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Dying of silk by natural dye

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ABSTRACT: In this report we describe how the dried leaves of Thespesia populanea have been estimated for their potential as a source for natural dyeing of silk fabric. These dried leaves are abundant, cheap and readily available as an agricultural byproduct. Extracting procedures from the leaves were undertaken under different operating conditions such as extraction pH (10), time (90 min) and temperature (95°C). The dyeing was carried out using 10% o.w.f. dye concentration with and without the use of alum, tannic and tartaric acid mordants. It was found that mordants have a significant effect on the color of dyed silk fabrics. The color strength (K/S), coordinate values and color fastness properties of the dyed samples were assessed. The K/S values were enhanced with the increase in dye absorbance and the mordant used. The dyed silk samples which were tested for antimicrobial activity against gram-positive and gram-negative bacteria showed acceptable fastness and antibacterial properties.

INTRODUCTION

With public awareness of the health hazards resulting from potent chemical usage, environmentally friendly and biodegradable products are regaining popularity in the textile industry. Natural dyes extracted from plants and insects are renewable and sustainable bioresource products with minimum environmental impact. These natural products have been known since antiquity for their use, not only as food ingredients [1-4] but also as raw material in textile dyeing.[5-6] There are numerous varieties of plant species available in the forests which have the potential to be used as raw material for different forest-based industries.[7-8] Among different plant species, some trees shed their leaves at one time in a year and create vibrant hues associated with autumn. During this season huge amounts of fallen leaves are available which would essentially be waste material.

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Thespesia populanea is a shrub or medium -sized evergreen tree, belonging to the family Malvaceae, commonly known as the Portia tree, found in tropical regions and in coastal forests of India. The leaves are edible and used for livestock. The leaves also have medicinal properties[9] and the theflavinoids present are acacetin (Fig. 1) and quercetin[10-11] (Fig. 2). Decoction extracted from these leaves is used to treat high blood pressure, and tea brewed from these leaves is used to treat rheumatism and urinary retention, and have antidiabetic activity. The main objective of this study however, is the extraction of natural dye from T. populanea waste leaves and its application on silk fabric.

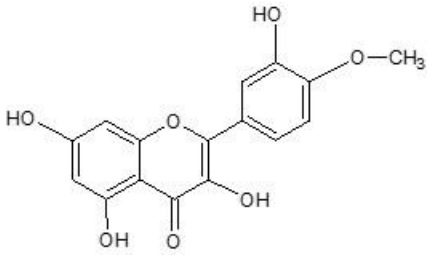

Figure 1. Acacetin

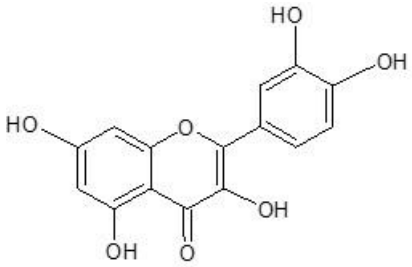

Figure 2. Quercetin

METHODS/EXERIMENTAL

Raw material

The plant material, in this case, the fallen leaves of the plant species Thespesia populanea were collected from the Jnanabharathi campus, Bangalore University, Bangalore. The collected dry leaves (2000 g) were washed under water to remove dust particles and shade -dried at room temperature (25±2°C). This processed plant material was ground to powder form in an electrically operated grinder.

Textile material and chemicals

Plain woven, degummed mulberry silk fabric weighing 52 g/m2 with a yarn density of 122 ends/inch and 106 picks/inch was used for dyeing. The material was supplied by Central Silk Board, Bangalore, India for this study. Potassium aluminum sulphate [$KAl(SO_4)_2.12H_2O$], tannic acid [$C_{76}H_{52}O_{46}$], tartaric acid [$C_4H_6O_6$] were used as mordants, all of which were of analytical grade and purchased from Merck.

Extraction of colorant

The aqueous extraction of dye was carried out under distilled water with varying conditions, such as pH, duration and temperature. In each case, the absorbance at maximum wave-length(λmax) was measured using Hitachi-U-2000 UV-Vis absorbance spectrophotometer.

Mordanting process

The dyeing experiments were carried out using silk fabric by adopting pre and post-mordanting techniques. . The samples were treated with different mordant solutions before and after dyeing. The silk fabric was dipped in 30 ml of prepared 5% mordant solution at 1:30 MLR at 50-60°C for 20 min. Then the mordanted silk fabric sample was air dried for 10 min. The dye solution was prepared at 1:30 MLR with 10 % dye and the mordanted sample was dipped in dye solution for 60 min at 90°C. The dyed materials were then washed first with cold water followed by soap solution and then again washed thoroughly with running water. The wet samples were dried at room temperature. Mordanting was carried out after dyeing for post -mordanted samples.

Dyeing

Dyeing of silk fabric samples were carried out at 95°C in a dye bath maintained at pH 5 containing 10% shade (owf) at MLR 1:50 in a HTHP dyeing machine for 45 min. After dyeing, samples were rinsed and soaped at 40°C for 10 min using non-ionic soap and then rinsed.

Determination of K/S value

Color strength value was measured using reflectance measurement. Color values were evaluated by means of K/S and CIELAB color difference values with illuminant D65/100 observer on Greatag Macbeth Color Eye 7000 A Reference Spectrophotometer. The K/S values were assessed using the Kubelka-Munk equation (1)

$K/S = (1-R)^2/2R$ …… equation (1)

Where, K is the coefficient of absorption; S, the coefficient of scattering and R, the surface reflectance value of the sample at a particular wavelength.

Measurement of fastness properties

The test for rubbing fastness was performed according to ISO-X12 using crockmeter. Light fastness test was performed according to ISO 105 B02 using Xenotest light fastness apparatus and the color fastness to perspiration test was according to ISO 105 E04. Washing fastness test was according to ISO 105 C02 using Launder O meter.

Antimicrobialproperties

Escherichia coli (E. coli) a gram-negative bacterium, was used as it is a popular test organism known for its confrontational action against common antimicrobial agents. Staphylococcus aureus (S.aureus), a pathogenic gram-positive bacterium was used as it is known to cause major cross infections in hospitals and is the most frequently evaluated species[14-17]

Quantitative assessment by percentage reduction test (AATCC 100-2004)

Specimens of the test material were shaken in a known concentration of bacterial suspension and the reduction in bacterial activity in standard time was measured. The efficacy of the antimicrobial treatment was determined by comparing the reduction in bacterial concentration of the treated sample with that of the control sample expressed as a percentage reduction in standard time.[18-19] The percentage of reduction of the microorganisms by using dye was expressed as follows:

% Reduction = [(A-B)/B] ×100

Where A and B are the surviving cells (CFU/ml) for the flasks containing the control (blank silk fabric) and test samples (natural dye treated silk fabric), respectively, after 18 h of contact time.

Wash durability test

The wash durability of the antimicrobial properties of the dyed sample was evaluated after different wash cycles. The samples were washed with 5% neutral soap solution for 20 min. The washed samples were tested for its retention of antimicrobial activity after 0, 1, 5, 10 and 15 launderings by AATCC test method.

RESULTS AND DISCUSSION

Figure 3 shows the effect of extraction time on dye yield at different pH while maintaining the temperature at 95°C. It is clear that at every pH the maximum yield was obtained at 90 min extraction time. Thereafter, notable increase on yield was not observed, and hence pH 9 and 90 min time are found to be optimum for dye extraction. The dye yield was 22%.

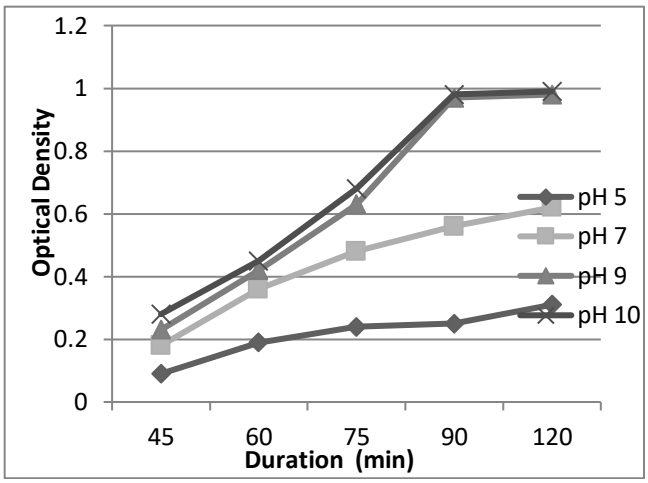

Figure 3. Effect of time on extraction at different pH (temperature 95°C)

Color strength and color co-ordinates of dyed samples

CIE Lab system was used to evaluate the color parameters, where L* refers to lightness-darkness values, a* values run from negative (green) to positive (red), b* values run from negative (blue) to positive (yellow), c values represent the

chroma and h corresponds to hue angle. L*, a*, b*, c and h values of dyed silk samples are given in Table 1. Results obtained indicate that lightness (L*) values are found to be less significant in the case of all the samples. Control and tannic acid mordant has a comparatively higher darkening effect than others which is evident from L* value data. c values show alum and tannic mordanting results in brighter shades whereas tartaric acid increases dullness of shades. K/S value of the silk samples shows that use of mordants considerably amplified dye absorption leading to higher K/S values in case of mordanted samples than un-mordanted samples. Tartaric acid mordant was found to have a more prominent effect on color strength than others.

Mordant	Method	K/S	L*	a*	b*	C	h
Undyed	-	-	90.8	2.3	0.25	2.2	365.15
Control	-	2.6	72.8	5.2	19.63	20.1	74.74
Alum	Pre	4.6	80.8	1.8	18.6	20.3	86.65
	Post	4.9	82.8	0.8	15.2	18.3	86.85
Tannic	Pre	2.2	72.7	5.6	19.3	20.2	74.54
	Post	3.4	73.7	4.3	16.6	17.2	73.68
Tartaric	Pre	4.8	81.4	0.8	15.1	15.4	87.35
	Post	5.4	82.1	1.6	12.8	12.2	83.68

Table 1. Colorimetric data of silk samples dyed with Thespesia populanea leaves extract

Color fastness properties

Color fastness properties (washing and rubbing and light fastness) of the Thespesia populnea dyed silk samples are given in Table 2. The washing fastness properties of mordanted samples were found to have been enhanced and have well to very good ratings of 4-5, whereas unmordanted samples have been found to have reasonably good washing fastness ratings of 3-4. No staining on adjacent fabrics was observed. Light fastness of dyed silk samples were found to be better after mordanting compared to unmordanted silk samples. Dry and wet rubbing fastness of mordanted samples were found to have very good ratings of 5, and quite good ratings of 4 on the gray scale respectively, whereas dry and wet rubbingfastness of un-mordanted samples were found to have fairly good to good ratings of 3-4.

Mordant	Method	Light	Washfastness		Rubbing fastness	
			CC	CS	Dry	Wet
Control	-	2	3	4	4	3
Alum	Pre	4	4	5	5	4
	Post	4	4-5	5	5	4
Tannic	Pre	3-4	4	5	5	4
	Post	4	4	5	5	4
Tartaric	Pre	3	4	5	5	4
	Post	4	4-5	5	5	4

Table 2**.** Fastness properties for silk fabric dyed with Thespesia populanea leaves extract

Note: cc color change, cs color staining, control: silk dyed without mordants

Antimicrobial properties of dyed silk samples

To find the efficacy of the antimicrobial properties of the dye the mordanted and dyed samples were evaluated quantitatively using AATCC Test 100-2004 in order to obtain results for percent reduction of bacteria for silk samples dyed with and without being pre-mordanted with alum, tannic and tartaric acid. Tannic acid mordanted samples showed higher antibacterial activity against both E. coli and S. aureus. The activity increased 21.5 % with E. coli and 23.2 % with S. aureus compared to the control sample. The bacterial inhibition may be attributed to the active substances present within the coloring matter, as also in the mordant.

Table 3. Antibacterial activity of Thespesia populanea leaves dyed silk fabrics with and without mordant.

Shade 20%	Reduction (%)	
	E. coli	*S. aureus*
Control	75.5	74.8
Alum	78.4	76.3
Tannic acid	97	98
Tartaric acid	77.5	75.9

Wash durability test to confirm antimicrobial properties

The antibacterial activity of the dye in solution when tested against selected microbes assesses its efficacy on the dyed silk fabrics. Investigations were carried out to study the influence of the unmordanted dyed sample (after washing with soap solution followed by water) on the growth. Effectiveness of the unmordanted dyed sample against the microorganisms is shown in Fig. 4. Microbial % reductions in the dyed silk samples were observed over four different laundering cycles of 1, 5, 10 and 15. Fabrics showed that significant antimicrobial activity was retained in the fabrics dyed with the extract for up to 5 washes. After 5 wash cycles it was reduced to less than 60 % of the initial activity against both kinds of bacteria.

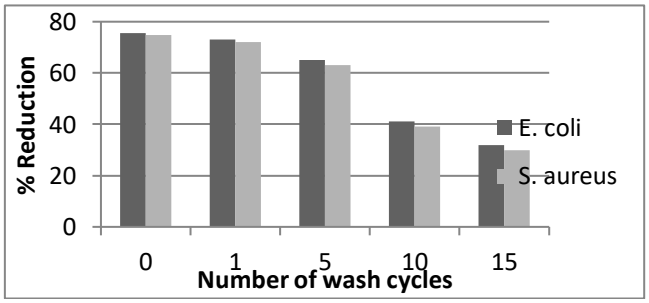

Figure 4. Number of wash cycles

CONCLUSION

In the present study the major emphasis has been on extraction of dye, colorimetric assessment, and evaluation of fastness and antimicrobial properties of silk fabrics dyed with extracts from fallen leaves of Thespesia populanea. Maximum dye extraction was observed at 95°C and pH 10. It was found that with increasing the extraction time, the dye extraction efficiency increased, but an optimum time of 90 min was considered for extraction of the dye in order to make the technology more cost effective. Mordants have a significant effect on the dyeing performance. All the dyed fabrics had good color fastness to washing, rubbing and acceptable color fastness to light. The antimicrobial tests demonstrate an exciting opportunity for the dyed textile to be used as potential prospective material in the manufacture of protective clothing against common infections when used in hospitals, hotels, etc. The current findings clearly demonstrate that extraction of natural colorants from abundantly available waste leaves of the plant Thespesia populanea can be a sustainable technique towards using waste as raw material for forest-based industry.

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