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Ethiopian Dye plants As a Source of Natural Dyes for Cotton Dyeing

[1]Abera Kechi , [2]R.B. Chavan , [3]Reinhart Moeckel

[1, 2] Institute of Technology for Textile, Garment and Fashion Design, Bahir Dar University, Ethiopia
[3] Textile Technologies and Design Faculty, Hof University of Applied Sciences, Germany

[2]Corresponding author: rbchavan@hotmail.com

Abstract:

The increasing demand for eco-friendly and sustainable dyes for textile coloration has prompted the revival of natural dyes particularly in Asian countries. However, in African country like Ethiopia though very rich in biodiversity, no scientific study has been made to identify natural dye yielding plants and explore the possibility of using natural dyes for coloration of natural fibers. Traditional natural dyeing experience is very limited and remains only in the hands of few craftsmen. In the present study an attempt has been made to identify 13 dye yielding plants from north- west and south- west regions of Ethiopia. The aqueous color extracts were used for the dyeing of un-mordanted and pre-mordanted cotton. The dyed samples were analyzed in terms of color yield (K/S), color difference (L* a* b*) and wash, rubbing and light fastness properties. As expected the cotton samples pre-mordanted with alum and ferrous sulphate showed increased depth of shade and improved wash and rub fastness compared to un-mordatned cotton. Though the wash and crock fastness results were commercially acceptable, light fastness was poor and there was a scope for its improvement. The study is of practical nature and of industrial importance. The chemical structure analysis of the color extracts is not within the scope of the present study.

Keywords: Color; Dye plant; Dyeing; Extraction; Fastness; Mordant

1.0 Introduction:

Natural dyes were the main source of colorants for textiles until the end of 19th century. However, after the discovery of synthetic dyes, natural dyes were completely replaced by synthetic dyes. The ease of application, availability of wide range of colors, better reproducibility and improved quality of dyeing at lower cost are the main advantages of synthetic dyes over natural dyes. However, many of the synthetic dyes particularly azo dyes are found to be carcinogenic and harmful to health and environment (Ratna, 2012). The present awareness and demand for environmentally friendly textile products has been the driving force for a revival of natural dyes for textile coloration. The demand for natural dye also equally increased for coloration of food, pharmaceutical and cosmetics products. Approximately 10,000 different types of synthetic dyes and pigments are produced worldwide annually. According to the Global Industry Analysis (GIA) forecast the global market for pigments and dyes is to reach 9 million tons and US$24.2 billion by the year 2015 (GIA 2013). It is estimated that 10-15 % of dye is lost in the effluent during dyeing process (Iqbal and Ashiq, 2007).The environmental impact caused by textile processing can be minimized either by constructing effective effluent treatment plants or by using dyes and chemicals that are environmentally friendly. One of the possible solutions for the second alternative is using of natural dyes. It is for this reason; nowadays considerable research work is being undertaken around the world on the application of natural dyes for textile coloration.

Nature has gifted us more than 500 dye yielding plant species which can be used for coloration of textiles (Naqvi, 1980, Mahanta, and Tiwari, 2005). Coloring agents of these plants are derived from various parts of plants such as roots leaves, barks, and fruits (Adeel, *et al*., 2009, Arora etal., 2012, Goodarzian and Ekrami, 2010). Dye yielding plants, unlike synthetic dyes may contain more than one chemical constituent, each exhibiting different color and properties operating singly or in combination with the different groups, depending on their chemical structure and composition(Siva,2007;

Samanta and Agarwal, 2009, Wanyama et al., 2010). The colorants from plant origin used in dyeing different fibers are mainly flavonoids, anthraquinone and indigoids. Most commonly found flavonoids are flavonols, flavonones, and anthocyanins (Bechtold et al., 2006, Mishra et al., 2012). These flavonoids give variety of yellow, brown and green shades (Tiwari et *al*., 2010). Many researchers have shown that few natural dyes possess desirable coloring properties and good performance on natural fibers which are comparable to highly rated synthetic dyes (Siva, 2007; Purohit et *al*., 2007; Padama and Rakhi, 2007; Kadolph, 2005). Most of the natural dyes have no substantivity for the fiber except indigo and required to be used in conjunction with mordants (Dominique, 2007). A mordant usually is a metallic salt which forms a coordinating complex with the coloring component of natural dye and also a link with the fibre. Alum, Ferrous sulphate, copper sulphate, Sodium or Potassium dichromate, Stannous chloride are commonly used mordants for natural dyeing. Among these metal salts presently only Aluminum and Ferrous salts are recommended on the ground of environmental friendliness (Samanta and Konar, 2013).

In Ethiopia natural dyes have been utilized for colorations of textiles for a long time as evidenced from the colorful pictures drawn on canvas before 19th century and ancient dyed cultural clothes found in Orthodox churches and museums.

Figure 1 **Traditional Ethiopian painting with natural colors**

(Source: http//www.google.co.in/Search/Traditional Monastery cloth of Ethiopia/Images)

Ethiopia is reported to have over 6500-7000 plant species of which about 15 % are endemic (Centre for education, 2012). However the dye-yielding plants in Ethiopia have neither been identified, nor documented scientifically. In view of the imminent and large biodiversity of the country it is expected that several plants may have important color constituents for textile coloration. The present study has been designed to secure this gap and it is focused on identification of few potential dye plants through dyeing and testing the color strength, CIE L* a* b* values and fastness properties of dyed sample using standard methods. Structural identification of the exact nature of the chemical compounds of dyes, which are responsible for color are beyond the scope of the current study.

2.0 Materials and Methods

2.1Materials

2.1.1 Fabric

A commercially prepared ready- to-dye, plain weave 100 % cotton fabric of 140 g/m^2, 32 ends/ inch and 24 picks/ inch was purchased from Bahir Dar Textile Share Company, Ethiopia.

2.1.2 Dye plants

Traditionally used 13 varieties of dye yielding plants were collected from North-West region (Daga Esthifanos Monastery) situated in Lake Tana and South-West (Bonga) region of Ethiopia. The sites from where dye plants sources were collected are shown in Figure 2 and the biodiversity of North-West region is shown in Figure 3

North -West Ethiopia: Daga Esthifanos, where dye plant samples were partly collected

South -West Ethiopia: Bonga another site where dye plant samples were partly collected

Figure 2 North-West and South-West regions of Ethiopia

Figure 3 Bio-Diversity of North-West region of Ethiopia (Source:http://www.panoramio.com/photo/86282546)

The Ethiopian (Amharic) and scientific names of dye plants are given in Table 1. The Amharic names of dye plants are used in the text.

Table1: The Amharic and scientific names of dye plant sources

S No	Amharic name	Scientific name	Plant part used	Geographical distribution
1	Abalo	Combretumcollinum	Leaves	Ethiopia, Southern -Africa, Madagascar…
2	Bamiba	Ficus sycamores	Bark	Ethiopia, Kenya, Sudan, Egypt, Eritrea…
3	Dokima	Syzygium guineense	Bark	Ethiopia & many parts of Africa…
4	Fesson	Scadoxus multiflorus	Flower	Ethiopia,Zimbabwe,South Africa …
5	Gimea	Tagets minuta	Flower	Ethiopia, Eritrea, Djibouti, Kenya…
6	Gesho	Rhamnus prinoides	Leaves	Occurs from Ethiopia to South Africa
7	Nech Girar.	Acacia sp.	Flower	Ethiopia, Madagascar, Pacific Islands....
8	Inkoy	Flower Ximenia A.	Flower	Native to tropics including Ethiopia…
9	Tikur –inchet	Pygeum Africanum	Leaves	Ethiopia and most African countries....
10	Warica	Ficus sycomores	Bark	Ethiopia, Kenya, Sudan, Egypt…
11	Wonbela	Eriobotrya japonica	Bark	Ethiopia, Egypt, Kenya, Sudan ….
12	Zana	Stereospermum	Leaves	Ethiopia, Zambia, D.R.of Congo…
13	Zembaba	Stereospermum	Seed	Ethiopia, Senegal, Northern part of Sub –Saharan Africa...

2.1.3 Chemicals

Laboratory grade alum (KAl $(SO_4)_2$ .12 H_2O) and ferrous sulphate ($FeSO_4$. 7 H_2O) were used as mordants. Sodium sulphate (Na_2SO_4) was used to facilitate dye bath exhaustion. Industrial soap was used for wash fastness test. The dye baths were maintained at pH 7 using dilute solution of sodium carbonate (Na_2CO_3).

2.2 Methods

2.2.1 Color Extraction

The selected natural dye plant sources were first dried by exposing to sunlight, and then converted to powder form by using laboratory size reduction machine. Powdered dye plant sources were conditioned at room temperature for 24 hours before storing in to plastic container with screw cap. Color extraction from the known weight (70% o.w.f) of dye plant source was carried out for 1 hour by boiling with water with 1:20 material liquor ratio (MLR). The mouth of the beaker was covered with aluminum foil to prevent the excessive loss of water during extraction through evaporation. After the boiling period the dye extract in hot condition was filtered through fine cotton fabric. The filtrate was cooled and again filtered through Whatman filter paper.

2.2.2 Mordanting

Cotton fabric sample was pre-mordanted with alum and ferrus sulphate mordants at 100^0 C for 45 minutes with 1:40 MLR and with concentration of 5% o.w.f. The pre-mordanting was carried out in the machine used for dyeing.

2.2.3 Dyeing

Dyeing of unmordanted and pre-mordanted cotton fabric was carried out using the extracts of each dye plant. The dye plant source used was 70% on weight of fabric (o.w.f.). Each dye extract was diluted suitably to get 1:20 M L ratio for dyeing. The dyeing temperature was raised to 80^0 C with gradient of 2.5^0C/minute and dyeing was continued for 1 hour using Mathis LABOMAT sample dyeing machine. Sodium sulphate with concentration of 3g/L was added to dye bath after half of the dyeing time to facilitate exhaustion. After completion of dyeing time the fabric was rinsed several times with cold water followed by hot water. The dyed samples were then air dried.

2.2.4 Fastness

Dyed fabric samples were tested for wash, light and rubbing fastness properties. Wash fastness was performed at 40^0C and 60^0C according to ISO 105 C10: 2006 (A) and (C) test specifications respectively. Rubbing and light fastness tests were performed using ISO 105 –X12:2002 and DIN EN ISO test methods.

2.2.5 Color strength (K/S)

Reflectance of the dyed samples was measured on Minota Chroma-Meter CR 210 spectrophotometer. Using the built-in software program of the instrument the color strength was expressed in terms of the highest K/S value.

2.2.6 CIE L*a*b*

The same instrument was used for measuring CIE L* a* b* and K/S values. During measuring of the CIE L* a* b*, the sample dyed without mordant was considered as standard and samples dyed with mordants were considered as matches.

3. Results and Discussion

3.1 Dyeing of cotton

Before dyeing, the color from each dye source was extracted by boiling with water. The pH of the aqueous extracts was measured at 25^0C using VWR110 pH meter (Table 2).

Table 2: pH of dye extracts in aqueous medium

Dye Source	pH of extract
Abalo	3.48
Bamiba	4.86
Dokima	4.80
Fesson	4.42
Gimea	4.67
Gesho	4.23
Nech- Girar	3.95
Inkoy	4.39
Tikur -inchet	4.39
Warica	3.37
Wonbela	4.94
Zana	5.05

All the extracts showed acidic pH and the acidity varied from plant to plant. The acidity of dye plant extracts may be due to the presence of acid component like Tannic acid or Gallic acid along with other coloring components in each dye plant source.

Extracted dye solution was neutralized to pH 7 using dilute solution of sodium carbonate before dyeing. The objective of including direct dyeing technique (without mordant) in the present study was to identify the dye plants having direct affinity to cotton fabric in the absence of mordant (Tiwari et *al*., 2010; Dominique, 2007; Kulkarni, *et al*., 2011; Samanta and Agarwal, 2009; Tsatsaroni and Liakopoulou-Kyriakides, 1995).

3.2 Fastness properties

The purpose of dyeing is not only to impart color to the fabric but the color should be resistant to external agencies like washing, light and rubbing/crocking for commercial acceptability. Hence it was essential to test these fastness properties of shades produced from various dye plants selected in the present study.

3.2.1 Wash fastness

The dyed samples were subjected to wash fastness at 40^0 C and 60^0 C. The wash fatness results shown in Table 3 are expressed in terms of change in shade and degree of staining of white fabric.

In case of direct dyed (without mordant) samples, 9 dye plants, out of 13 have shown commercially acceptable wash fastness grade i.e. ≥ 3 at 40^o C, whereas only 4 dye plants showed fastness grade of ≥ 3 at 60^0 C washing. The Wash fastness for most dye plants was improved with alum and iron mordants particularly at 40^0 C wash test. With increase in washing temperature from 40^0 C to 60^0 C, the fastness grades were decreased for most dye plants. The improvement in wash fastness with iron mordanting was more significant than alum mordanting both at 40^0 C to 60^0 C wash tests. An improvement in fastness grade for mordanted cotton was expected due to metal complex formation between the coloring component and the metal salt. All the dyed samples showed fastness grade of 4-5 for degree of staining indicating the absence of staining of adjacent white fabric during washing both at 40^o C and 60^0 C for mordant and unmordant dyed cotton.

Table 3: Wash fastness of dyed cotton

Dye plant	Color change at 40^0C			Color change at 60^0C			Staining of at 40^o C			Staining of at 60^o C		
	DD	AMD	IMD	DD	AMD	IMD	DD	AMD	IMD	DD	AMD	IMD
Abalo	4	4	4	3	3	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Bamiba	2-3	4	3	1-2	2	3-4	4-5	4-5	4-5	4-5	4-5	4-5
Dokima	3	3-4	4	3	3	3-4	3-4	3-4	3-4	3-4	3-4	3-4
Fesson	3-4	3	4	2	2-3	3	4-5	4-5	4-5	4-5	4-5	4-5
Gimea	4	3-4	3	1	2-3	2-3	4-5	5	5	4-5	4-5	4-5
Gesho	2	3-4	3	1-2	1-2	1-2	4-5	4	4-5	4-5	3	3
Nech-grar	4	4	4-5	3-4	3-4	4	4	4-5	4-5	4	4-5	4
Inkoy	2-3	2	3	1	2	2	4-5	4-5	4-5	4-5	4-5	4-5
Tikur-inchet	4	4	4	3-4	3-4	3-4	4-5	4	4	4	4	4-5
Warica	2-3	2-3	4	1-2	2-3	3	4-5	4	4-5	4	4-5	5
Wonbela	4	3-4	3-4	2-3	4	3	4-5	5	4-5	4-5	4	4-5
Zana	3-4	3-4	4-5	2	2-3	2-3	4-5	4-5	4-5	4-5	4-5	4-5
Zembaba	4	4-5	4-5	3-4	3-4	3-4	5	4	4-5	4	4	4-5

Note: DD: Direct dyed; AMD: Alum mordant dyed; IMD: Iron mordant dyed.

3.2.2 Rubbing and light fastness

Table 4: Rubbing and Light fastness of dyed cotton

Dye plant	Dry rubbing fastness (Staining)			Wet Rubbing fastness (Staining)			Light fastness (Blue Wool standard)		
	DD	AMD	IMD	DD	AMD	IMD	DD	AMD	IMD
Abalo	5	5	5	4-5	3-4	4	3	3-4	5
Bamiba	5	5	5	4-5	3-4	4	2	1-2	4
Dokima	5	5	5	4	4-5	4-5	2-3	3	2
Fesson	4-5	5	5	5	5	4-5	3	3	2
Gimea	5	4-5	5	4-5	3-4	4	2-3	1-2	3-4
Gesho	5	5	5	4-5	4	4	3	2-3	3
Nech-grar	5	5	5	4-5	4-5	3-4	3	3	3
Inkoy	5	5	5	4	4	4-5	1-2	1-2	2-3
Tikur-inchet	5	5	5	3-4	4	4-5	1-2	2	3
Warica	5	5	5	3	3-4	3-4	2	2-3	2-3
Wonbela	5	5	4-5	4	3-4	3-4	2-3	2-3	3
Zana	5	5	5	5	4	4	3	1-2	3
Zembaba	5	5	4-5	4	4-5	4-5	2-3	2	3-4

Note: DD: Direct dyed; AMD: Alum mordant dyed; IMD: Iron mordant dyed.

The dry and wet rub fastness varied from 3-4 to 5. Most of natural dyes show moderate to good rub fastness and does not require any after treatment (Samanta and Agarwal, 2009),. The observations on rub fastness shown in Table 4 are in accordance with the literature.

Light fastness property of most dye plants as shown in Table 4 is poor to moderate except for iron mordanted Abalo and Bamiba. General comparison of light fastness of unmordanted and mordanted dyed cotton did not show much difference indicating that metal complex formation for the dye sources used in the present study did not help in improving the light fastness. Low light fastness is a common feature of many natural dyes as reported in the literature (Samanta and Agrawal, 2009).

From the commercial acceptability point of view the dyed or printed sample must have a minimum fastness grade of 3 and above. The wash and rubbing fastness results indicate that the dye sources used in the present study meet the wash and rubbing fastness requirement at 40^{0}C wash test with few exceptions. However, there is scope for improvement of light fastness.

3.3 The color strength (K/S)

The K/S values were measured both for samples dyed without and with mordants. The results are shown in Figure 4.

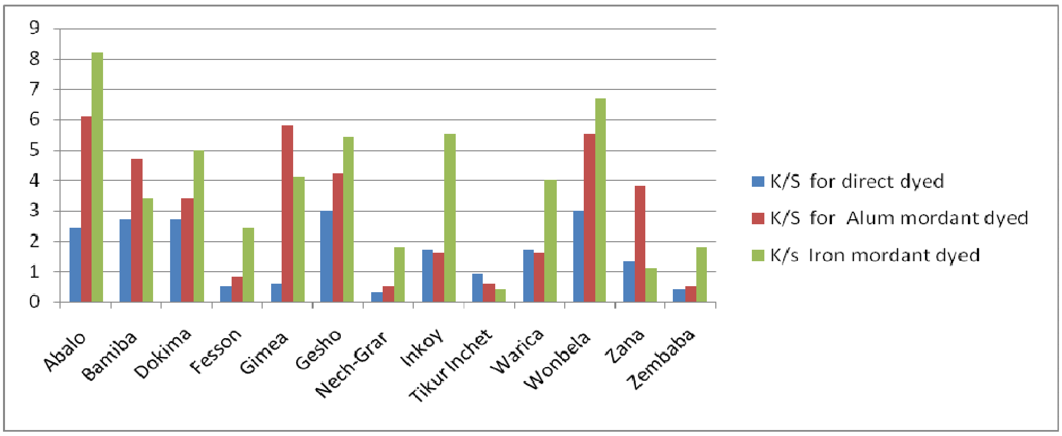

Fig 4: The K/S values of cotton fabric dyed with and without mordants.

In case of unmordanted cotton, the dye sources like Fesson, Gimea, Nech- Girar, Tikrur-inchet, and Zembaba showed only staining after dyeing which is reflected in low K/S values indicating that these dye sources do not have direct affinity to cotton. Other dye sources showed reasonably good dyeing as shown by higher K/S values reflecting in direct affinity to cotton. All the pre-mordanted samples showed higher K/S values compared to unmordanted dyed cotton. The increase in K/S values was higher in case of iron mordanted cotton. This kind of observations was expected because one of the purposes of mordanting is to bind the dye on cotton sample in the form of metal complex formation which results in increase dye uptake. This is attributed to the fact that the metal ions of mordants act as electron acceptors from electron donating groups of dye to form co-ordination bonds with the dye molecule, making them insoluble in water (Mongkholrattanasit *et al.*, 2011). Functional groups such as hydroxy, amino or carboxy on the fiber can occupy the unoccupied sites on metal ion interaction with the fiber. Thus, a ternary complex is formed by the metal ion of which one site is linked with the fiber and the other site is with the dye. This is depicted schematically in Figure 5.

Better color strength results are dependent on the metal salt used (Kamel *et al.*, 2009). Strong co-ordination tendency of Fe enhances the interaction between the fiber and the dye, resulting in high dye uptake (Jothi, 2008).

Thus the increase in K/S values after mordanting as shown in 5 was due to increase in dye uptake as well as deepening of the shade due to metal complex formation. The shade obtained after mordanting also depends on the nature of metal salt used for complex formation. The alum produced reddish brown shades whereas iron mordant produced grey shades.

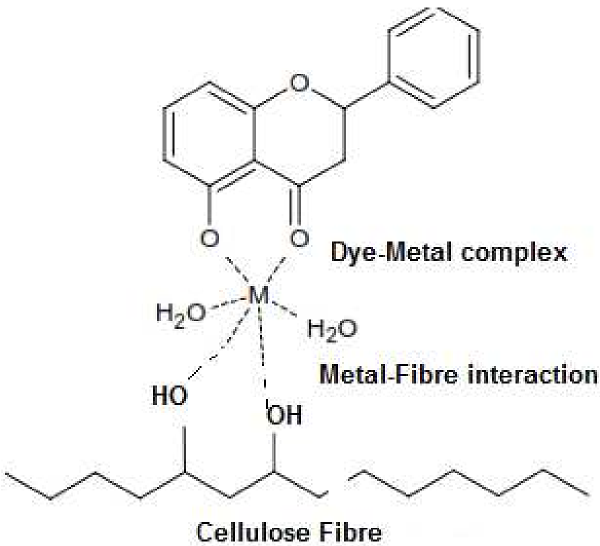

Figure 5 Schematic representation of Dye-Metal-Fibre interaction

3.4 The CIE L a* b* values of samples dyed without and with mordants*

The color of any object including dyed or printed textiles is a qualitative perception of human eye and varies from person to person. The Commission Internationale d'Eclairage (CIE) France developed a system for color quantification. This system is known as CIE system. The CIE system is based on the appearance of color to the theoretical standard observer. The color is expressed in terms of tristimulus values (X, Y, Z) derived from the relative amount of characteristics wavelengths of light absorbed by the color of the object. The X, Y, Z values can be determined with the help of colorimeter or spectrophotometer. X, Y, Z values are measured on 0-100 scale. The higher values indicate lightness and lower values darkness of color. In 1976 CIELAB system was introduced. The CIELAB coordinates L* (lightness), a* (red-green) and b* (blue-yellow) can be obtained by mathematical transformation of the X, Y and Z tristimulus values. The advanced spectrophotometers are equipped with software to calculate CIELAB values while the colored object is subjected to color measurement (X-rite,2013). In the present study an attempt is made to analyze the dyed samples in terms of CIELAB coordinates. In order to quantify the color of dyed samples, the CIE L* a* b* values were measured for samples dyed in the presence and absence of mordants. During the CIE L* a* b* measurement the sample dyed without mordant was considered as standard and sample dyed with mordants as matches. Each CIE L* a* b* value was measured three times and the average results are reported in Table 5

Table 5: The CIE L* a* b* values of cotton fabric samples dyed with and without mordants

Dye plant	DD				AMD				IMD			
	L*	a*	B*	C*	L*	a*	B*	C*	L*	a*	B*	C*
Abalo	79	3.0	21	22	69	7	21	42	42	1	6.2	6.4
Bamiba	54	6.6	12	12	49	8	16	18	54	4	10	11
Dokima	74	7.2	19	20	69	7	22	23	49	3	7.1	7.6
Fesson	86	4.7	13	14	78	4	16	17	50	3	7.5	8
Gimea	82	2.3	12	12	71	8	45	45	55	2	14.5	10
Gesho	70	3.2	16	16	64	3	27	27	55	3	18.7	19
Nech-grar	88	1.7	11	11	85	2	13	14	72	5	20.2	21
Inkoy	77	7.7	17	19	75	6	20	21	46	3	8.5	9
Tikur-inchet	76	8.6	14	17	69	13	14	19	57	4	6.3	8
Warica	76	8	20	22	73	7	23	24	56	5	17.3	18
Wonbela	77	4.3	22	22	73	2	31	40	46	1	7.1	7
Zana	86	3.2	12	12	70	2	28	28	77	2	12.5	13
Zembaba	84	4.2	8	9	81	6	15	16	72	3	7.7	8

The Lightness (L*) indicates the increase or decrease of the depth of shade. Lower values of L* means deepening of shade and higher values of L* indicates lower shade depth. Comparison of L* values indicated that in case of pre-mordant dyed samples there was deepening of shade. The decrease in L* value was more significant for iron (II) mordant dyed than alum mordant dyed samples, indicating greater color deepening with iron(II) mordant. The color deepening with mordant is attributed to the metal complex formation between dye and metal and it's binding with the fiber as explained previously..

The a* values indicate the redness of shade. Higher the value of a*, reveals more redness in color. The alum mordanted samples showed higher redness compared to iron mordanted samples due to change in shade towards red- brown compared to shade change towards grey in case of iron mordanted samples. No definite trend was observed in case of yellowness (b*) values.

The chroma(C*) value with alum mordanting is increased for all dye plants, whereas with iron (II) mordanting it is reduced for most dye plants except for three i.e. Zana, Gesho, and Nech –Grar, indicating the colors of the sample with alum mordanting were more pure compared to colors produced on cotton dyed without mordant and with iron (II) mordant.

4.0 Conclusion:

The wash fastness at 40^0 C and rub fastness for pre-mordant dyed samples were acceptable (≥3) whereas the wash fastness deteriorated when the wash temperature increased from 40^0 C to 60^0 C. This is acceptable commercially as only cold washing is recommended for natural dyed or printed fabrics**.** Light fastness for most dye plants was poor. Dyeing after mordanting; produced deeper shades compared to direct dyeing without mordant. The improvement in color strength with iron mordant was more significant than alum. The CIE L*a*b* values showed deepening of shade after mordant dyeing. Alum mordant showed redness and more pure shades compared to iron mordant. The overall performance of mordant dyed samples in terms of wash and rubbing fastness was good to a level of commercial acceptance, whereas, there was scope for further improvement of light fastness.

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