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Extraction of eco-friendly natural dyes from *Justicia adhatoda* leaves and their application on wool and silk fabrics

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Abstract

Natural dyes are widely used for textile industries, due to their harmless effects and harmful consequences of synthetic dyes. Natural dyes work on cotton, silk and wool etc. In this article, the leaves of *Justicia adhatoda* are used as a colorant for dyeing of wool and silk fabrics. Various extracts (aqueous, butanol, chloroform and hexane) of *Justicia adhatoda* leaves have been prepared and investigated. The obtained dyes yield a variety of shades and are processed with mordants such as alum and copper sulphate. From the K/S values, it is found that hexane extracted dye generated maximum color depth among the four solvent systems. Also, the dyed wool fabrics show higher K/S values than the silk fabrics. The use of mordants gives rise to better dyeing, exhibiting darker shade and improved color strength than without mordanting conditions. The fastness properties of samples are good to very good (4 to 4-5), suggesting the formation of dye-mordant complex.

Keywords: Natural dyes; *Justicia adhatoda*; Hexane extract; Eco-friendly dyeing; Solvent extraction

1. Introduction

Natural dyes are those colorants obtained from plants, vegetables, animals and minerals which have their own merits having no match. An ample range of shades can be attained from various parts of plants including root, barks, leaves, flowers and fruits. These dyes find use in coloration of textiles, food, drugs and cosmetics [1]. They were applied to the fiber without any pretreatment of the dye-material or the textile. Small quantity of dyes is also used in coloration of paper, leather, shoe polish, wood [2].

Natural dyes are mostly known substantive and must be applied on textile with the help of mordant, usually a metallic salt, having an affinity for both the coloring matter and fiber [2, 3]. The metal ions of these mordants act as electron acceptor for electron donor to form coordination bond with the dye molecule, and are capable of forming weak to medium interaction forces and thus can act as bridging material to create substantivity of natural dyes/colorants [4, 5].

There has been increasing interest in natural dyes, as the public is becoming more aware of the ecological and environmental problems related to the use of synthetic dyes. The use of natural dyes cuts down significantly on the amount of toxic effluent resulting from the synthetic dye processes. Natural dyes have also been used for dye sensitized solar cells and for printing. It is reported that, some natural dyes not only dye with unique and elegant color but they

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also provide antibacterial and UV protected functions to fabrics. Thus these natural dyes are applied on fabric of cotton, wool and silk [11].

In the present work natural leaves extract of *Justicia adhatoda* is used to dye wool and silk fabrics [8,9]. The color of *Justicia adhatoda* leaves is extracted using four different extraction medium viz. 100% water, Butanol, chloroform and hexane [7]. The optical densities of extracted crudes, color depth in terms of K/S and washing fastness are compared in different extraction mediums. The effect of mordanting in such dye is also investigated and compared.

Justicia adhatoda is an evergreen shrub belonging to *Acanthaceae* family which grows to a height of 4-8 feet and is characterized by broad, lanceolate (sharp and pointed like a lance) green leaves measuring 10 to 16 centimeters in length and 5 centimeters wide. The wood of the stem is soft and makes a great charcoal for gunpowder. The flower has large, attractive, white petals, streaked with purple on the lower lip. The leaves, roots and flowers of *Justicia adhatoda* are used extensively in traditional Indian medicine for treating bronchitis, tuberculosis and other lung and bronchiole disorders [6]. The various extracts of the plant have been reported for their antibacterial and anti-inflammatory properties.

2. Material and methods

2.1. Plant and Fabric Material

Leaves of *Justicia adhatoda* are collected from Bibi Kaulan Botanical garden, Guru Nanak Dev University, Amritsar.

2.2. Preparation of Extracts

Extraction of dyes is carried out for characterization of the colorant as well as analyzing the effect of newly found dye resources on fabric. The fresh leaves of *Justicia adhatoda* (3 kg) are washed with tap water and then dried at room temperature. The Ethanol extract is collected and distilled under reduced pressure in a pre-weighed round bottom flask. The crushed dried leaves are packed in percolator and dipped in ethanol. After 24 hours, ethanol extract is collected and distilled under reduced pressure over rotatory evaporator. Fresh ethanol solvent is added and again the same procedure is repeated 2-3 times. The combined extract is distilled and weighted which is termed as crude extract. The crude extract is suspended in water and fractionated with various solvents with increase in polarity n-hexane < chloroform < n-butanol < water resulting in hexane, chloroform, n-butanol and aqueous fraction respectively [10]. Then the cooled extract is filtered through a filter paper and solvent is removed through a rotary evaporator.

2.3. Dyeing procedure

The fabric used is firstly immersed in non-ionic detergent solution prepared by dissolving 2g in 1000 ml of water. For the dyeing purpose 2% stock solution of each extract (aqueous, butanol, chloroform and hexane), was prepared by dissolving 2 g of it in 10 ml of their respective solvents and made it 100 ml by addition of distilled water.

Pre-mordanting treatment is conducted using two mordants (a) alum (b) copper sulphate. Fabric samples are pre-mordanted by treating with 5.0% (on the weight of fabric) alum and copper sulphate at 80°C for 30 min with material to liquor ratio of 1:30. After mordanting the samples are rinsed in cold water to remove the excess of mordant and used for dyeing purpose. Afterwards, dyeing of pre-mordanted fabric is performed for 1 hour at 90°C in an open water bath beaker dyeing machine at 1:30 material to liquor ratio. The dyed samples are then rinsed with cold water and dried in open air. The fabric samples have also been dyed in in absence of mordants under same conditions.

After dyeing of mordanted and without mordanted fabric samples, soaping of these fabrics is carried out by using non-ionic detergent in order to remove excess dye. For this process detergent solution (2g/L) is prepared and the fabric sample is dipped in it for 15 min at 60 °C. After soaping sample is rinsed with cold water to remove soap from sample and dried it at room temperature.

2.4. Evaluation of samples

The analysis of shades and colors appeared on the samples are undertaken via instrumentation procedure to determine the existence of colour. Analysis using Spectrophotometer spectraflash 600 Plus CV UV is performed to assess the color of dyed samples and to check their color fastness when subjected to washing. In order to evaluate the absorption spectroscopy in UV-visible spectral region, Ultraviolet–visible spectrophotometer was employed which measures the absorption of a beam of light after it has passed through a sample or after reflection from sample surface.

3. Results and discussion

To determine the depth of the color on the dyed fabric, K/S values were determined for all the samples. The colors obtained in dyeing are recorded and shown in Table 1 and Table 2 along with their K/S values (mentioned in brackets) for unmordanted and mordanted samples respectively.

For unmordanted samples, it has been observed that wool samples are deeper in depth than silk counterparts (as given by the higher K/S values of wool dyeing). Upon mordanting, the K/S values get higher which demonstrates that the fabric has higher color strength. The enhancement in color of dyed fabric attributes to the fact that when fabric is impregnated with mordant, the dye react with mordant forming a chemical bond and attaching it firmly to the fabric. These extracted dyes contain phenolic groups which may help in covalent bond formation with fabric, thereby, resulting in good fixation of dye on the fibrous material. Moreover, these phenolic groups can form metal chelation with different mordants.

Table 1 Dyed Wool and Silk Samples (Without Mordanting)

Extract	**Wool Fabric (K/S value)**	**Silk Fabric (K/S value)**
Aqueous	 (3.46)	 (2.15)
Butanol	 (3.45)	 (1.99)
Chloroform	 (5.35)	 (5.00)
Hexane	 (7.87)	 (3.22)

Table 2 Dyed pre-mordanted wool and silk fabrics

Extract	**Mordant**	**Wool Fabric**	**Silk Fabric**
Aqueous	Alum $CuSO_4$	 (2.14)  (8.55)	 (1.65)  (5.58)
Butanol	Alum $CuSO_4$	 (5.95)  (11.20)	 (1.88)  (4.75)
Chloroform	Alum $CuSO_4$	 (4.65)  (6.30)	 (2.40)  (3.09)
Hexane	Alum $CuSO_4$	 (23.10)  (25.5)	 (9.80)  (10.40)

The samples have also been checked for their optical density measurements which provide information of absorption of incident light by the sample and give the λ_{max} values of the absorbed light by the sample. Figure 1 shows absorption spectrum of leaves extract in different solvents such as aqueous, butanol, chloroform and hexane at different pH value. From these plots it can be concluded that, λ_{max} value of each extract solution in acidic medium (pH 4) resembles with the λ_{max} values of the pure extract sample, while λ_{max} value in basic (pH 12) conditions are different. Thus acidic medium is suitable for dyeing purpose.

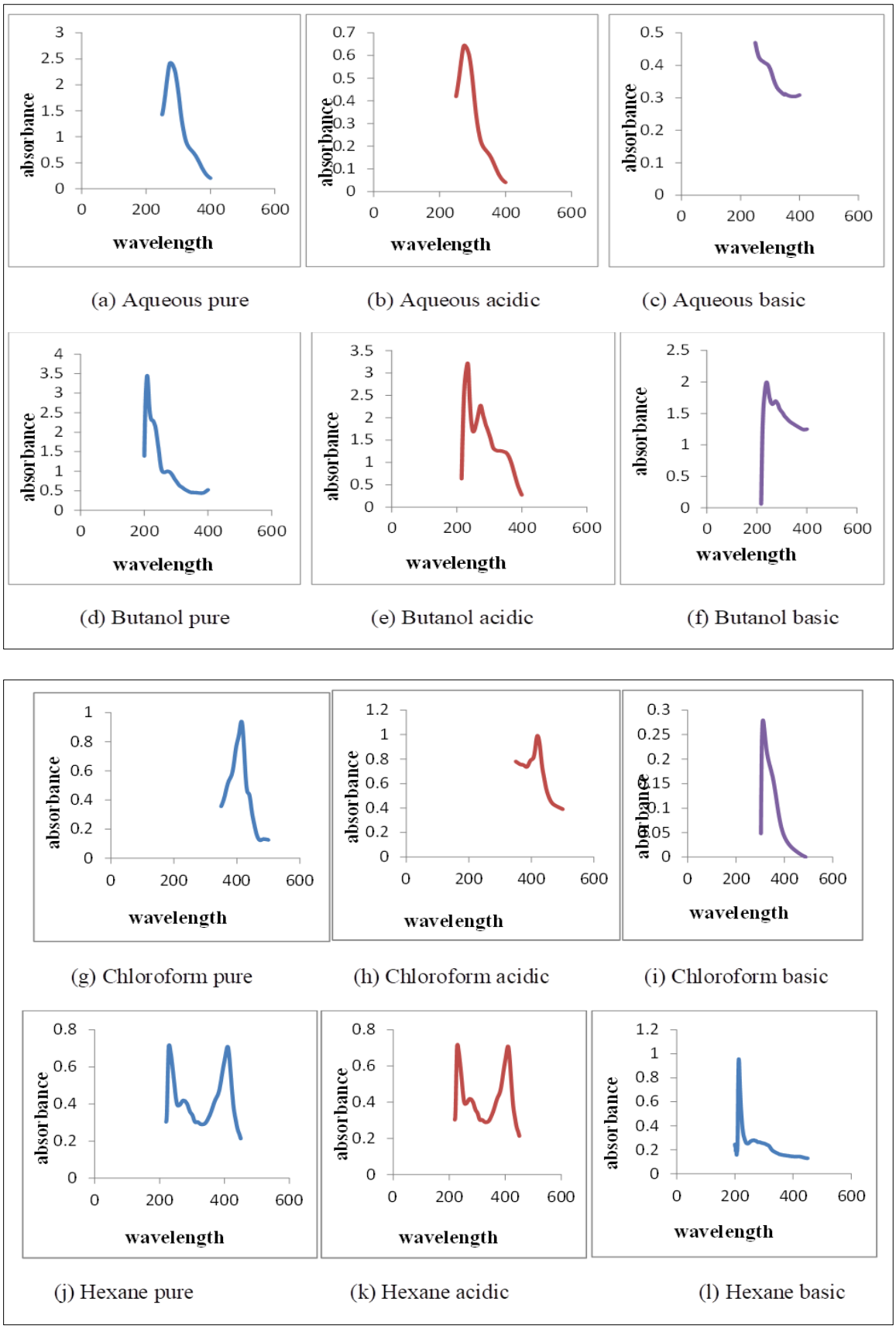

Figure 1 Absorption spectra of extracted solutions of *Justicia adhatoda* leaves

Table 3, 4, 5, 6 and 7 show washing fastness results of dyed samples and their gray scale rating. These values indicate that washing fastness ratings of the silk and wool fabric dyed with *Justicia adhatoda* leaf extracts are good to very good (4 to 4-5). The color staining rating on worsted wool component of the SDC multifiber strip is the highest on all other components, may be because of the intrinstic high substantivity and inaccessibility of the morphology of fiber at 60°C. The color staining rating of pre-mordanted fabric is much better than that of unmordanted fabric. It is because the alkaloids present in extracts forms metal chelation with mordant, hence after mordanting improve washing fastness as they are insoluble in water.

Table 3 Washing fastness results on multifiber strip (samples without mordant)

Extract	**Staining of SDC multifiber strip SCA BUC N P A W**	**(Change in Color) Wool Fabric**	**(Change in Color) Silk Fabric**
Aqueous			
Butanol			
Chloroform			
Hexane			

Table 4 Washing fastness results of staining on wool and cotton (pre-mordanted wool)

Extract	**Mordant**	**(Change in color) Wool**	**Staining on cotton**	**Staining on wool**
Aqueous	Alum			
	$CuSO_4$			
Butanol	Alum			
	$CuSO_4$			
Chloroform	Alum			
	$CuSO_4$			
Hexane	Alum			
	$CuSO_4$			

Table 5 Washing fastness results of staining on wool and cotton (pre-mordanted silk)

Extract	Mordant	(Change in color) Silk	Staining on cotton	Staining on wool
Aqueous	Alum			
	$CuSO_4$			
Butanol	Alum			
	$CuSO_4$			
Chloroform	Alum			
	$CuSO_4$			
Hexane	Alum			
	$CuSO_4$			

Table 6 Gray scale rating of color fastness data (without mordant)

Extract	Fabric	Change in color	Color staining					
			SCA	BUC	N	P	A	W
Aqueous	Wool	4-5	5	5	4-5	5	5	4-5
	Silk	3-4	4-5	4-5	4-5	4-5	5	4-5
Butanol	Wool	1	4-5	5	4-5	5	5	4-5
	Silk	1	4-5	5	4-5	5	5	5
Chloroform	Wool	1	4-5	4	4	4-5	4-5	4-5
	Silk	1-2	4-5	5	5	4-5	5	4-5
Hexane	Wool	1	4-5	4-5	5	3-4	4-5	3-4
	Silk	1	4-5	4-5	4-5	4	4	3-4

Table 7 Gray scale rating of color fastness data (pre-mordanted samples)

Extract	Fabric	Mordant	Change in color	Color staining	
				Cotton	Wool
Aqueous	Wool	Alum	4-5	4-5	4-5
		CuSO4	2-3	4-5	4-5
	Silk	Alum	4-5	4-5	5
		Cuso4	3-4	4-5	4-5
Butanol	Wool	Alum	1	4-5	4
		Cuso4	1	4-5	5
	Silk	Alum	1	5	5
		Cuso4	1-2	4	4-5
Chloroform	Wool	Alum	1	4-5	4
		Cuso4	1-2	4-5	5
	Silk	Alum	3	4-5	4-5
		Cuso4	1	4	4-5
Hexane	Wool	Alum	3	4	4-5
		Cuso4	2	4	5
	Silk	Alum	1	4-5	4-5
		Cuso4	1	4	4-5

4. Conclusion

This study demonstrated that *Justicia adhatoda* leaves can be used as the colorant for dyeing of wool and silk fabrics using different extraction of *Justicia adhatoda* leaves. Among the various organic solvent extraction methods used, aqueous extraction system is found to be the best one. But other solvent systems are also found to be equally satisfactory so far as dye exhaustion and color yields are concerned. The dyed samples were of medium brown, dark brown and greenish brown shades with acceptable fastness properties. It is found that hexane extracted dye generated maximum color depth among the four solvent systems. The wool fabric dyed with *Justicia adhatoda* leaf extract shows higher K/S values than the silk fabrics. The uses of mordants give rise to best dyeing, exhibiting darker shade than without mordanting conditions concluded from the higher K/S value. The use of mordant not only improves color strength but also provide shade difference. The fastness properties of sample are good to very good (4 to 4-5), this being attributes to the formation of dye-mordant complex.

Compliance with ethical standards

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Disclosure of conflict of interest

We also declare that this article has not been submitted elsewhere and will not be submitted elsewhere until a decision at Annals of Ayurvedic Medicine about its publication is made. We also declare that every author listed in the manuscript are fully aware of this submission and are accountable for everything mentioned in the article. Consent of submission is hereby given by all authors enlisted in the manuscript.

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