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Extraction of a Natural Dye from *Sesbania aculeata* Plant

Charu Swami[1], Sangita Saini[1] and V.B. Gupta[2]

[1]Department of Home Science, Faculty of Arts
Dayalbagh Educational Institute, (DEI), Agra, India

[2](Retired.) Department of Textile Technology
Indian Institute of Technology, Delhi, (IITD), India

ABSTRACT

*The focus on environmental concerns is increasingly causing the textile industry to explore natural sources of dyes as opposed to synthetic dyes. As a representative effort in this direction, the current paper describes the process of dyeing silk and cotton-polyester blend with the aqueous extract of Sesbania aculeata plant. This plant is a green manure plant with several traditional applications. Shade cards were prepared and evaluations were made on the basis of two fabrics dyed, five mordants and three different mordanting techniques applied. Fastness properties of the dyed fabrics were tested. For color measurements K/S values and CIE L*a*b* values were calculated.*

Keywords: Natural dyeing, Sesbania aculeata, aqueous extraction, silk, polyester-cotton

1. INTRODUCTION

Sesbania is a genus of flowering plants in the pea family, *Fabaceae*. Fabaceae or Leguminosae is an important and third largest family of flowering plants. It is commonly known as the legume family, pea family, bean family or pulse family. The main species of this family include *Sesbania aculeata /bispinosa, Sesbania sesban, Sesbania grandiflora and Sesbania speciosa* which are found throughout the world. They grow in diverse environments and climates. The plant is native to India, Pakistan, China, Sri Lanka, Africa, Southern United States and Philippines [1]. Plants of this genus are used to increase the soil's nitrogen content. Sesbania species are known to fix between 500 to 600 kg/hectare of nitrogen per year in the form of green manure, soil improver and other agro-forestry uses. Some traditional applications of Sesbania are in the medical field as astringent, antihelminthic, antibiotic and anti-tumor agents [2]. The leaves of *Sesbania aculeata* yields good concentration of (+) - pinitol which is an anti-diabetic agent [3].Sesbania is also being used as fuel wood, for fodder, in paper industry and as bio-mass for producing electricity.

From the perspective of the current research, out of the various species of Sesbania, *Sesbania aculeata* locally known as "Dhaincha" was chosen for the present study due to its low maintenance and operation cost. The initial experiments exhibited deep brown stains deposited by the plant on fabrics upon processing. A highly desirable aspect of this plant, in contrast to the other natural dyes based on vegetable and fruit sources, is that its usage in making the natural dye does not result in any wastage of an otherwise commercial product.

2. MATERIALS AND METHODS

2.1 Materials

2.1.1 Selection of Plant source for Dye Extraction

India, including several countries have rich reservoir of natural flora and fauna from which dyes can be extracted. However, a large number of these remain unexplored hitherto. In the present experiment, a plant, *Sesbania aculeata,* was selected for the above purposes. This is locally known as "Dhaincha" which was collected from the fields near Agra city[i].

Sesbania aculeata is an erect sub shrub and reaches up to a height of one to two meters. It has fibrous and pithy stems with long leaves. The growing period of the plant is from May to September. It bears purple-spotted yellow flowers from September to November in Indian climatic conditions. It produces pods which contain light brown beans. Stems are fairly thick, glabrous and branched from the base. The leaves are pinnate, 1.2–2.5 cm. long, 0.3 cm. wide and are glabrous [4] (refer Figure 1). The dye was extracted from the leaves and tender stems of the plant.

Figure 1. *Sesbania aculeata* Plant

2.1.2 Fabric used

Dyeing was carried out on
(i) Seri Silk of GSM 55 and
(ii) Polyester-Cotton blended (67-33%) fabric of GSM 95

2.1.3 Chemicals and Mordants used

The chemical mordants such as alum, copper sulfate, ferrous sulfate, potassium dichromate, and stannous chloride were used. These mordants were applied in concentrations ranging from 1-2% during pre-mordanting, simultaneous mordanting and post mordanting methods.

2.2 Methods

2.2.1 Extraction of colorant

The dye was extracted from the plant using aqueous extraction method. The plant for the dye stuff was first shredded. The shredded material consisting of leaves and tender stems of *Sesbania aculeata* plant (100 gm.) were dissolved in distilled water (1000 ml.) and allowed to boil in a beaker kept over water bath for extraction for 3 hours, at 65°C. By the end of 3 hours nearly complete extraction of the dye color from the leaves and stems of *Sesbania aculeata* could be carried out. The solution was filtered and the extract obtained was used for dyeing purpose.

2.2.2 Identification of the extracted dye

The chemical constituents of the extracted dye were identified through various techniques.

Ultra violet-visible spectroscopy: The extracted dye was dissolved using water as solvent and scanned through UV-Visible – 1601 Shimadzu UV Spectro, Spectrophotometer. Wavelength of the dye at lambda (λ) max was measured and the compounds present in the extracts were interpreted.

Fourier-transform-infra-red spectroscopy: FT-IR of extracted dye was traced on Vertex 70 model of Bruker.

2.2.4 Fastness testing of dyed samples

The dyed samples were tested for their fastness properties to various agencies.
Laundero-meter *(Paramount Model)*: For testing the Wash fastness.
Crockmeter (*Paramount Model*): For testing the Rub fastness (Wet and Dry).
Glass box case: For testing Sun-light fastness.
Color matching system: The reflectance of the dyed samples was measured on a Premier Colorscan.

2.2.4 Scouring of silk and polyester blend fabrics

For silk fabrics, scouring was carried out with solution containing 2 g/l of non-ionic detergent for 30 minutes. The scoured material was thoroughly washed with tap water and dried at room temperature. It was soaked in distilled water prior to dyeing or mordanting. In case of polyester-cotton blend, the fabrics were first scoured with a solution containing 5 g/l mild detergent for 1 hour. They were then washed, rinsed thoroughly and dried in air to remove all the impurities and starch present.

2.2.5 Mordanting

The treated silk and polyester-cotton blended fabrics were mordanted by five different metal mordants using three different mordanting methods. The different mordants that were used on silk and cotton-polyester blend were prepared as discussed below.

To prepare a mordant, it was first mixed with cream of tartar (potassium hydrogen tartrate) in a small portion of boiling water (50 ml) and rest of the amount added later (150 ml). The mordant was dissolved in water to make the liquor ratio 1:50. The wet sample was immersed into the mordant solution and then it was heated. Temperature of the dye bath was raised to 60°C over half an hour, and left at that temperature for another 30 minutes. The mordanted material was then rinsed with water thoroughly, squeezed and dried.

Three different mordanting methods were adopted, which are discussed below.
a. Pre-mordanting method: In case of pre-mordanting method, the mordants were prepared as per the procedure mentioned above and dissolved in warm water at temperature of 60°C and the fabrics were soaked for 1 hour in the mordant solution before the dyeing of the samples.
b. Simultaneous-mordanting method – In this case, the fabrics were dyed in the same bath with dye and mordant put together.
c. Post-mordanting method – In this method, the fabrics were first dyed and subsequently mordanting was done.

2.2.6 Dyeing

A step wise dyeing of pre-treated, post-treated and simultaneous mordanted for the two different fabrics with five different mordants was carried out. The dying process was carried out in a dye bath for 3 hours at temperature of 65° C, keeping the M: L ratio as 1:40, and original pH level (6.2) maintained. Dyed fabric was washed with cold water and dried at room temperature. It was then dipped in 2% sodium chloride solution (brine) at room temperature for 1 hour for fixing. In the end, the dyed material was washed thoroughly in cold water to get rid of extra dye.

2.2.7 Measurement of color strength

The color yield of both dyed and mordanted samples were evaluated by light reflectance measurements using Premier Colorscan Machine.

The color strength (*K/S* value) was assessed using the following Kubelka – Munk Equation: [5]

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

where *R* is the decimal fraction of the reflectance of dyed fabric.

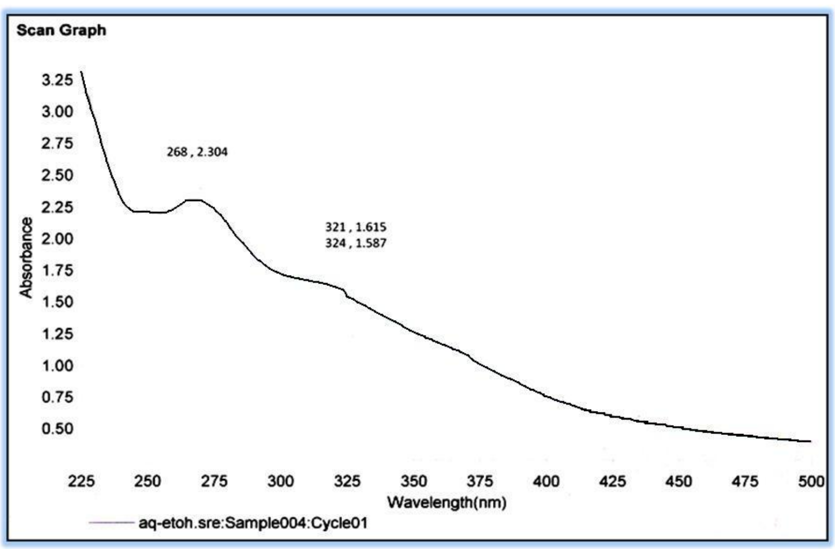
To investigate color quality in a systematic way, color measurement is vital. Color indices derived from CIE *L*a*b** measurements depict the hue color. The CIE *L*a*b** values were ascertained for five mordants and three different mordanting conditions for silk fabrics and polyester-cotton fabrics. Chroma (C) is a measure of intensity or saturation of color and Hue angle (H) is derived from the two coordinates *a** and *b**.

2.2.8 Fastness testing

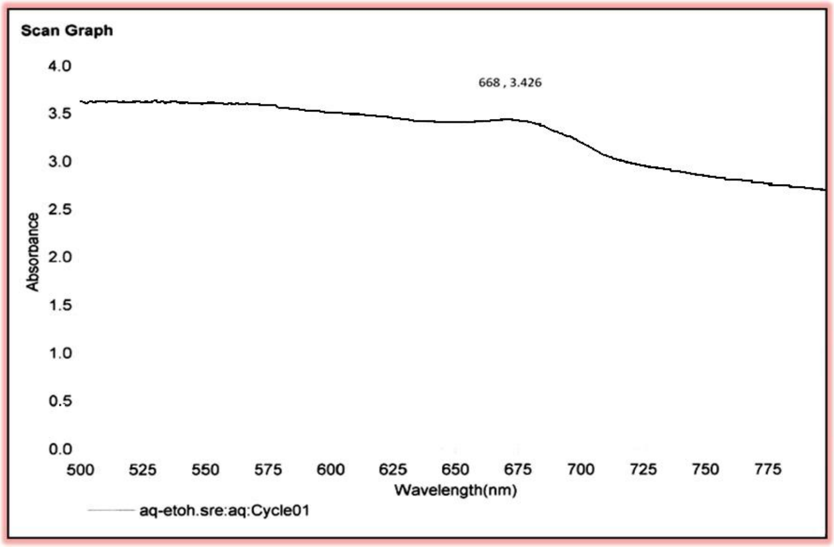
The dyed samples were tested according to AATCC standards. The specific tests were: Color fastness to washing -Test Method 61, 2 (A), Rubbing fastness (dry and wet) -Test Method 8, Sun-light fastness -Test method 16 specifications in coordination with AATCC Test method 181 [6].

CHEMICAL COMPOSITION OF THE COLORANT

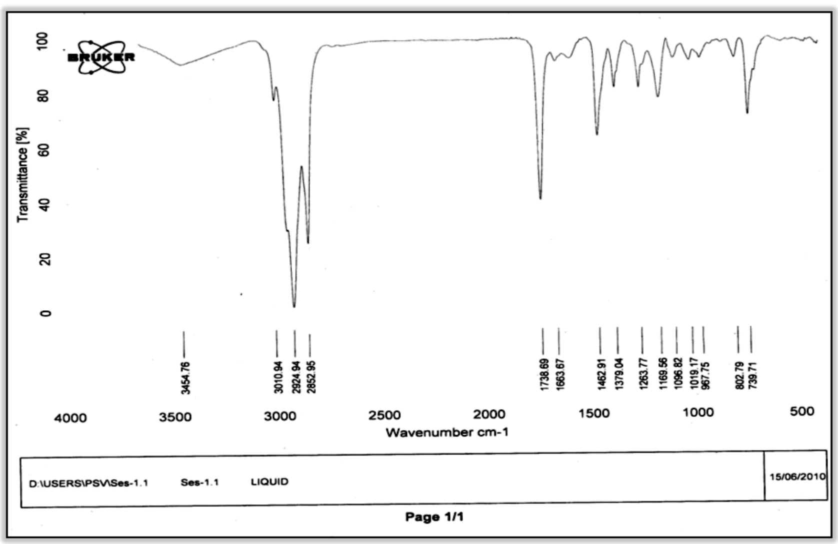
Through various measurement techniques like UV-VISIBLE, and IR spectroscopic techniques, used to analyze the pigments, the λ max of the *Sesbania aculeata* dye extract was found to be 268 nm. in the UV region, which falls in the spectral range of flavonoids (200-500 nm.), whereas in the Visible region the λ max was 668 nm., that is the spectral range of chlorophyll (refer Graph 1 and 2). The IR- Intense peak at 3340 cm-1 shows presence of poly-hydroxyl groups(Polyphenol), (refer Graph 3). It can therefore be inferred that the extract of the *Sesbania aculeata* plant contains flavonoids, clorophyll, and polyphenols (refer Figure 2).



Graph 1: UV Spectra of the aqueous extract of *Sesbania aculeata* plant



Graph 2: Visible Spectra of the aqueous extract of *Sesbania aculeata* plant



Graph 3: IR spectroscopy graph of *Sesbania aculeata* showing intense

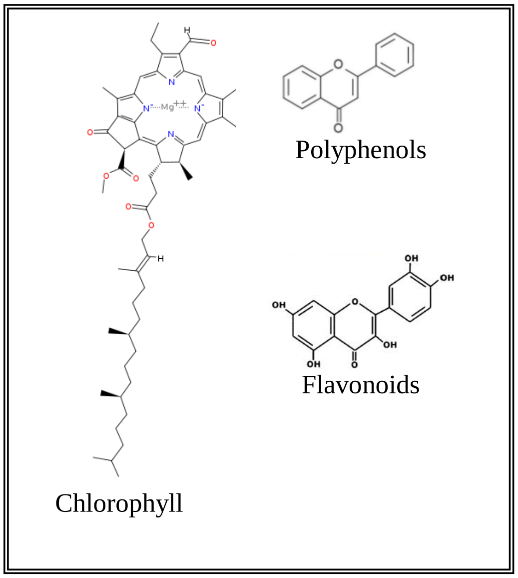
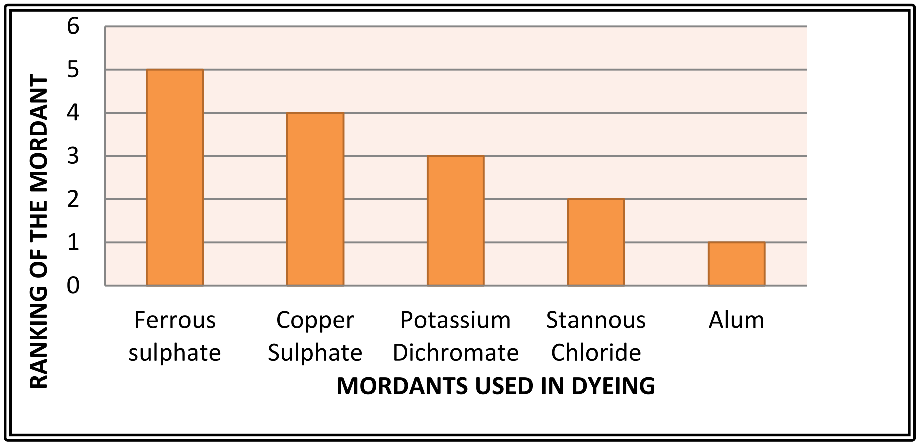


Figure 2. Chemical constituents of *Sesbania aculeata* plant extract

3. RESULTS AND DISCUSSIONS

4.1 Colors obtained from dyeing with Sesbania aculeata

Different shades were obtained on silk and polyester-cotton blend in controlled conditions with five mordants and three different mordanting conditions. Different shades obtained ranged from light to dark shades of beige, yellow and browns (*cream – beige – golden yellow - sandy brown – muddy brown – brown – tan*). In general, tans or dark shades were obtained through sulfates and chromes, while chlorides and alum gave lightest of creams and their tints. Ferrous sulfate and copper sulfate gave a dark range of browns on silk. The order of reactivity was Fe>Cu> Cr>Sn> Al as shown in Graph 3.



Graph 3: Ranking of mordants on the scale of 1-5 on the basis of depth of color obtained

The different shades obtained with the aqueous extract of *Sesbania aculeata* in controlled conditions, and with the use of different mordants and mordanting techniques as obtained on Premier Color Scan Spectrophotometer, are shown as in Figures 3 for (silk) and 4 for (polyester-cotton).

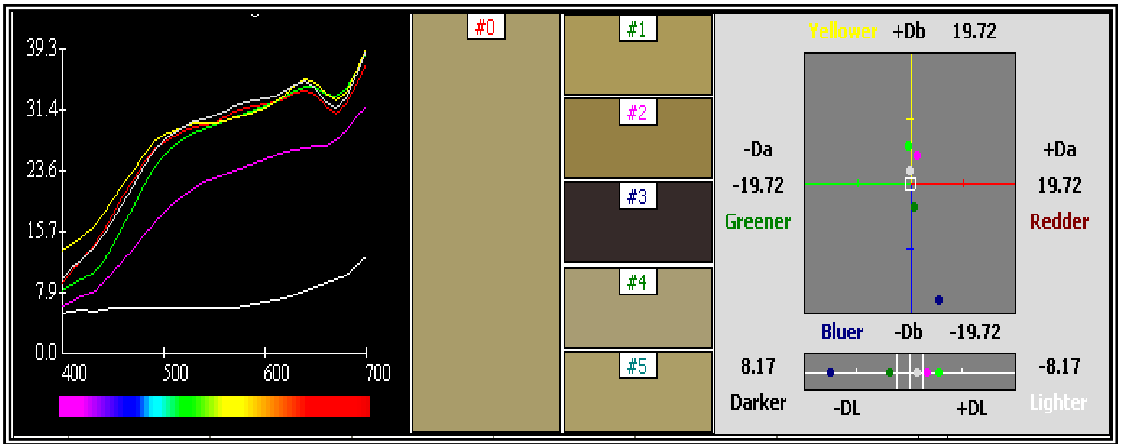


Figure 3. Silk - Pre mordanting shades (*0 – Control, 1 – Alum, 2 – Cuso$_4$, 3 – Feso$_4$, 4- $K_2Cr_20_7$, 5 – Sncl$_2$)*

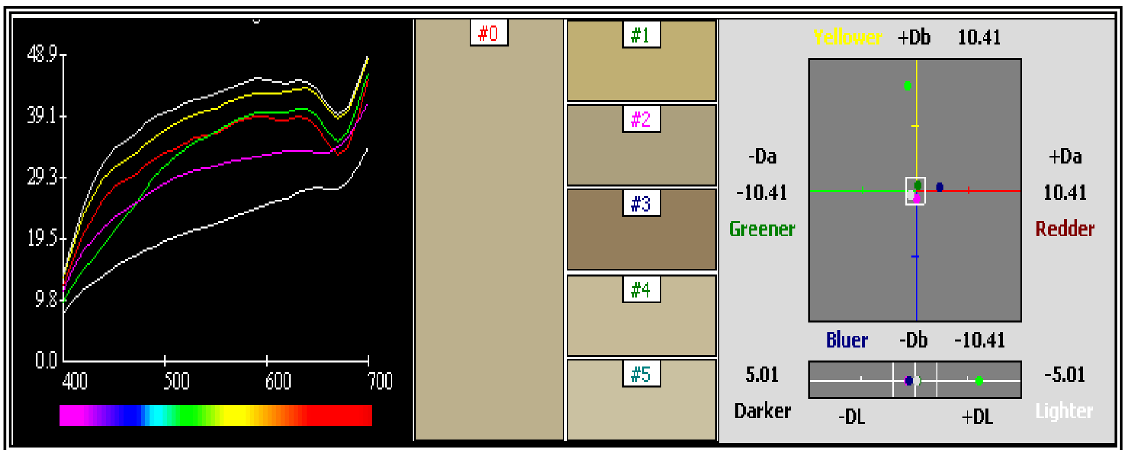


Figure 4. Polyester cotton- Pre mordanting shades (*0 – Control, 1 – Alum, 2 – Cuso$_4$, 3 – Feso$_4$, 4- $K_2Cr_20_7$, 5 – Sncl$_2$)*

4.2 Optimization of mordants with K/S and color hue changes

Different mordants were used in 1-2% concentration, keeping in mind the toxicity factor of some mordants, particularly, copper and chromium. Varied hues were obtained from pre, simultaneous and post-mordanted silk and polyester-cotton fabrics with Alum, $CuSo_4$, $FeSo_4$, K2Cr207, and $SnCl_2$ when dyed with aqueous extract of *Sesbania aculeata*. As shown in Tables 1 and 2 , the different mordants not only cause difference in hue color, *L** values and brightness index, but also cause significant changes in *K/S* values [7]. Through *L* a* and b** values, it can be seen that mordants which show higher values of *L**, show lighter shades, while lower *L** values signify deeper shades for the dyed fabrics. Similarly, negative *a** and negative *b** represent green and blue color respectively.

Table 1. *L* a* b* C* H values for pre, simultaneous and post-mordanted silk fabric dyed with *Sesbania aculeata***

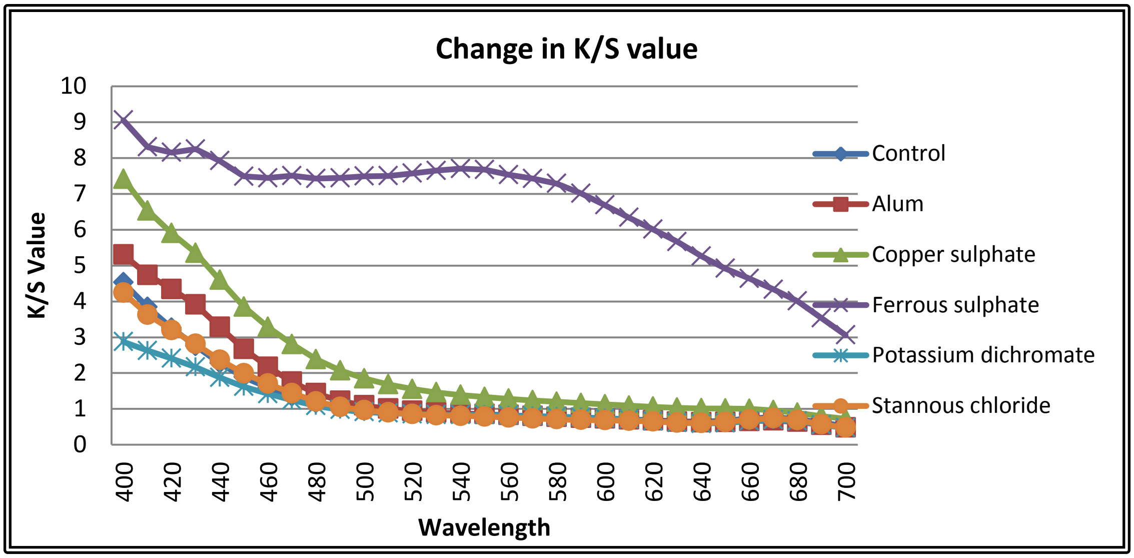
PRE - MORDANTING	L*	a*	b*	C*	H*
Control (Silk)	61.238	-2.231	19.386	19.514	96.598
Alum	63.531	-2.572	25.170	25.301	95.868
Copper sulfate	62.584	-1.040	23.819	23.842	92.535
Ferrous sulfate	55.072	3.172	1.667	3.583	27.712
Pot. dichromate	59.659	-1.517	15.988	16.060	95.454
Stannous chloride	61.794	-2.308	21.298	21.423	96.219
SIMULTANEOUS MORDANTING					
Control (Silk)	61.616	-2.158	19.601	19.719	96.316
Alum	64.529	-1.114	25.909	25.933	92.497
Copper sulfate	62.621	-2.508	22.420	22.560	96.416
Ferrous sulfate	61.820	0.150	20.961	20.962	89.554
Pot. dichromate	58.005	-1.298	10.175	10.257	97.303
Stannous chloride	62.880	-2.272	22.953	23.065	95.687
POST-MORDANTING					
Control (Silk)	61.530	-2.439	19.258	19.412	97.251
Alum	61.547	-2.574	19.080	19.253	97.716
Copper sulfate	60.886	-2.735	18.066	18.272	98.641
Ferrous sulfate	59.266	-0.895	13.939	13.968	93.709
Pot. dichromate	59.686	-0.230	15.441	15.443	90.889
Stannous chloride	64.632	-3.576	28.076	28.303	97.292

Analysis of the data of Table 1 reveals that the results of CIE *L*a*b** values of dyed silk fabrics are consistent in nature. The low values of *L** in case of ferrous sulfate and potassium dichromate used as mordants indicate the depth of color achieved. This shows that the coloristic efficiency of *Sesbania aculeata* dye is high for silk with Fe, Cu, Kr used as mordants. The general trend of the values obtained show trend towards greenness and yellowness in the sample with *a** having negative whereas *b** with positive values.

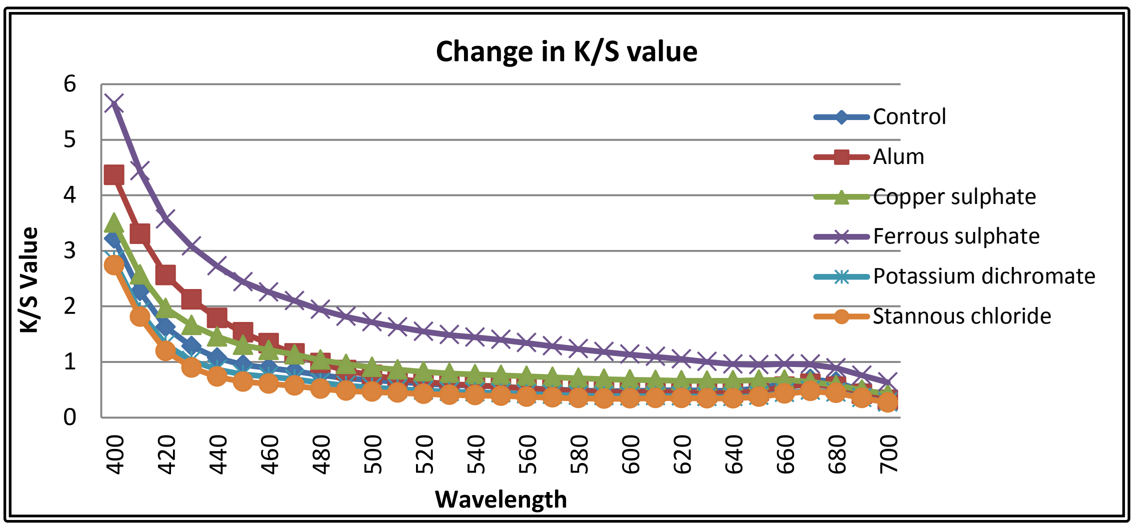
Table 2. *L* a* b* C* H values for pre, simultaneous and post-mordanted polyester cotton fabric dyed with *Sesbania aculeata***

PRE-MORDANTING	L*	a*	b*	C*	H*
Control (Poly+Cot)	66.574	-1.110	13.271	13.317	94.815
Alum	69.588	-1.936	21.679	21.765	95.137
Copper sulfate	66.197	-1.077	12.599	12.645	94.920
Ferrous sulfate	66.320	1.189	13.587	13.639	84.965
Pot. dichromate	66.708	-0.960	13.765	13.798	94.024
Stannous chloride	66.656	-1.566	12.925	13.020	96.942
SIMULTANEOUS MORDANTING					
Control (Poly+Cot)	67.815	-1.267	12.652	12.715	95.753
Alum	69.715	-0.975	18.284	18.310	93.087
Copper sulfate	68.011	0.529	14.277	14.287	87.843
Ferrous sulfate	68.096	0.792	14.402	14.424	86.817
Pot. dichromate	67.333	-1.803	10.552	10.705	99.729
Stannous chloride	69.334	-0.403	17.577	17.577	91.349
POST-MORDANTING					
Control (Poly+Cot)	67.193	-1.205	12.638	12.695	95.481
Alum	66.121	-0.649	8.730	8.754	94.286
Copper sulfate	66.130	-1.056	9.103	9.164	96.651
Ferrous sulfate	65.599	0.068	7.805	7.805	89.465
Pot. dichromate	66.958	0.218	11.790	11.792	88.905
Stannous chloride	68.187	-0.838	15.467	15.490	93.136

The relative comparison of both the fabrics, polyester blended fabrics show least *K/S* values in terms of dye absorption and the intensity of the shades in case of the blended fabrics. The review suggests that the natural dyes do not have much affinity for synthetics or their blends [8]. Different mordants show change in *K/S* values as shown in Graphs 4 and 5.



Graph 4: Change in K/S values of pre-mordanted silk dyed with aqueous extract of *Sesbania aculeata*



Graph 5: Change in K/S values of pre-mordanted polyester-cotton dyed with aqueous extract of *Sesbania aculeata*

4.3 Fastness of the dyed samples

The dyed samples were tested for the wash fastness. The grey scale rating was done on a scale of 1-5. Good wash fastness was achieved in pre-mordanted silk and polyester-cotton blend between 4 and 5 when compared with other mordanting techniques. The pre mordanting method forms an insoluble complex between dye molecule and metal mordant. This results in higher affinity (refer Table 3 and 4), and thus results in better fastness properties [9]. In case of other mordanting methods, the fastness properties observed was satisfactory.

Table 3. Fastness properties of silk dyed with aqueous extract of *Sesbania aculeate*

AQUEOUS EXTRACT	FASTNESS PROPERTIES			
SILK	**Wash**	**Dry Rubbing**	**Wet Rubbing**	**Light**
Un- mordanted	4	3.5	3	3
PRE-MORDANTING	**Wash**	**Dry Rubbing**	**Wet Rubbing**	**Light**
Alum	4.5	4	3.5	3.5
Copper sulfate	4	4	3	3.5
Ferrous sulfate	4	4	3.5	3
Potassium dichromate	4.5	3.5	3.5	3
Stannous chloride	4	4	4	3
SIMULTANEOUS-MORDANTING	**Wash**	**Dry Rubbing**	**Wet Rubbing**	**Light**
Alum	4	4	4	3.5
Copper sulfate	4	3.5	3	3
Ferrous sulfate	4	4	4	3
Potassium dichromate	4	3	3.5	3.5
Stannous chloride	3.5	3.5	4	4
POST-MORDANTING	**Wash**	**Dry Rubbing**	**Wet Rubbing**	**Light**
Alum	4	4	3	3.5
Copper sulfate	4	3.5	3	3
Ferrous sulfate	3.5	4	3	3.5
Potassium dichromate	3.5	3.5	3	3
Stannous chloride	3.5	3.5	3	3

Table 4. Fastness properties of polyester-cotton dyed with aqueous extract of *Sesbania aculeata*

AQUEOUS EXTRACT	FASTNESS PROPERTIES			
POLYESTER-COTTON	**Wash**	**Dry Rubbing**	**Wet Rubbing**	**Light**
Un- mordanted	3.5	3	2.5	2.5
PRE-MORDANTING	**Wash**	**Dry Rubbing**	**Wet Rubbing**	**Light**
Alum	4.5	4	3.5	3.5
Copper sulfate	4	3.5	3	3.5
Ferrous sulfate	4	4	3.5	3
Potassium dichromate	4	3.5	3.5	3
Stannous chloride	3.5	4	4	2.5

SIMULTANEOUS-MORDANTING	Wash	Dry Rubbing	Wet Rubbing	Light
Alum	4	4	3.5	3.5
Copper sulfate	3.5	3	3	2.5
Ferrous sulfate	3.5	3.5	3.5	3
Potassium dichromate	3.5	3.5	3.5	2.5
Stannous chloride	4	4	3.5	3
POST-MORDANTING	**Wash**	**Dry Rubbing**	**Wet Rubbing**	**Light**
Alum	4.5	4	3	3
Copper sulfate	3	3.5	2.5	2.5
Ferrous sulfate	4	4	3.5	3.5
Potassium dichromate	3.5	3.5	3	3
Stannous chloride	4	3.5	3.5	3.5

Dry rubbing fastness was better compared to the wet rubbing fastness. Sun light fastness of the samples dyed with *Sesbania aculeata* was found to be satisfactory. Better fastness properties were observed in silk as compared polyester-cotton.

4. CONCLUSIONS AND FUTURE RESEARCH

The environmental activists support the use of natural colorants as they are seen to be using renewable resources, causing minimum pollution and having low risk to human health. Aqueous extract of *Sesbania aculeata* yields shades of cream, beige, yellow and browns. The darkest shades were obtained with ferrous sulfate, alum gave the lightest shades. Vankar et al., 2009[10] also reports that the iron provides the dark shades obtained whereas Alum being alkaline in nature removes the colors and yields lighter shade.

The dye extract used for dyeing silk gave brilliant colors in conjunction with metal mordants compared to polyester cotton blend. Out of all the mordanting techniques, the best results were obtained in case of pre-mordanting method in terms of good dye ability and it also gave good to excellent wash and rub fastness.

Future research can be conducted by testing the application of the *Sesbania aculeata* dye on other fabrics with different mordants and mordanting techniques. Commercial aspects of the dye could also be worked out. Thus, application and use of this dye will contribute significantly in attaining a safe, eco-friendly and green environment.

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[i] *Agra is major urban city in Northern part of India with temperate climate and arid land. It is also known in the world for the famous monument 'Taj Mahal'.*