Dyeing and flame retardant finishing of silk fabric: an ecofriendly approach

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Abstract

In the current context of environmental contamination due to disposal of textile-manufacturing waste, substitution of synthetic dyes and chemicals with bio-colour or bio-extract is a big confront. Thus, the need of the hour is to develop green chemicals or dyes for sustainable textile processing with an eco-friendly approach. In light of above, a bio-colour has been extracted from *Bauhinia vahlii* bark and applied on the degummed and bleached silk fabric along with a bio-acid (citric acid). Dyed silk fabrics were tested for their color strength and fastness properties like color fastness to washing, water crocking and light and found satisfactory results. Banana peduncle extract (BPE) is also applied on the dyed fabric to improve the fastness properties and enhance the functional properties like flame retardant by pad-dry-cure method. Dyed and BPE treated silk fabric was tested and analysed for their color fastness properties and mechanical properties, like thickness, bending length, flexural rigidity and crease recovery and the results were recorded. Notably, evaluation of dyed and BPE treated silk fabric was carried out for flame retardancy according to ASTM D1230 standards and promising result was obtained.

Keywords Silk · *Bauhinia vahlii* bark · Natural dye · Banana peduncle extract · Flame retardant

1 Introduction

The textile industry is the major consumer of synthetic dyes used for dyeing and printing of textile material [1]. Synthetic dyes are hazardous, carcinogenic to human body, destroying eco-system and toxic to aquatic biodiversity [2]. Due to the current environmental consciousness, extraction of colorants from natural substrates and its application on textiles has drawn considerable attention in recent past. Several studies were carried out on the extraction of natural dyes and its application on textile material. Demand of natural dyes is increasing continuously as their extraction and application on textile material does not require strong acids and alkalis. Moreover, natural dyes are biodegradable, have wide variety non-toxic, non-carcinogenic, easily available and renewable, eco-friendly; provide

a wide range of shades with acceptable levels of color fastness properties [3]. Now, it is the need of the hour to use natural colorants in textile application instead of synthetic dyes. Coloring substances can be extracted from the different parts of the plant like roots, barks, leaves, fruits and flowers and also from the other natural sources. Natural colorants are not only used for their medicinal value and anti-oxidant properties but also to enhance the aesthetic value of the textile product [4, 5]. Therefore, to address some of these issues this study was aimed to dye silk fabric with eco-friendly natural dye extracted from bark of *Bauhinia vahlii* plant.

B. vahlii Plant belongs to the class Angiospermae and family Leguminosaceae [6]. It is a giant climber and one of the most abundant Indian Bauhinia species. The plant is distributed in the Sub-Himalayan region and in Assam,

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Central India, Bihar, Odisha, Eastern and Western Ghats of India. Their leaves and stem barks have been used frequently in folk medicine as remedies for the treatment of diabetes, infections, pains and inflammatory processes and have been confirmed through numerous biological studies [6, 7].

B. vahlii bark is composed of ligno cellulose and its aqueous extract contains different phyto constituents, such as alkaloids, carbohydrates, glycosides, saponins, flavonoids and triterpenes, tannins and anthraquinones. Presence of tannins and anthraquinones in *B. vahlii* bark extract indicates that it can be used as coloring material. [4, 8]. Most of the natural dyes contain –OH group in their structure and can be esterified with a non-polymeric polycarboxylic acids such as citric acid [5]. In this present study silk fabric is dyed with *B. vahlii* bark extract with citric acid (Fig. 1).

Silk, known as “the queen of fibers” worldwide is an exclusive textile material for its lusture, wearing comfort ability and drapability [9]. Textile materials with different functional properties like flame retardant, antimicrobial is in great demand nowadays and also do value addition to the product. The high nitrogen and sulphur content of silk fabric makes it less flammable than other fibres. However, it still cannot meet the strict compliance of fire safety regulations for flame retardant textile and apparel [10]. Use of synthetic chemicals to impart flame retardant property in the silk fabric retard sustainability of textile processing. Some of the plants contain phosphate, chloride, silicate and other

minerals, metal oxides and mineral salts; they have potential to be utilized to impart flame retardancy to the cellulose and other textile materials [11].

In the study developed by Gérard et al. [12], the mineral analysis of the banana peduncle extract revealed the presence of high amount of Nitrogen (9.6 mg/g), Calcium (84.67 mg/100 g), Sodium (363.16 mg/100 g), potassium (83.67 mg/100 g), Phosphorus (0.058 mg/100 g), iron (0.077 mg/100 g), and magnesium (39.05 mg/100 g). Due to presence of such high mineral content banana peduncle extract has potential to act as a flame retardant. The aim of the present study was to improve the fastness properties of dyed silk fabric and also to impart flame retardant finish, by dyeing the silk fabric with *B. vahlii* bark extract and treated with BPE.

2 Experimental

2.1 Materials

A 40 GSM silk habutai plain woven fabric was sourced from the local fabric market of Bhubaneswar, India having ends/ inch and picks/inch 120 and 104 and warp and weft yarn count 22 and 55 denier, respectively. *B. vahlii* bark was collected from Simlipal reserve forest, Odisha, India. Sodium carbonate of Ramken was purchased from Bhubaneswar, India. Lemon juice was collected from fresh lemon purchased from local market. Banana peduncle was sourced from the local farmer of Bhubaneswar.

2.2 Bark extract of *B. vahlii* in aqueous medium

B. vahlii Bark was dried in shed at room temperature for 7–10 days, cut into small pieces and grinded in a home grinding machine. The grinded fibrous powder was soaked in the distilled water for 30 min in 6 different pots with material to liquor ratio 1:20. Anhydrous Na_2CO_3 was added in the pots and heated for 2 h as per the conditions mentioned in the Table 1. Extraction process was carried out in the water bath. Extracts were collected in six different pots by neutralization with citric acid and named as dye solution 1, dye solution 2, dye solution 3, dye solution 4, dye solution 5 and dye solution 6 according to the pot number. Figure 2 shows the extraction process from collection of bark to drying of the bark and then grinding and extraction in aqueous medium. Figure 3 shows color spectrum of *B. vahlii* bark

Fig. 1 *B. vahlii* plant [13]

Table 1 Temperature and Na_2CO_3 concentration for aqueous extraction from *B. vahlii* bark

	Pot 1	Pot 2	Pot 3	Pot 4	Pot 5	Pot 6
Temperature		60 °C			90 °C	
Na_2CO_3 (on the weight of bark)	Nil	2%	4%	Nil	2%	4%

Fig. 2 Bark extract of *B. vahlii* in aqueous medium from plant to solution [14]

Fig. 3 Color spectrum of *B. vahlii* bark extract in aqueous medium (spectrophotometer images). Note: deg degree Celsius, N Neutral pH, % Na_2CO_3 sodium carbonate aqueous solution (weight/volume)

extract dye solutions in spectrophotometer. Difference in coloring substance concentration is clearly visible from the color spectrum.

2.3 Application of dyes

Degummed and bleached silk fabric was dyed with all the six dye solutions extracted from *B. vahlii* bark in different temperature as mentioned in Table 2. Extracted solutions were used directly without any dilution, having material to liquor ratio 1:30. Dyeing was carried out on paramount water bath without any mordanting agent for 1 h. pH of the bath was adjusted to 2 by using citric acid i.e. lemon juice. Dyed samples were rinsed with cold and hot water to remove the superficial dyes. Total 12 samples were developed from the 6 different dye solutions.

2.4 Extraction process of banana peduncle extract

Collected Banana peduncle was cleaned and washed properly with running water and cut into pieces of 15 to 18 inches. Peduncle pieces were further sliced

lengthwise having 2 to 3 mm in thickness and passed through Laboratory Padding Mangle. Banana peduncle extract was collected from the padding mangle tray and stored in a container. The pH of the BPE was 5.35 at the time of extraction.

2.5 Application of banana peduncle extract

To improve the fastness properties and also to enhance the functional properties i.e. flame retardancy of the dyed fabric, sample number 11 was chosen and treated with Banana peduncle extract with pad-dry-cure method. Dyed fabric sample was impregnated in the banana peduncle extract and passed through the padding mangle to remove the excess solution and also to control the add-on percentage of the banana peduncle extract. The sample was dried for 5 min at 80 °C and then cured at 140 °C for 3 min.

The weight gain % i.e. percentage increase in sample weight was calculated as per the following equation.

$$\text{Weight gain \%} = \left[(W_2 - W_1) / W_1 \right] \times 100 \dots \dots \dots$$

for silk fabric dyed with BV bark extract

$$\text{Weight gain \%} = \left[(W_3 - W_1) / W_1 \right] \times 100 \dots \dots \dots$$

for dyed silk fabric treated with BPE

where W_3 , W_2 AND W_1 are the oven dry weight of dyed silk fabric treated with BPE, silk fabric dyed with *B. vahlii* bark extract and control silk fabric, respectively [10].

Table 2 Dyeing temperature using different dye solutions

Temperature	Dye solution 1	Dye solution 2	Dye solution 3	Dye solution 4	Dye solution 5	Dye solution 6
Room temp.	✓	✓	✓	✓	✓	✓
60 °C	✓	✓	✓			
90 °C				✓	✓	✓

3 Measurement

3.1 Color strength

Color strength of the *B. vahlii* bark extracts in aqueous medium were measured in Xrite color i7 Spectrophotometer in transmittance mode. The silk fabric samples dyed with *B. vahlii* bark extracts in different conditions were also checked for their color strength with the help of the same spectrophotometer in reflectance mode.

3.2 Fastness properties

Silk fabric samples dyed with *B. vahlii* bark extracts were tested for their colorfastness properties. Samples were tested for colorfastness to washing, Water, crocking and light as per the ISO 105 C06, ISO 105 E01, ISO 105 X12 and ISO 105 B02 respectively.

3.3 Flammability assessment

Control silk fabric, dyed silk fabric (sample no 11), undyed silk fabric treated with BPE and dyed silk fabric (sample no 11) treated with BPE were tested for their response to heat and flame under controlled condition. Here sample no 11 was selected randomly for BPE treatment and further flammability assessment. All the Samples were tested for flammability in Digiflame, 45° flammability tester of Paramount as per ASTM D1230. 10 samples were tested for each category. Samples size were 150 mm × 50 mm. Flame spread time, burning rate and char length were measured during testing.

3.4 Mechanical properties

Silk fabric dyed with *B. vahlii* bark extract and silk fabric dyed with *B. vahlii* bark extract and treated with BPE were

tested for their mechanical properties like thickness, bending length, flexural rigidity and crease recovery. Testing was conducted inside institute's laboratory under standard atmospheric condition.

4 Results and discussion

4.1 Bark extracts color strength

Table 3 shows the percentage color strength of *B. vahlii* bark extract, in aqueous medium and the data reveals that there is a remarkable increase in color strength, with increase in extraction temperature and pH of the bath. Color strength value was increased up to 760%, when extraction was done at 90 °C with 4% Na₂CO₃ as compared to extraction at 60 °C in neutral condition. It is also clear from the table that percentage color strength of the *B. vahlii* bark extract is directly proportional to the extraction temperature and Na₂CO₃ concentration of the extraction bath. This was happened may be due to presence of lignocelluloses in the *B. vahlii* bark. Figure 4 shows that solⁿ 1 (60 deg N) transmit maximum light through the dye solution as it is the lightest one among all. Whereas solⁿ 6 transmit very less amount of light through the solution due to presence of more coloring material in the solution as compared to others.

4.2 Color strength of dyed sample

Figure 5 shows the samples, dyed with different extract solutions in different temperature to get wide range of shades, when applied on silk fabric in exhaust method. It is clear from the images that the dyed samples are very bright in color and lustrous, which can be converted into attractive products for practical application. Percentage color strength of all the dyed samples was measured in spectrophotometer and the results are tabulated in

Table 3 Percentage color strength of *B. vahlii* bark extracts in aqueous medium

Sol ⁿ no	Standard name	L*	a*	b*	c*	h°	
1	60 deg N	97.58	−0.07	5.22	5.22	90.8	
	Trial name	DL*	Da*	Db*	DC*	DH*	%STR-SWL
4	90 deg N	−1.52 D	0.55 R	4.29 Y	4.30 B	−0.45 R	166.95
2	60 deg 2% Na ₂ CO ₃	−7.02 D	2.41 R	10.79 Y	10.96 B	−1.46 R	292.45
3	60 deg 4% Na ₂ CO ₃	−13.46 D	5.24 R	21.86 Y	22.35 B	−2.43 R	491.92
5	90 deg 2% Na ₂ CO ₃	−17.03 D	5.95 R	25.08 Y	25.64 B	−2.60 R	642.22
6	90 deg 4% Na ₂ CO ₃	−19.23 D	9.31 R	31.82 Y	32.96 B	−3.64 R	760.42

Solⁿ no dye solution, L* value of lightness and darkness, a* value of red and green, b* value of yellow and blue, c* value of chroma, h° value of hue, DL* difference in lightness and darkness, Da* difference in red and green, Db* difference in yellow and blue, DC* difference in chroma, DH* difference in hue, %STR-SWL single wavelength strength percentage

Fig. 4 Percentage transmittance curve of *B. vahlii* bark extracts in aqueous medium

Silk Fabric samples dyed with *Bauhinia Vahlia* Bark Extract

Fig. 5 Silk fabric samples dyed with *B. vahlii* bark extract in different temperature

Table 4. Results show that there is remarkable improvement in shade depth. Data indicate that sample no 12 is 400% darker than the sample no 1. Moreover, data also shows that the shade depth is increased from first to last sample uniformly.

4.3 Fastness properties

Table 5 illustrates the color fastness properties of silk fabric samples dyed with *B. vahlii* bark extracts. Out of 12 samples, 3 samples were selected on basis of shade depth for the testing purpose. Sample number 1 exhibits excellent result in color fastness to washing, water and crocking. However, sample shows moderate result in colorfastness to light test, may be due to its lower shade depth. Sample number 5 evidences very good results against all tests. In case of sample number 11, color change in washing gives moderate result, though it is accepted as per

the international standards. Other results are very much accepted as per the standard norms.

Table 6 demonstrates the improvement in color fastness properties of sample number 11 after BPE treatment against its original sample. Results show that there is significant improvement in fastness properties.

4.4 Flammability test

Table 7 shows the weight gain percentage of dyed and BPE treated fabric as well as 45° flammability test results. Weight of the dyed silk fabric is increased by 2.63%, whereas dyed and BPE treated silk fabric gain 7.89% weight compared to the control silk fabric. Flammability testing has been done as per ASTM D1230 of control silk fabric, silk fabric dyed with *B. vahlii* bark extract, undyed silk fabric treated with BPE extract and silk fabric dyed with *B. vahlii* extract and treated with BPE. Flame spread time data of control silk fabric and dyed silk fabric indicate that,

Table 4 Percentage color strength of silk fabrics dyed with *B. vahlii* bark extract

Sample no	Standard name	L*		a*	b*		C*	h°
1	60 deg N Cold	85.97		1.86	4.54		4.9	67.74
	Trial name	DL*	Da*	Db*	DC*	DH*	CV-SWL	%STR-SWL
7	60 deg N/60 deg	− 1.78 D	1.92 R	3.32 Y	3.81 B	− 0.39 R	1.019	114.51
2	90 deg N/Cold	− 2.35 D	0.47 R	4.67 Y	4.59 B	0.96 G	1.235	138.83
4	60 deg 2% Na ₂ CO ₃ /Cold	− 5.07 D	2.83 R	8.15 Y	8.62 B	0.28 G	1.235	138.83
3	60 deg 4% Na ₂ CO ₃ /Cold	− 6.71 D	5.16 R	11.60 Y	12.7 B	− 0.20 R	1.3	146.11
5	90 deg 4% Na ₂ CO ₃ /Cold	− 8.35 D	6.22 R	10.59 Y	12.24 B	− 0.94 R	1.512	170
6	60 deg 2% Na ₂ CO ₃ /60 deg	− 12.07 D	6.44 R	9.78 Y	11.64 B	− 1.23 R	1.652	185.65
8	90 deg 2% Na ₂ CO ₃ /Cold	− 7.49 D	3.44 R	10.56 Y	11.10 B	0.45 G	1.659	186.5
13	90 deg N/90 deg	− 12.40 D	6.21 R	11.65 Y	13.19 B	− 0.69 R	1.804	202.76
9	60 deg 4% Na ₂ CO ₃ /60 deg	− 15.94 D	9.91 R	14.80 Y	17.73 B	− 1.67 R	2.064	231.98
11	90 deg 2% Na ₂ CO ₃ /90 deg	− 23.84 D	10.88 R	17.05 Y	20.16 B	1.60 R	3.458	388.75
12	90 deg 4% Na ₂ CO ₃ /90 deg	− 24.83 D	12.20 R	18.24 Y	21.86 B	− 1.88 R	3.642	409.37

Cold room temperature

Table 5 Color fastness properties of silk fabric dyed with *B. vahlii* bark extract

Sample number	Color fastness to washing		Color fastness to water		Color fastness to crocking		Color fastness to light
	Color change	Staining on wool	Color change	Staining on wool	Dry	Wet	
1	4–5	5	4–5	5	5	5	5
5	4	4–5	4–5	4–5	5	4	6
11	3–4	4	4	4	4–5	4	6

Table 6 Color fastness properties of silk fabric dyed with *B. vahlii* bark extract and treated with BPE

Sample number	Color fastness to washing		Color fastness to water		Color fastness to crocking		Color fastness to light
	Color change	Staining on wool	Color change	Staining on wool	Dry	Wet	
11	4	4	4–5	4–5	5	4–5	7

there is hardly any significant difference in terms of their flammability properties. Average burning times were 5.1 and 5.3 s in case of control silk fabric and dyed silk fabric respectively. Whereas the standard deviations were 0.36 and 0.52 s in case of control silk fabric and dyed silk fabric respectively. However, char of dyed fabric indicates that the resistance against burning compared to control fabric as evidence in Figs. 6 and 7.

Dyed silk fabric treated with BPE and undyed fabric treated with BPE extract exhibit excellent performance against flame. Samples did not ignite and left an average char length of 34 and 42 mm respectively as mentioned earlier with standard deviation 2.79 and 3.01 mm. Dyed and BPE treated silk fabric samples showed great result in all the cases and Fig. 8 proclaim the same. It is also clear

from the image that char looks uneven and wavy, which is clearly indicating the burning resistance. This may be due to mineral present in the BPE extract. Though all the flammability test results come under Class I category of ASTM D1230 and considered by trade to be acceptable, dyed and undyed silk fabric treated with BPE evidenced excellent result.

4.5 Mechanical properties

Compression test results in Table 8 indicate that there is an increase in fabric thickness due to dyeing with *B. vahlii* bark extract and after treatment with BPE. Bending length and Flexural rigidity test results indicate that the silk fabric dyed with *B. vahlii* bark extract have better

Table 7 Flammability of silk fabric after dyeing and finishing

Samples	Weight gain (%)	Flame spread time (s)	Char length (mm)	Burn-ing rate (mm/s)
Control silk fabric	–	5.1	BEL	27.45
Dyed silk fabric	2.63	5.3	BEL	26.41
Undyed silk fabric treated with banana peduncle extract	5.09	DNI	42	–
Dyed silk fabric treated with banana peduncle extract	7.89	DNI	34	–

DNI do not ignite, BEL burn entire length

handle properties than the control silk fabric in both warp and weft wise direction. This may be due mechanical agitation during dyeing process and due to the presence of

different phyto chemicals in the *B. vahlii* bark extract. Dyed silk fabric treated with BPE shows better handle properties in terms of their bending length and flexural rigidity in warp wise compared to control and dyed silk fabric sample. However, in weft wise direction, it shows higher bending length and flexural rigidity. Crease recovery result shows that there is no significant difference between the samples.

5 Conclusion

B. vahlii bark extract is a very good source of natural dye. Color yield percentage from the bark will increase in higher temperature and higher alkaline pH due to presence of lingo-cellulose in the bark. Silk fabric dyed with *B. vahlii* bark extract gives very bright color and wide range of shades. Silk fabric dyed with *B. vahlii* bark extract shows very good results against color fastness to washing, water, crocking and light test. Fastness properties were further when dyed silk fabric treated with BPE extract.

Fig. 6 Flammability images of control silk fabric **Fig. 7** Flammability images of silk fabric dyed with *B. vahlii* bark extract

Fig. 8 Flammability images of silk fabric dyed with *B. vahlii* bark extract and treated with BPE

Table 8 Compression and bending properties of silk fabric after dyeing and finishing

	Surface thickness (mm)	Fabric weight (g/cm ²)	Bending length (cm)		Flexural rigidity (mg cm)		Crease recovery angle (degree)	
			Warp	Weft	Warp	Weft	Warp	Weft
Control silk fabric	0.09	0.0038	1.90	2.03	26.06	31.79	97	103
Dyed silk fabric	0.11	0.0039	1.78	1.79	22.00	22.36	94	102
Undyed silk fabric treated with BPE	0.11	0.0040	1.88	2.08	26.57	35.99	96	105
Dyed fabric treated with BPE	0.12	0.0041	1.65	2.15	18.41	40.74	92	105

BPE treated silk fabric exhibit excellent performance in 45° flammability test. Sample did not ignite during the test and left average char length of 34 mm. After dyeing with *B. vahlii* bark extracts fabric shows better handle properties. Whereas after BPE treatment fabric shows warp wise lower bending length and flexural rigidity and

weft wise more. Crease Recovery result is found satisfactory and within the acceptable limit.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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