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Efficacy of natural dye from *Gerardiana diversifolia* on pashmina (Cashmere) shawls

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The present investigation deals with studies on potentiality of a colour extract from *Gerardiana diversifolia* as a source of natural dye, and on identification of chemical constituents responsible for dyeing ability. The efficacy of the extracted dye(s) on pashmina (Cashmere) fabrics has been evaluated for colour strength and fastness properties. Phytochemical studies reveal that the quinone and tannin are major components contributing to dyeing properties. The extracted colorant from *Gerardiana diversifolia* has potential to dye pashmina fabric with brown and grey shades having excellent wash and good to very good light fastness properties.

Keywords: Cashmere, *Gerardiana diversifolia,* Natural dye, Pashmina, Wool

1 Introduction

India is one of the mega diversity countries having about 0.5 million plants, 0.17 millon angiosperms, more than 400 crop species and equal number of wild relatives[1]. The plant kingdom of India is a treasure house of diversified natural products. Lots of plants possess therapeutic properties and main sources of drugs as Ayurvadas. Another important attribute of such products of plant origin is their contribution as source of natural dye. The natural colourants derived from these sources are preferred due to their health effectiveness and good therapeutic properties[2].

The plants or trees do not contain dyestuff as such but the colouring compounds occur as complex organic substances such as glucosides, flavonoids, anthraquinones, etc.[3] Only a few of them are freely soluble in water but others are either insoluble (e.g. indigoid) or sparingly soluble (e.g. flavonoids). The amounts of dyestuffs vary in plants of the same spices depending upon the variety, soil and climate in which they have been grown. This drawback leads to variance in resultant shades and does not allow reproducibility of a shade.

Gerardiana diversifolia (Urticaceae family and also known by its vernacular name Dhol kanali) is a weed plant found in the entire Himalayan belt and can be located from very low to high altitudes, i.e. 1800-3600 m above the sea level. This region belongs to part of Garwal inhabitants of higher Himalayan region, where the plant grows extensively as a weed. It is a robust, perennial herb with stinging bristles. The plant comes under category of wild edibles, the leaves and flowers are mainly preferred for eating due to good source of proteins, multivitamins and mineral contents[4]. The herb also has medicinal values, for example oral intake of the extract obtained by boiling fresh leaves of the plant can cure fever due to cold. This traditional herbal medicine is used by Tibatian, Tamanga and Bhotiya communities of the Himalayan region[5]. The root part of the herb has a good source of natural colourant and local artisans used them to colour handicrafts and apparels having low colour fastness properties.

In the present study, the chemical constituents of root portion of Dhol kanali possessing dyeing capability have been identified and their efficacy as natural colorant evaluated by using different non-toxic metal mordents for dyeing pashmina shawls. Attempt have also been made to improve the fastness properties of the colouring substances on the substrates.

2 Materials and Methods

2.1 Materials

Pashmina shawl fabric having an areal density of 95 g/ m^2 was used as substrate for this study.

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The fabric was mild scoured with a 0.5 gpL nonionic detergent (Wellchem speciality JU) at 50^o C for 30 min, washed with warm water, rinsed with cold water and dried at ambient condition. $Al_2(SO_4)_3$, $SnCl_2$, and $FeSO_4$ were used as mordants. All other chemicals were of LR grade.

2.2 Phytochemical Analysis

The root of the Dhol kanali was procured from local market in Kullu District of Himachal Pradesh. The roots were dried in shadow and grinded into powder using lab model grinding machine. The powered root (600 g) was transferred into a conical flask and the material was refluxed with methanol (1.8L) for 24 h at 64 ^{o}C on Soxhlet apparatus. The extract thus obtained was dried in oven. The crude extract (90g) was dissolved in 200mL methanol for fractionation of compounds. The solution was then shacked with 200mL of petroleum ether and fraction separated. The same process is repeated by using chloroform, ethyl acetate, and distilled water in tandem. The separated fraction is dried in oven and quantified as % weight of methanol extract. The different compounds in the solvent extract were identified by standard test methods[6-9] as summarized in Table 1.

2.3 Dyeing and Mordanting

The ground roots were soaked in water overnight at 1:30 M : L ratio, boiled for 1 h and extracted colorant was filtered through Whatmen No.1 filter paper. Dyeing was carried out in bath comprising 5% colorant (owm) and acidified with 0.5 gpL acetic acid (*p*H 5-6) at material to liquor ratio of 1:40. The fabric was dyed at 100 ºC for 1 h on a boiling bath . On completion of dyeing, the sample was removed, rinsed with water and finally dried at ambient condition. The dyed pashmina fabric material was mordanted with 3% mordant (owm) in simultaneous mordanting techniques. The mordant was added in the same dye bath after 1 h of dyeing as per standard procedure[10-12]. Pashmina fabrics, dyed and differently mordanted, are given in Table 2. A portion of the dyed fabric was subjected to an after wash treatment with non-ionic detergent (1.0%) at 60 ºC for 30 min and the resultant fabric was designated to ADW.

2.4 Colorimetric Estimation of *K/S* Value and Fastness Properties

The colorimetric values L, a, b and *K/S* of dyed or detergent washed samples were recorded at 10^o observer angle in JAYPAK 4802 Colour matching system (Jay Instruments Ltd, Mumbai, India), using D65 lamp as illuminant.

The wash fastness of the dyed samples was evaluated as per standard procedure ISO 105-C10:2006[10]. The light fastness of the dyed material was determined using Q-Lab Xe-1-S light fastness tester using standard procedure described elsewhere[12]. For assigning grades to evaluate both wash fastness and light fastness the ratings were given from 1 to 5. The fastness rating of 5 indicates excellent fastness and the rating of 1 indicates very poor fastness.

3 Results and Discussion

3.1 Phytochemical Evaluation

The yield of methonolic extract of Dhol kanali root powder is found to be 15%. Further fractionation of the methanol soluble material by successive separation with petroleum ether, chloroform, ethyl acetate and finally distilled water in a separating funnel yield 3.5, 4.5, 2.5 and 66 % of sub-fractions respectively. Column chromatography of the petroleum ether soluble fraction on a silica gel column as stationary phase, using dichloro methyl:

Table 1—Experiment details of phytochemical analysis of various sub-fractions

Target compound	Reagent applied/ Experimental procedure	Colour	Ref. No.
Glycosides	Anthrone plus one drop of concentrated sulphuric acid, followed by warming on hot water	Dark green	6
Coumarins	Sodium hydroxide (10%)	Yellow	7-9
Flavanones	Sodium hydroxide (10%)	Orange	7
Tannins	Ferric chloride solution (10%)	Dark blue or greenish black	7-9
Phenols	Few drops of aqueous ferric chloride (10%)	Blue or green	6
Quinones	(i) Conc. Sulphuric acid 1mL/mL of extract	Red	7-9
	(ii) Sodium hydroxide (1 mL, 10%)	Bluish green or red	
Carbohydrates	Benedict's reagent(5mL) followed by boiling for 2 min.	Red precipitate	6
Steroids	Glacial acetic acid (1mL) plus acetic anhydride(1mL) per mL of extract, followed by two drops of concentrated sulphuric acid	Red to blue and finally to bluish to green	6

petroleum ether (50:50) as eluent furnish a light green compound (0.01g). Similarly, chloroform fraction was fractionated into green (0.04g) and brown (0.10g) compounds. The ethyl acetate fraction was washed with petroleum ether and chloroform. A brown red (5.0g) compound is obtained which responds positively to tannin test. The distilled water fraction upon washing with petroleum ether and diethyl ether affords a white compound (0.1g) which responds positively to test glycosides. An oily white compound is separated out upon extraction of the distilled water soluble fraction with chloroform which responds positively to test steroid. The residual red brown precipitate gives positive response to test for quinone, carbohydrate and tannin(8-9%).

3.2 Colorimetric Value

The yield of dye crude extract is found to be 15% (over the weight of dried powder). The dye take up of crude dye extract on pashmina fabric is found satisfactory. The L*, a* and b* values of dyed pashmina fabrics is shown in Fig. 1(a), (b) and (c). The lightness L* value of the dyed samples declines upon mordanting, as dye - metal coordinate complex generally reduces the lightness of dyed textiles with modification in the chrome of the shade. The decreasing trend is observed with a* value in the mordanted samples on green-red axis of CIE Lab scale, indicating that the dyed samples tend to be greener than control dyed samples. However , the decline in a* is found marginal when alum or stannous chloride is used as mordant but drop is remartkably high in case of ferrous sulphate mordanted fabric. An opposite trend is observed in case of b* value on blue-yellow axis. The observed b* value for mordanted samples shows an increasing trend, though marginal once again, indicating that alum and stannous chloride mordants produce enhanced yellowness as compared to control dyed sample. The ferrous sulphate mordanted fabric exhibits a sharp fall in b* value, indicating a shift of shade towards blue region (hypsochromic shift). The afterwashed fabric follows the same trend in all cases and the tonal differences between detergent wash and unwashed fabrics are found marginal.

Table 2—Sample code of pashmina fabrics

Sample details	Sample code
Scoured	Control
Dyed with colour extract alone	Dye
Dyed with colour extract + $Al_2(SO_4)_3$ mordant	Dye + alum
Dyed with colour extract + $SnCl_2$ mordant	Dye + stannous
Dyed with colour extract + $FeSO_4$ mordant	Dye + ferrous
Dyed and ringed only	R
After detergent wash	ADW

3.3 *K/S* Value

The dye strengths of dyed fabrics were determined by comparing the *K/S* values of dyed pashmina fabrics [Fig.1(d)]. The *K/S* value of dyed sample without mordanting is found to be 0.558, which increases within the range of 0.621 - 0.681 for different mordants. The highest value of 0.681 is achieved with stannous chloride mordanted fabric. This may be attributed to higher aggregation of dye-metal complex during mordanting with stannous chloride as compared to other mordants. A significant change in shade is observed in detergent-washed fabrics which remains steady for all, but for ferrous sulphate mordanted fabric. The abrupt change in shades explains the reason for obtaining high *K/S* and

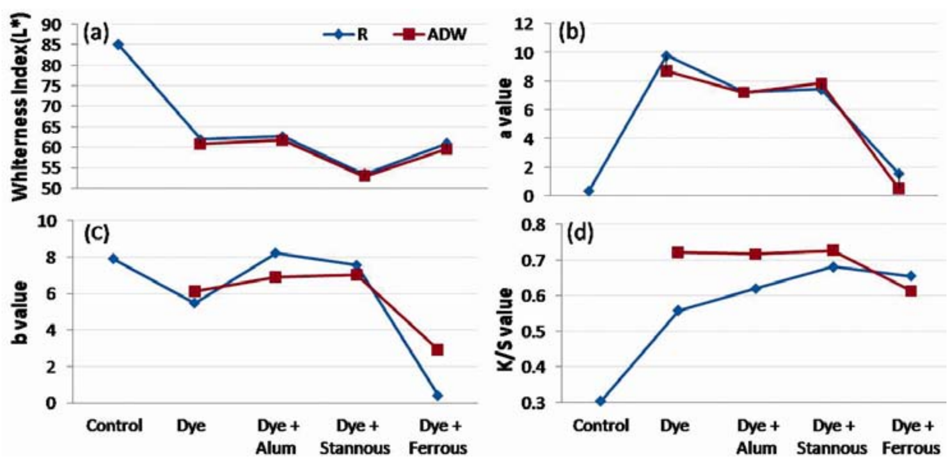

Fig.1—L, a, b and *K/S* value of pashmina fabrics

Table 3—Colours of cashmere (pashmina) shawl dyed with Dhol kanali root

Sample	Colour obtained	Washed sample	Wash fastness	Light fastness
Dye			5	3
Dye + Alum			5	3-4
Dye + Stannous			5	3-4
Dye + Ferrous			4-5	3-4

low ‘L’ value. On the contrary, the increase in depth of shades in unwashed fabrics is gradual (in parity with the observed *K*/*S* values) which attains a saturation level when stannous is used as mordant.

3.4 Shades of Colour and their Fastness Properties

The shade produced on pashmina fabrics and its fastness properties to light and washing are given in Table 3. The fabric dyed with extracted colorant from *Gerardiana diversifolia* is found to produce brown shades with or without using alum or stannous chloride as mordants. Such a high depth of shade may be attributed to high tannin content (8-9%) in the root. However, a grey shade is produced when ferrous sulphate is used as mordant. The fastness to washing is found to be excellent when dyed with colourant alone. The colour estimates obtained for mordanted fabrics after detergent wash are also rated excellent. The shades obtained for control, stannous chloride and alum mordanted fabrics are darker as also manifested by their higher *K*/*S* values after washing. The colour change due to *p*H is attributed to the presence of coumarin in the chemical constituent of Dhol kanali. Ferrous mordanted fabric shows a slight reduction in *K/S* value after washing, having fastness property ranging from excellent to very good. The light fastness property for the mordanted fabric is observed in the range of 3 – 4 against 3 for control fabric which is satisfactory.

4 Conclusion

4.1 Quinone and tannin are major components in root part of Dhol Kanali (Gerardiana diversifolia) responsible for its dye ability.

4.2 The extracted colorant has potential to dye pashmina fabric, producing brown and grey shades with excellant wash and good to very good light fastness properties with and without mordanting.

4.3 The yield of crude extract of colouring component is more than 10 %. Thus, it could be utilized for commercial application on pashmina and wool materials.

References

1 *Manual on Exploration and Collection of Plant Genetics Resources and Related Indigenous Knowledge* (National Bureau of Plant Genetic Resources, New Delhi), 2000.
2 Chengaiah B, Rao M K, Alagusundaram M & Chetty C M, *Int J Pharm Tech Res,* 2(1) (2010) 144.
3 Marhareta Sequin-Frey, *J Chem Education*, 58(4) (1981) 301.
4 Sundriyal M & Sundriyal R C, *Current Sci*, 85(6) (2003) 731.
5 Tamang G, *Our Nature*, 1(2003) 37.
6 Misra C S, Kumar P, Lipin D M S, Joel J, Kumar A, Veettil T & Thankamani V, *Int J Res Phytochem Pharmacol*, 1(2) (2011) 77.
7 Salna K P, Sreejith K, Uthiralingam M, Mithu A P, Milton M C J & Fleming A T, *Int J Pharm Pharmaceut Sci*, 3(3) (2011) 291.
8 Khyade M S & Vaikos N P, *Afri J Biotechnol,* 8 (2009) 6434.
9 Gothandam K M, Aishwarya R & Karthikeyan S, *J Phytochem,* 2(4) (2010) 01.
10 *ISO 105-C10: 2006 Textiles-Test for Colour Fastness – Partc10: Colour Fastness to Washing With Soap or Soap and Soda* (Bureau of Indian Standards), 2006.
11 Mathur J P & Bhandari C S, *Indian J Fibre Text Res*, 26 (2001) 432.
12 Raja A S M, Pareek P, Shakyawar D B & Sarfaraz W A, *Adv Appl Sci Res,* 3(2012) 156.