidic		Alkali		Dry	Wet
					CC	CS	CC	CS	CC	CS		
Control		---	---	1	4	5	4	5	4	5	5	4
01	A	Pre	2	3	4	5	4	5	5	5	5	4
02			5	3	4	5	4	5	5	5	5	4
03			10	4	4-5	5	3-4	5	5	5	5	4
04		Sim	2	2	4	5	4	5	4-5	5	5	4
05			5	2	4	5	4	5	4-5	5	5	4-5
06			10	4	4	5	4-5	5	4	5	5	4-5
07		Post	2	2	4	5	4	5	4-5	5	5	4-5
08			5	4	4	5	4-5	5	4-5	5	5	4-5
09			10	4	4	5	4-5	5	4	5	5	4-5
10	B	Pre	2	2	4	5	3-4	5	4	5	5	4-5
11			5	3	4	5	3-4	5	4	5	5	4-5
12			10	4	4	5	3-4	5	4	5	5	4-5
13		Sim	2	2	4	5	4	5	4	5	5	4-5
14			5	2	4	5	4	5	4	5	5	4-5
15			10	2	4-5	5	4	5	4	5	5	4-5
16		Post	2	2	4	5	4-5	5	4	5	5	4-5
17			5	4	4-5	5	4-5	5	4	5	5	4-5
18			10	4	4-5	5	4-5	5	4	5	5	4-5
19	C	Pre	2	2	4	5	3-4	5	4	5	5	4-5
20			5	3	4	5	3-4	5	4	5	5	4-5
21			10	4	4	5	3-4	5	4-5	5	5	4-5
22		Sim	2	2	4	5	4	5	4	5	5	4-5
23			5	2	4	5	4	5	4-5	5	5	4-5
24			10	2	4	5	4	5	4-5	5	5	4-5
25		Post	2	2	4	5	4-5	5	4	5	5	4-5
26			5	4	4	5	4-5	5	4	5	5	4-5
27			10	4	4-5	5	4-5	5	4-5	5	5	4-5

A: alum; B: tannic acid; C: tartaric acid, Pre: pre-mordanting; Sim: simultaneous mordanting and Post: post mordanting

CC: Colour change, CS: Colour staining

Table 5—Zone of inhibition for *Plumeria rubra* dye against selected microbes

Sl No	Microbes	Dye conc.	Zone of inhibition (diameter in cm)
1	*Escherichia coli*	5	-
		10	-
		15	-
		20	-
2	*Basillus subtilis*		
		5	-
		10	-
		15	-
		20	-
3	*Candida albicans*		
		5	-
		10	0.25
		15	0.3
		20	0.3
4	*Pseudomonas aeruginosa*		
		5	-
		10	-
		15	-
		20	-
5	*Kerria lacca*		
		5	-
		10	-
		15	-
		20	-
6	*Erwina carotovora*		
		5	-
		10	-
		15	-
		20	0.2
7	*Klebsiella pneumoniae*		
		5	-
		10	-
		15	-
		20	-
8	*Staphyloccus aureus*		
		5	-
		10	-
		15	-
		20	-

conc: concentration

Antimicrobial activity of *Plumeria rubra* dye extract on substrate

The effect of concentration of dye on antimicrobial activity was studied further and results are summarized in Table 5. The zone of inhibition (diameter) was recorded in each case. It was observed that increase in dye concentration led to increased inhibition reflected by enhancement in diameter only in case of *Candida albicans*. It may be concluded that the extracted dye from *Plumeria rubra* flowers used on cotton substrate were slightly effective against only few microbes.

Conclusion

The extracts of *Plumeria rubra* flowers as a new source of natural dye showed good results on cotton and silk fabrics. The use of different mordants proved the effectiveness of the dye fixation as seen from the fastness results. The flowers are available abundantly, are eco-friendly, inexpensive and hence can be used for dyeing in cottage and in small scale industries, thereby giving better employment opportunities to rural folks and also helping them to promote rural entrepreneurship for development of rural economy. It also encourages usage of waste lands for cultivation of these plants yielding dyestuff, afforestation of wasteland and provides consequent additional source of income to rural population. The mordants used in the present study are eco-friendly in nature; and hence will not lead to pollution problems that are usually caused by the use of heavy metals. The dye extracted from *Plumeria rubra* flower can be efficiently used for producing value added environment friendly silk apparel and other textile products.

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