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A Study on Green Dyeing of Cotton with Ethanolic Extract of *Sesbania aculeata*

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Abstract:
In the recent past, the pollution resulting from the production and use of synthetic colorants, has received increased worldwide awareness. This has led to a significant revival of interest in natural colorants in the last few years. There is a realization amongst consumers and in textile industry for a need to re-invent natural dyes to impart color to textiles. This paper concerns with extraction of dyes from a plant, *'Sesbania aculeata'*, commonly known as Dhaincha. This plant belongs to the family of *Fabaceae*, which is commonly known as the legume family. It is a crop generally cultivated as green manure for increasing the nutritive value of the soil. This plant has low maintenance and tending operation costs with high yields. 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 edible or a highly commercial product. In this paper, ethanolic extract of *Sesbania aculeata* was used to dye cotton fabric using five different mordants with three different mordanting techniques. Color measurements were carried out to evaluate the shades obtained. The dyed fabrics were subjected for analysis in terms of *K/S* and CIE *L*a*b values as well as their fastness properties.**

Keywords: Natural dyeing, *Sesbania aculeata*, Ethanolic extraction, Cotton, Color measurements, Color fastness.

1.0 Introduction:
Natural dyes and colorants are an essential part of the world's ecological and cultural heritage with the use of plants, seashells and coccid insects to create color which is common to all civilizations(Adeel et al., 2009). Dyes from natural sources in India have an ancient history interwoven with its culture, with natural dyeing being practiced since Bronze Age. This era of natural dyes flourished till the discovery of man-made synthetic dye by William Henry Perkin in 1856. The first synthetic dye, mauveine and many more dyes which were developed thereafter, quickly replaced the traditional natural dyes (Druding, 1982). They were less expensive and offered a vast range of new colors. These artificial dyes imparted better properties to the dyed materials and thus ended the large-scale market for natural dyes. But yet again the revival of natural dyes has begun due to the hazardous and carcinogenic nature of synthetic dyes and an eco-friendly drive around the globe. Natural dyeing also symbolizes craft practices which reflect a harmonious, sustainable relationship with the eco system and the local plant reservoir (Cardon, 2010).

Indian flora and fauna are rich in many untapped sources that have not been explored so far. In this regard, plant sources which have potential use in textile dyeing industries could be explored further for commercialization. Natural dyes have better bio-degradability and are obtained from renewable sources. Efforts are now being made to identify the raw materials from plant sources and to standardize the recipes for their use. Thus, utilizing natural dyes to impart color on the fabric has a number of advantages over synthetic dyes. These include: (i) better eco-friendliness, (ii) possibility of premium pricing and (iii) variety of colors produced from single dye source. However, natural dyes have some disadvantages too, such as tedious extraction process, limited and dependent availability of sources along with some processing and application intricacies. Some of these limitations are being addressed by the researchers working in the area of natural dyes. (Sachan and Kapoor, 2007, Siva, 2007, Pruthi et al. 2010).Due to eco-consciousness, there is a growing awareness amongst consumers and in textile industry for a need to develop natural dyes to impart color to textiles. In continuation of this exploration, a plant, *'Sesbania aculeata'*, commonly known as Dhaincha, was chosen for the present research.

Sesbania is a species of flowering plants in the pea family, *Fabaceae*. *Fabaceae* or Leguminosae is an important and third largest family of flowering plants, which is commonly known as the legume family, pea family, bean family or pulse family. The name 'Fabaceae' comes from the defunct genus Faba. Fifty species of Sesbania have been described in tropical and subtropical regions of the world (Char, 1983). *Sesbania aculeata,* is the species most commonly found in India. Locally, it is known by the name Dhaincha, Danchi and Dunchi. It is an erect, low annual sub shrub and reaches up to height of one to two meters. It has fibrous, pithy stems with long leaves. The leaves are pinnate, 1.2–2.5 cm long, 0.3 cm wide and are glabrous. It bears purple-spotted yellow flowers from September to November in Indian climatic conditions (refer Figure a). It produces pods which contain light brown beans.

Figure a*: Sesbania aculeata* plant

Oven-dry fiber of *Sesbania aculeata* is reported to contain 0.71% ash, 0.94% fats and waxes, 2.3% nitrogenous matter, 9.76% pentosan, 16.3% lignin and 85.2% holocellulose (63.6% alpha cellulose). According to Duke (1981), seeds of the genus Sesbania are reported to contain trypsin inhibitors and chymotrypsin inhibitors. The leaves of *Sesbania aculeata* yields good concentration of (+)- pinitol which is an anti-diabetic agent. (Misra and Siddiqi, 2004).The plant *Sesbania aculeata* along with its various species like *Sesbania Sesban, Sesbania Grandiflora* has several uses as green manure (adding 150 kg nitrogen/hectare) and is used for erosion control, hedges, intercropping "mother plants," nitrogen fixation, and windbreaks, for fodder and fuel wood. It is used for manufacturing of paper, particle boards, pipes, ropes and as sizing and thickening agent. Lately, the *Sesbania aculeata* plant is used as biomass and supplies 128 kW of electricity at 240 V. in Bihar. (Prasad, 2009).It also has several medicinal uses and used in treatment of various eye, skin and inflammations (Oudhia, 2003).

2.0 Materials and Methods:

2.1 Materials:

2.1.1 Source: *Sesbania aculeata* plant locally known as "Dhaincha" was collected from the fields near Agra city, India.

2.1.2 Substrate: 100% cotton fabric was used, tested by NITRA Laboratories, Ghaziabad, India.

2.1.3 Chemicals: Laboratory grade solvents, reagents, and mordants such as Alum, Ferrous sulphate, Copper sulphate, Potassium dichromate and Stannous chloride were used.

2.1.4 Equipment'sUsed:

- A Soxhlet and a distillation assembly was used for ethanolic extraction of the dye.

- An open bath beaker dyeing machine equipped with programmable control of temperature was used to carry out all dyeing studies.
- The absorbance of dyeing solution was measured using a Shimadzu- UV visible spectrophotometer.
- *K/S* values and CIE *L*a*b** values were measured on Premier Color Scan Spectrophotometer.

2.2 Methods:

Solvent extraction method was used for extracting the dye. This extraction method is useful in case of dye plants containing flavonoids, anthraquinones and aglycones which are poorly soluble in water and therefore are extracted completely in the presence of solvents (Vankar, 2006).

2.2.1 Preparation of Raw Material:

The leaves and the stems were washed well with tap water to remove the dust and were dried in shade. The dried material was finely grounded to form a fine powder.

2.2.2 Extraction of Dye:

100 gms.of dried *Sesbania aculeata* plant material was weighed and 400 ml. of ethanol was added.It was heated to 60°C in a round bottom flask attached to the soxhlet for 60 minutes. The extract was then filtered, and the filtrate was evaporated to near dryness using a distillation assembly at 50°C. Distilled water was added to this extract obtained in purified form in the proportion of 1:4 (refer Figure b).

Figure b: Ethanolic extract of *Sesbania aculeata* plant

The absorbance of the dye solutions was measured on the spectrophotometer in order to identify the compounds present.

2.2.3 Scouring of Cotton Fabric:

For cotton fabrics, scouring was carried out with solution containing 5g/l of non-ionic detergent for 30 min. The scoured material was thoroughly washed with tap water, rinsed thoroughly and dried at room temperature to remove all the impurities and starch present. It was soaked in distilled water prior to dyeing or mordanting.

2.2.4 Mordanting:

The scoured cotton fabrics were mordanted by different mordants. Three different processes of mordanting used were pre, simultaneous and post-mordanting methods in concentration of 1-2%.
The mordanted material was then rinsed with water thoroughly, squeezed and dried.

2.2.5 Dyeing:

Dyeing of the mordanted samples was carried out in a dye bath at a temperature of 65° C for three hours. The material: liquior ratio was maintained as 1: 40. After the dyeing was complete dyed samples were dipped in brine solution at room temperature for 1 hour for fixing of the dye. The dyed material was dried and then washed thoroughly in cold water to get rid of extra dye.

2.2.6 Color Measurement:

Several color measurements were carried out to evaluate the shades obtained on dyeing cotton with ethanolic extract of *Sesbania aculeata*.

- The color strength (*K/S* value) was assessed using the Kubelka – Munk Equation:(Kamel, 2009), $K / S = (1- R)^2 / 2R$
  where *R* is the decimal fraction of the reflectance of dyed fabric.
- The CIE *L*a*b**, *C, H* values were ascertained for five mordants and three different mordanting conditions. Chroma *(C)* is a measure of intensity or saturation of color and Hue angle *(H)* is derived from the two coordinates *a** and *b*.*

2.2.6 Fastness Testing:

After the color measurements, the dyed samples were tested for fastness to various agencies.

- Wash fastness of the dyed samples was anaylsed as per the AATCC (American

Association of Textile Chemists and Colorists) Test Method 61, 2 (A). The dyed specimens were cut in size in 5 x 10 cm. and were washed in Launder-o-meter. A detergent solution was prepared using detergent at concentration of 5 gm. per litre. The prepared samples were then placed in the canisters along with the 8-10 steel balls and then fixed in the launder-o-meter. These were subject to 5 cycles for approx. 20 minutes with temperature maintained at 38˚C.

- Dry rubbing and wet rubbing fastness was tested as per AATCC Test Method 8. For testing the dry specimen, the dyed sample was fastened to crockmeter base and rubbed with white test cloth under controlled conditions for approximately 20 cycles. Similarly, for testing the wet rubbing fastness, test samples were dipped in distilled water and were squeezed between blotting papers under 454 grams (l lb.) for one second, and then the entire process of dry rubbing fastness was repeated.
- Sun-light fastness was evaluated with AATCC Test method 16 specifications in coordination with AATCC Test method181. The dyed samples were tested in day light behind glass. The experiment was carried out in day light exposure wooden cabinet with a glass top. The half portions of the dyed samples of size 3"x6" were covered with a thick black paper sheet, so that they are not exposed to the light. This testing was carried out in a 24 hour cycle (AATCC manual 2008). All the samples were rated on the AATCC Grey scale with 1-5 ratings. The rating 5 denoted the maximum color fastness whereas rating 1 was imparted for the lowest fastness ratings.

3.0 Results and Discussions:

3.1 UV- Vis Analysis:

The ethanolic dye extract was subjected to Shimadzu UV Spectro, Spectrophotometer for absorption spectra analysis. Wavelength of the dye - λ max was measured and the compounds present in the extracts were interpreted as shown in Figure c and d. The lambda max. of the ethanolic dye extract in the UV range was 268 nm., and 666nm. in the visible region. These values fall in the spectral range of Flavonoids and Chlorophyll compounds respectively.

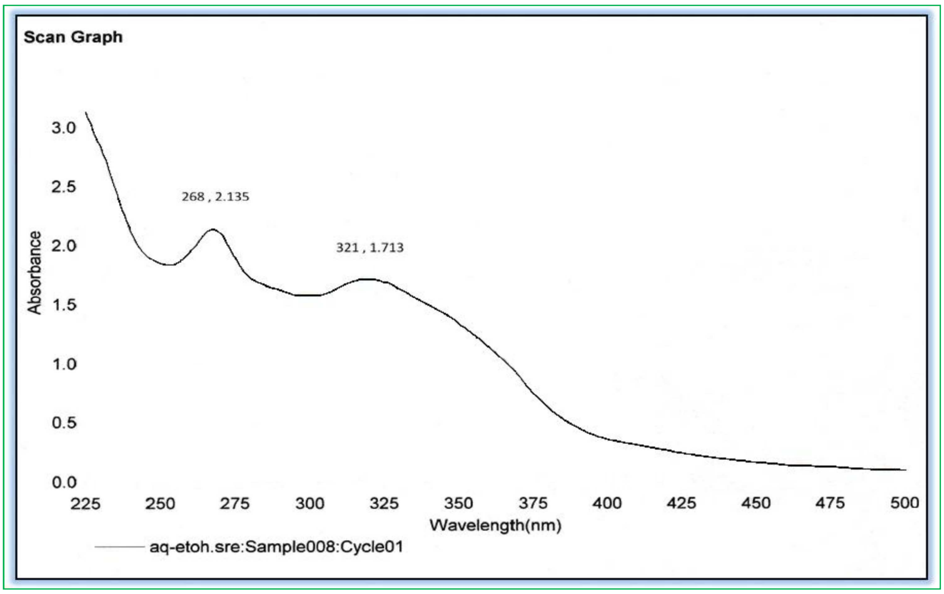


Figure c: UV Spectra of ethanolic extract of *Sesbania aculeata* plant

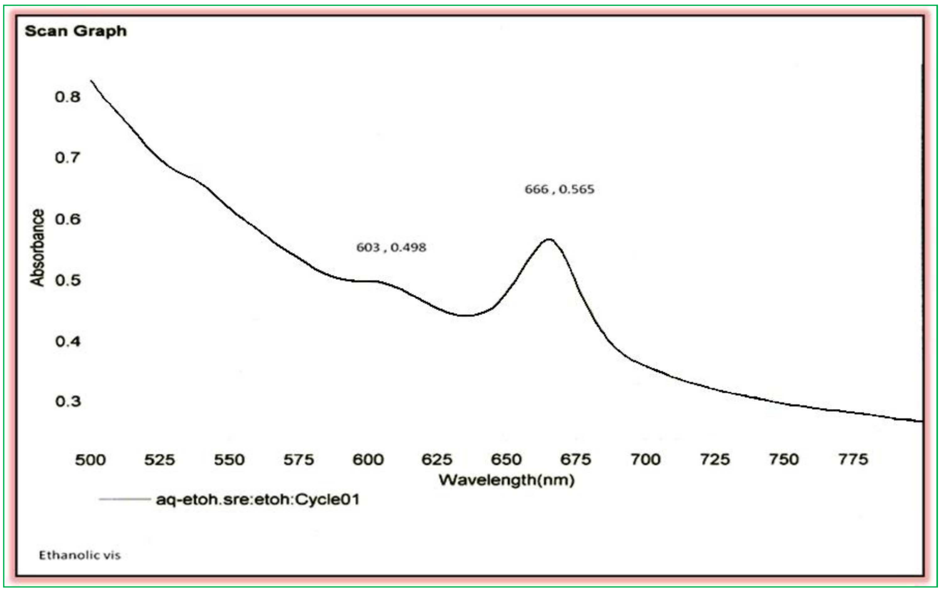


Figure d: Visible Spectra of ethanolic extract of *Sesbania aculeata* plant

3.2 Shades Obtained on the Dyed Fabrics:

The cotton samples dyed with ethanolic extract rendered light to dark shades of green. The shades ranged from – sap green – khaki – yellowish green – leaf green - army green – bottle green on the dyed fabrics, depending upon the mordant and the mordanting method used. The shade cards prepared on Premier Color Scan Spectrophotometer are shown as in Figure e.This pattern was followed in the researches carried out by Vankar et al. (2006) such as Dyeing of cotton with Hisbicus mutabilis or Sonicator dyeing of cotton, wool and silk with the Tagetus erecta extract (2009) indicated parallel results with similar shade cards being prepared for color evaluations. Kale et al. (2007) in a study on dyeing wool with Cosmos flowers and several others tabulated the similar parameters.

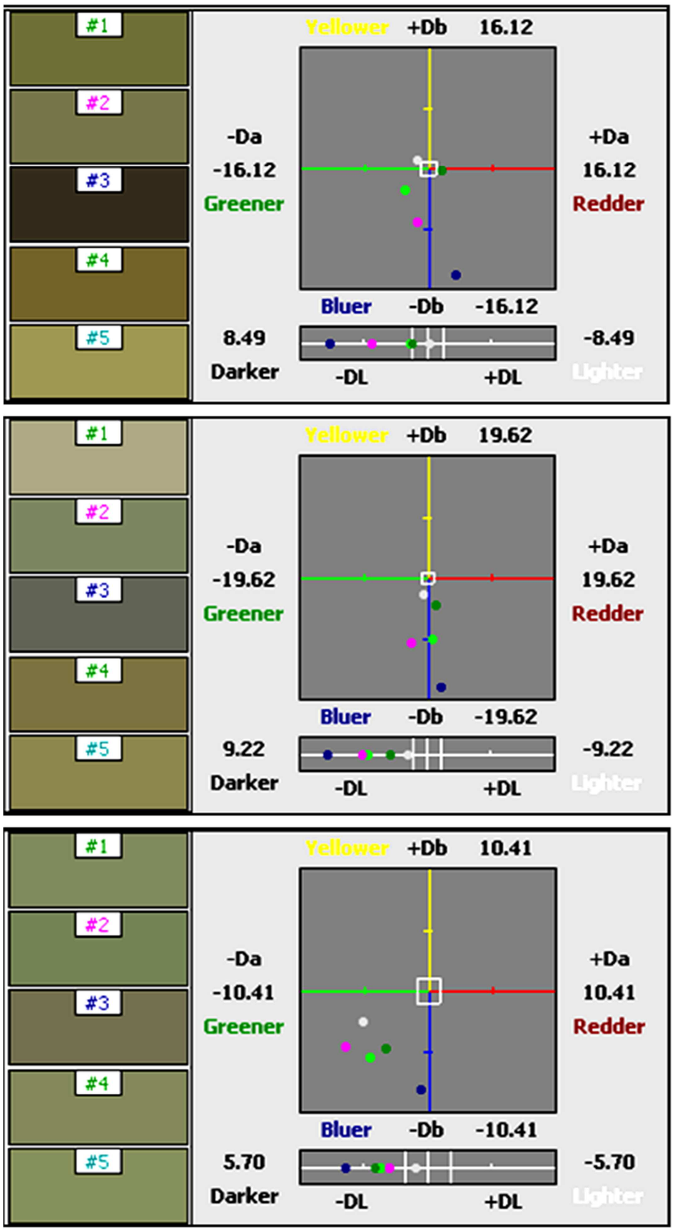
Mordants play an important role in imparting color to the fabric. Different shades and tints can be obtained by varying the concentration and combinations of different mordants (Kamel et al., 2009). It was thus observed that varied range of camouflage shades were obtained by change of mordants on dyeing with ethanolic extract of *Sesbania aculeata*. Usually tans, dark shades were obtained through sulphates and chromes while chlorides and alum gave lighter tints. According to Vankar (2009), alum, being alkaline in nature, removes the color and yields lighter shades with natural dyeing.

3.3 Measurement of K/S, L a* b*, C,* H *Values*:

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 cotton fabrics with Alum, Copper sulphate, Ferrous sulphate, Potassuim dichromate and Stannous chloride mordants when dyed with ethanolic extract of *Sesbania aculeata*. As shown in Table 1 the different mordants not only cause difference in hue color and in *L** values and brightness index values but also shows significant changes in *K/S* values. The higher the *L* number, the whiter the sample.Likewise, the lower the *L* number, the darker the sample. Similarly, the higher the *a* or *b* values, the redder or yellower the sample (Mehta and Bhardwaj 1998).

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. In the pre-mordanted samples dyed with ethanolic extract of *Sesbania aculeata,* the moderately low values of *L** showed the depth of shades of the samples. Similarly, negative *a** and negative *b** represent green and blue respectively (Vankar, 2009).In the pre-mordanting method highest *K/S* values were obtained with Ferrous sulphate as mordant (113.29) whereas in simultaneous-mordanting process, the

K/S values were lowest (17.07) with alum as mordant. The *a** values were negative, signifying the variation of green and grey shades in the samples. The relative comparison of these different mordanting methods shows that the pre-mordanting technique with mordants gave the best results in terms of shades achieved (refer Figure f). Since the pre-mordanting method forms an insoluble complex between dye molecule and metal mordant resulting in higher affinity acquired (Samanta, 2009), the simultaneous-mordanting and post-mordanting method gave average dyeing results in terms brightness of shades achieved as the mordant does not have enough chance to act upon the fabric.



(a) Pre- Mordanting method

(b) Simultaneous-Mordanting method

(c) Post-Mordanting method

(1 – Alum, 2 – $CuSo_4$, 3 – $FeSo_4$, 4- $K_2Cr_20_7$, 5 – $SnCl_2$)

Figure e: Colorimetric shades & values of dyed cotton with ethanolic extract of *Sesbania aculeata*

Table 1: Color Measurements for pre, simultaneous and post-mordanted dyed cotton with ethanolic extract of *Sesbania aculeata*

Pre-mordanting (Ethanolic)	***K/S***	***L****	***a****	***b****	***C****	***H****
Control	49.96	51.43	-3.44	21.31	21.58	99.15
Alum	55.47	50.27	-6.32	18.43	19.48	108.97
Copper sulphate	41.58	47.77	-4.82	14.23	15.03	108.75
Ferrous sulphate	113.29	44.95	-0.04	7.19	7.19	90.39
Potassium dichromate	64.78	50.43	-1.69	21.07	21.18	94.62
Stannous chloride	31.55	51.54	-4.77	22.38	22.89	102.07
Sim-mordanting	***K/S***	***L****	***a****	***b****	***C****	***H****
Alum	17.07	46.95	-2.72	11.54	11.86	103.28
Copper sulphate	30.84	46.55	-5.95	10.86	12.38	118.77
Ferrous sulphate	43.91	44.01	-1.47	3.69	3.98	111.81
Potassium dichromate	45.76	48.51	-2.16	16.93	17.07	97.32
Stannous chloride	37.17	49.86	-4.07	18.60	19.04	102.39
Post-mordanting	***K/S***	***L****	***a****	***b****	***C****	***H****
Alum	31.76	47.54	-6.62	12.81	11.86	117.36
Copper sulphate	36.17	47.97	-8.70	13.37	12.38	122.40
Ferrous sulphate	40.72	45.98	-2.55	10.10	3.98	104.18
Potassium dichromate	31.38	47.34	-5.29	13.56	17.07	111.34
Stannous chloride	31.73	49.11	-7.21	15.84	19.04	114.51

3.4 Fastness of the Dyed Samples:

The auxochrome of the dye, rate of the diffusion of the dye and the state of the dye inside the fibre are responsible for fastness properties of the dye (Jothi, 2008).The dyed samples were tested for the wash fastness and grey scale rating was done. Good wash fastness was achieved in pre-mordanted cotton between fastness grade of 4 and 5 when compared with other mordanting techniques. The wash fastness of the dyed samples was excellent whereas rubbing fastness ranging between 3.5 - 4 was good. Average to good light fastness was observed in cotton dyed with this dye (refer Table 2).

As observed, the pre-mordanted samples dyed with ethanolic extract of *Sesbania aculeata* had better fastness properties as compared to other mordanting methods (refer Figure g). Similar results on the best mordanting techniques in natural dyeing were obtained by Deo and Desai (2006) when dyeing of cotton and jute with tea as a natural dye. Vankar et al. (2006) report similar results while dyeing cotton and silk fabrics with natural dyes such as Terminalia arjuna, Punica granatum and Rheum emodi and so on.

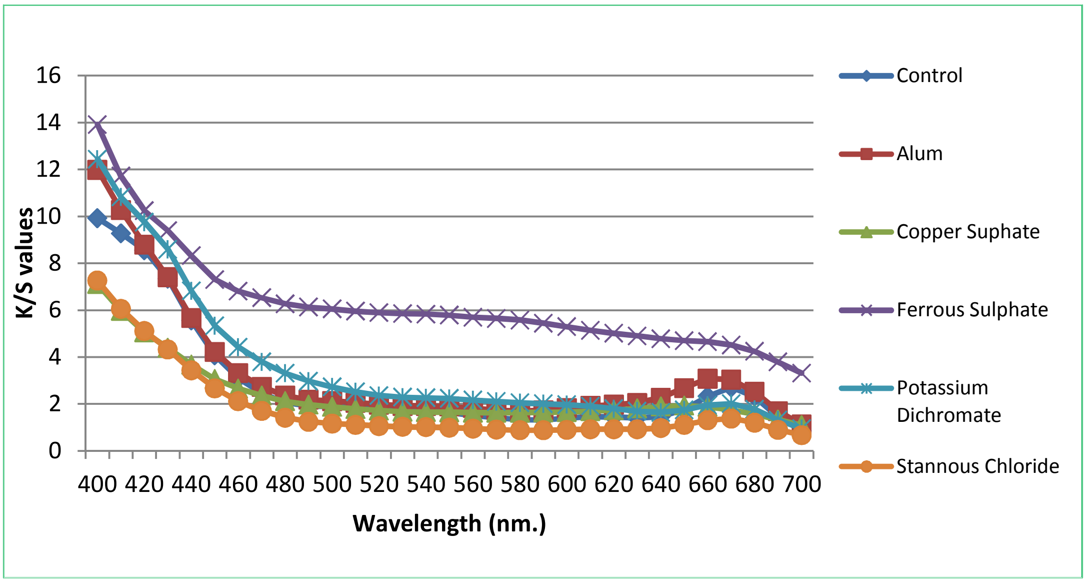


Figure f: Change in *K/S* values of pre-mordanted cotton dyed with ethanolic extract of *Sesbania aculeata*

Table 2: Fastness properties of cotton dyed with ethanolic extract of *Sesbania aculeata*

Ethanolic extract	Fastness properties			
	Wash	**Dry Rubbing**	**Wet Rubbing**	**Light**
Un- mordanted	3	3	2.5	2.5
Pre-mordanting	**Wash**	**Dry Rubbing**	**Wet Rubbing**	**Light**
Alum	4.5	4	4	4
Copper sulphate	4	3.5	3	3.5
Ferrous sulphate	4	4	3	4
Potassium dichromate	3.5	3	3	3
Stannous chloride	4	3.5	3.5	3.5
Simultaneous- mordanting	**Wash**	**Dry Rubbing**	**Wet Rubbing**	**Light**
Alum	4	3.5	4.5	3.5
Copper sulphate	3.5	3	2.5	3
Ferrous sulphate	4	4	3.4	3.5
Potassium dichromate	3.5	3.5	3	3
Stannous chloride	4	3	3.5	4
Post-mordanting	**Wash**	**Dry Rubbing**	**Wet Rubbing**	**Light**
Alum	4	4	4	3
Copper sulphate	3	3	3	2.5
Ferrous sulphate	3.5	4	3.5	3.5
Potassium dichromate	3	4	3	3
Stannous chloride	4	3.5	3.5	3.5

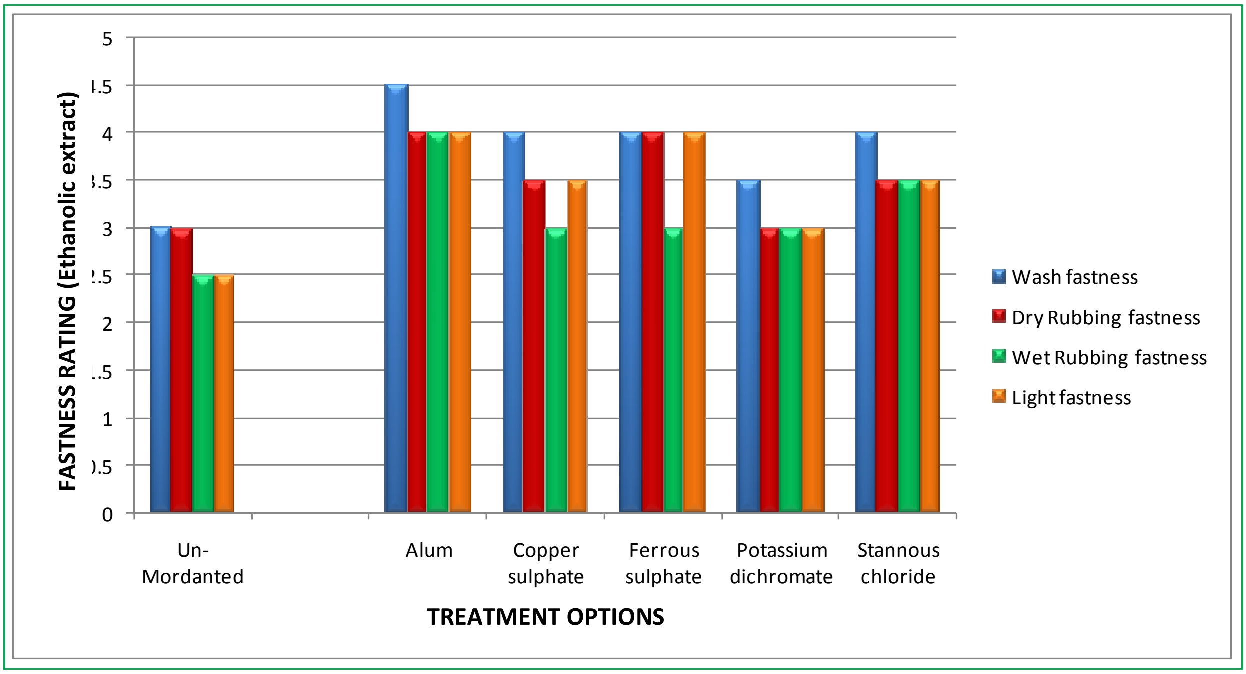


Figure g: Fastness ratings of un-mordanted and pre-mordanted cotton dyed with ethanolic extract of *Sesbania aculeata*

4.0 Conclusions and Future Research:

The present works shows that the ethanolic extract of *Sesbania aculeata* yields a range of camouflage shades. The fastness properties of the dyed samples were quite good. The plant of *Sesbania aculeata* is easy to tend and maintain, hence the plant economics is not high. While the exact cost benefit analysis was not conducted under the purview of the current experimental set up, some preliminary idea about the same can be obtained by referring to the similar studies documented in the past. For example, Kulkarni et al. (2011) looked upon dyeing of cotton with natural dye extracted from Pomegranate (Punica granatum) peel. This study carried out the cost benefit analysis of 1 kg. of pomegranate dye. This included the cost of raw materials and chemicals used, electricity consumption, packing transportation, labour and administration charges. Accordingly, the selling price of natural dye obtained from *Sesbania aculeata* could be calculated. The authors are continuing to perform experiments in this general area of research and exact economic analysis is currently under way and will appear as part of future research work. Hence, preparation of dry powder from the leaves of *Sesbania aculeata* can be a cheap source of natural dye having a good shelf life. Thus, application and use of this dye will contribute significantly in attaining a safe, eco-friendly and green environment.

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