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Effect of Selected Mordants on Fastness Properties of Cotton Fabric Dyed with Henna Leaves Extract

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ABSTRACT
Henna extracts were obtained from the leaves of *Lawsonia inermis* using ethanol, distilled water and calcium hydroxide as solvents. The ethanol extract gave the highest percentage yield (5.6%), followed by the calcium hydroxide with (1.9%) yield. The ethanol extract was use to dye cotton fabric using copper (II) sulphate, iron (II) sulphate and crushed babool seeds as mordants. The cotton fabric dyed in the absence of the mordants show poor fastness to washing and fair fastness to rubbing and wet pressing with very good fastness to dry and dump pressing, but While the fabric dyed using mordants show very good fastness to washing, dump and wet pressing, and an excellent fastness to rubbing and dry-pressing.

Keywords: Cotton fabric, Colour fastness, Henna Extracts, Mordants

INTRODUCTION
Dyes are coloured compounds derived from organic sources that are used to impart colour onto materials such as paper, leather, fur, hair, drugs, cosmetics. The word 'natural dye' covers all the dyes derived from the natural sources such as plants, animal and minerals (Sunata. & Vailaren, 1998). Natural dyes are known for their use in colouring of food substrates, leather, wood as well as natural fibres such as wool, silk, cotton and flax since ancient times (Saravanan *et al.,* 2013). However, nowadays the use of natural dyes has declined due to the availability of synthetic dyes. Although these synthetic dyes have moderate to excellent colour fastness properties, their environmental and health effects make them a source of concern. Furthermore, the poor fastness to wet treatment (moderate wash fastness and light fastness) of natural dyes has rendered their application quite ineffective (Samanta & Agarwal, 2009). Interestingly however, the use of appropriate mordant had been found to improve the colour fastness of natural dyes, (Gumel and Ali, 2012).

Henna (*Lawsonia inermis*) is widely used as a dyestuff in the tropics and subtropics for beautification (in the staining of hair, nails and beard) (Alarm *el al.,* 2007). Also the leaves of henna have been extensively used for dyeing silk and wool. Thus, a wide range of colours have been successfully imparted on fabrics in an acid bath containing potassium di-chromate, iron sulphate, tin chloride or alum. Specifically cotton hanks were found to be cement-grey coloured when treated with henna extract in the presence of acidified ferrous sulphate solution. (Rehsi & Daruvala, 1997).

A number of previous studies have provided information on the chemical constituents and applicability of henna. For instance, Alam et *al*., (2007) reported the extraction of Lawsone by adding lead-acetate into the plant extract and subsequent removal of the lead acetate in the presence of hydrogen sulphide (H_2S). The desired lawsone was then extracted using benzene as a solvent. Another research group isolated Lawsone (2-hydroxy-1,4 naphthoquinone) from the Indian henna. The structure was established by direct comparison with the synthetically prepared compound and its derivatives (Lluvia *et al.*, 2014). Bhattacharya *et al.,* (2004) studied the effect of some metal sulphates as mordants and found that the depth of dyeing can be improved by using different metal salts.

Das *et al.,* (2006) observed that that the employing ferrous sulphate and aluminum sulphate as mordants improve the dye uptake, light fastness and colour retention on repeated washing.

Another research group reported that different types of mordants and methods of mordanting significantly affect the rate of photo-fading. The use of copper or iron sulphate produced a high resistant to fade material, whereas tin chloride or alum does not (Gupta *et al.,* 2004). On the other hand, they observed that fastness to light was improved when post-mordanting treatment was conducted using copper or iron sulphate, but pre-mordanting was found to be more effective when the same mordants were employed (Gupta *et al.,* 2004).

In this study, the influence of different mordants in the dyeing of cotton fabric using henna leaves extract was investigated.

MATERIALS AND METHODS

Materials

Henna leaves were picked from Sharada Gidan Kwari plantation site. Cotton fabric (100%) was purchased from Kwari market, Kano. *Acacia nilotica* (Babool) seeds were obtained from the botanic garden of the Yusuf Maitama Sule University.

Reagents and Equipments

Tin chloride and iron sulphate were purchased from Sigma-Aldrich (St. Louis, USA). Sodium hydroxide, sodium hypochlorite and calcium hydroxide (analytical grades) were obtained from AcrÖs (New Jersey, USA). Iron (II) sulphate, copper (II) sulphate and sodium carbonate were purchased from Sigma-Aldrich (St. Louis, USA). Acetic acid, n-hexane, acetone and ethanol were purchased from Sigma-Aldrich (St. Louis, USA). Liquid detergent (Morning fresh) was purchased from market. All reagents and solvents were of analytical grade, and were used as received without further purification. IR spectra were recorded on a Perkin Elmer (Spectrum™ 400 FTIR Spectrometer, USA). UV Visible spectrophotometer (LAMB DA 35) (Massachusetts, USA), was used to determine the dye concentration in the aqueous medium.

Extraction of Henna Crude Extract

Three different media were used for the extraction. Thus, the first method, 100g of the powdered henna leaves were mixed with 250 mL distilled water, while and 250 mL of 1.5 M $Ca(OH)_2$ was used for the second method. The third method employed 500 mL ethanol added to 200g of the henna Leaves. the mixtures were separately stirred and allowed to stand for a week at room temperature. The resulting solutions were filtered, and the filtrate was subjected to freeze-drying to obtain the concentrated extracts (Sunita *et al*., 1998).

PRE-TREATMENT OF COTTON FABRIC

Desizing (Rot Steeping)

Four (4) cotton fabrics were cut in the dimensions of 8.5cm by 5.cm, weighing l.0 g each and were impregnated in water only and allowed to stand for 36 hours at room temperature. This process removes the added materials (such as starch, carboxymethyl cellulose) applied to yarn before weaving into fabric (Charles, 1992; Shamshad *et al.,* 2015).

Scouring

The desized cotton fabric was boiled in a bath containing 2% caustic soda for 2 hours. The scoured cotton fabric was then thoroughly washed with tap water and dried at room temperature (Charles, 1992; Shamshad *et al.,* 2015).

Bleaching

The cotton fabrics soaked into a bath containing l0 mL sodium hypochlorite, 90 mL distilled water and 0.5 g sodium carbonate for 15 minutes. The process was carried out at room temperature and the bleached materials were then treated with 0.1% acetic acid to neutralize the alkali on the fabrics followed by washing, rinsing and drying (Charles, 1992; Shamshad *et al.*, 2015).

Mercerization

The cotton fabrics were treated with 25% caustic soda for 1 minute at room temperature. The materials were then thoroughly washed with distilled water, rinsed and dried (Charles, 1992; Shamshad *et al.,* 2015).

Dyeing of Cotton Fabric without Mordant

1.0 g of dye extract was dissolved in a distilled water, made up to 60 mL of the dye bath, and the temperature raised to 40°C. Then 1g of pre-treated cotton fabric was immersed into the warm dye solutions at 80°C for 1 hour. The sample was finally rinsed and dried (Vankar *el al.,*2007).

Dyeing of Cotton Fabric using Babool Seeds as Mordant

Mordanting using babool seeds was carried out by soaking the fabric in the hot aqueous solution of the seeds. The aqueous solution was prepared by dissolving 1.5 g of the crushed and sieved babool seeds in distilled water (60 mL) and boiled for 90 minutes. Thus, after cooling of the aqueous solution containing the fabric, the material was removed, rinsed with distilled water and air-dried.

1.0 g of mordanted fabric was introduced into the dyeing solution (60 mL) at room temperature and the temperature was slowly raised to 80°C. The dyeing was maintained at this temperature for 60 minutes. After cooling of the solution, the fabric was removed, rinsed with distilled water and air-dried (Vankar *el al.,*2007).

Dyeing of Cotton Fabric using $CuSO_4$ as Mordant

0.05 g of $CuSO_4$ was dissolved in distilled water. 1 g of cotton fabric was immersed into the bath at 60°C. The temperature was raised to 90°C and held for 45 min. The fabric was removed from the mordant solution, rinsed with water and allowed to air dried (Iqbal *et al.,* 2008). The mordanted fabric was then dyed as above.

Dyeing of Cotton Fabric using $FeSO_4$ as Mordant

The mordanting and dyeing procedure, as in $CuSO_4$, was adopted, but using 0.05 g $FeSO_4$ as mordant (Iqbal *et al.,* 2008).

After-Treatment

The dyed fabrics were washed with soap to remove the unattached dye to the fibre. The washed fabrics were rinsed with water and air-dried.

COLOUR FASTNESS TESTS

The following tests were performed to determine the fastness of the dyed cotton fabric in this study.

Fastness to Wet Treatment

The wash fastness rating was assessed using grey scale as per ISO-105-A02 (loss of colour shade/depth) and ISO-105-A03 (extent of staining) as adopted by Samanta *et al.*, (2009). 0.5 g of dyed material was placed between two pieces of undyed white cotton fabrics. These pieces were held together by stitching around their edges. 5 mL of detergent solution and 2 g of anhydrous sodium carbonate were added to the volumetric flask and made to 1L with distilled water. The solution was heated at 60°C on water bath for 30 minutes. The sample was then removed, rinsed with distilled water and air-dried before assessment. The colour change of the dyed material and the degree of staining of the undyed white material were assessed using the grey scale.

Fastness to Rubbing

The dyed fabric in contact with undyed fabric were pinned together. The two were rubbed several times for 1 minute. The change in colour and degree of staining of the undyed fabric was assessed using the grey scale (Samanta *et al.*, 2009).

RESULTS AND DISCUSSION

Extraction of Henna Crude Extract

Ethanol was found to furnish the highest percentage yield of the dye extract (5.6%) compared to that of water and calcium hydroxide with percentage yields of 2.6% and 1.9% respectively. The lowest value obtained for calcium hydroxide could be attributable to the fact that $Ca(OH)_2$ dissolves the colouring matter slightly (Alam et *al*., 2007).

Table 1: Percentage Yield of Henna Extracts

Solvent	Yield (%)
Water	2.16
$Ca(OH)_2$	1.90
Ethanol	5.14

FTIR Spectroscopy of Henna Crude Extract

The characteristic absorption frequencies obtained from the IR analysis (Figure 1) indicated the presence of lawsone bands, with intensities between 3150-3000 cm^{-1} (=C-H aromatic overlapped by OH broad band), the band at 1737 cm^{-1} is assignable to C=O and 3600-3200 cm^{-1} is associated with O-H stretching, while 1639 cm^{-1} are for C=C conjugated. This is also in accordance with the observations reported by Alam *et al*., (2007).

The colouring matter isolated from henna leaves has substantivity to cotton and the use of mordants was found to produce varying shades. This could be due to the ability of iron sulphate and copper sulphate to form fibre-dye-metal complexes, resulting to different colours, hence, improved the fastness properties of the dyed fabrics towards washing, rubbing and pressing (Gumel & Ali, 2012).

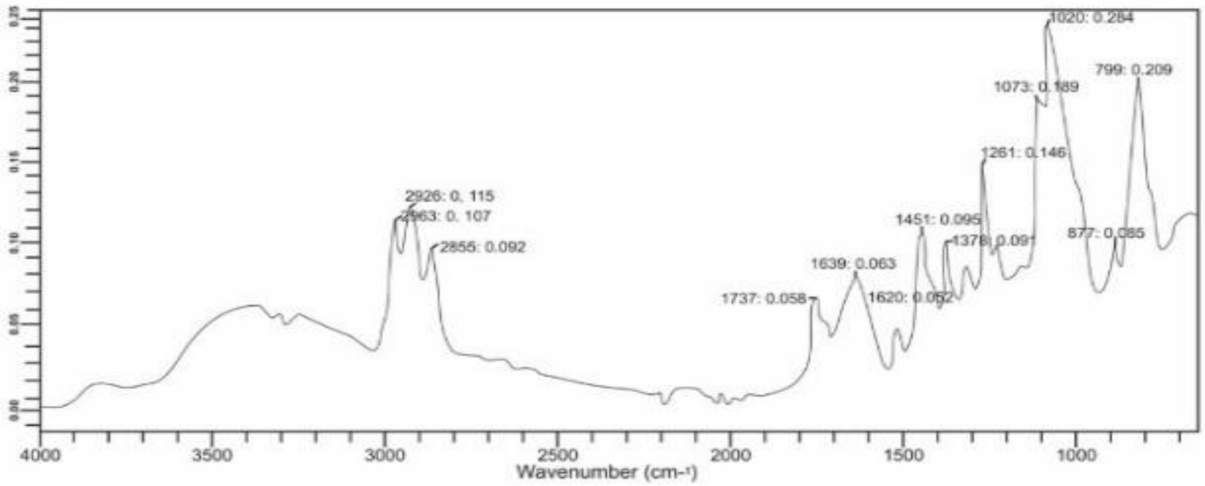

Figure 1: FTIR Spectrum of the Henna Ethanol Extract.

Table 2: Colours of the Dyed Fabrics using Different Mordants

Dyed Samples	Colour
A	Dark Green
B	Light Brown
C	Greenish Brown
D	Dark Brown

A = sample dyed without mordant, B= sample dyed with babool, C = sample dyed with copper (II) sulphate and D = sample dyed with iron (II) sulphate.

Effect of Babool as Mordant

The unmordanted dyed fabric showed poor fastness to washing and moderate fastness to rubbing and wet treatment but has a very good fastness to dry and dump pressing. While the mordanted dyed fabric subjected to washing, rubbing and pressing (dry, dump and wet) showed very good fastness to washing, dump and wet treatment, with excellent fastness to rubbing and dry pressing (Table 4).

Effect of $FeSO_4$ as Mordant

$FeSO_4$ mordant gave a slight improvement in light fastness for a simultaneous mordanting method and compared to that obtained without a mordant. The pre-mordanting and post-mordanting methods registered no change in fastness against that without mordant application. However, post-mordanting method had more staining for washing and for wet rubbing as a result which is inferior to all other mordanting method and the control without mordant.

Effect of $CuSO_4$ as Mordant

$CuSO_4$ mordant with pre-mordanting yielded improved light fastness compared to the one without using mordant. Simultaneous mordanting and post mordanting show no improvement on the dyeing of the cotton fabric. Post mordanting provided more staining for washing and wet rubbing. The pre and simultaneous mordanting methods had very faint staining.

FASTNESS PROPERTIES

Fastness to Wet Treatment

When a piece of dry white cloth was placed under a dyed cotton fabric (without mordant) and another dried white cloth was placed on top of the wet specimen (dyed fabric) and pressed for 20 seconds It was observed that there was change in colour (fading) of the dyed material and high degree of staining of the undyed material, indicating that the dye has poor fastness to wet treatment. But when mordants were used, there were increase in fastness to wet treatment, with specimen C having the highest fastness properties followed by C and D as shown in Table 4. This improvement in the fastness to wet treatment could be due to the ability of the mordant to form complex thereby fixing the dye onto the fabric (Samanta *et al.*, 2009).

Fastness to Rubbing

This test is designed to determine the degree of color which may be transferred from the surface of a dyed fabric to another test cloth (adjacent fabric) due to rubbing. The rubbing fastness is conducted using 'crock meter', and it works on the principle of abrasion; dry and wet, where crock meter creates the rubbing motion to stimulate the action of a human finger and forearm.

In the case of dry rubbing, the margin of the dyed fabric was observed to fade as a result of the loosely or unfixed dye molecules which are removed and adhered to the surface of the next fabrics adjacent to the dyed fabric (rubbing cloth).

In the wet rubbing, the fabrics dyed using mordants show excellent fastness to rubbing (crocking). This could be due to the ability of the mordants to form complexes with the dye which would enable its fixation onto the fabrics. But for the fabric dyed without using mordant, it was observed that the dyed fabric has very poor fastness to rubbing as the unfixed dye dissolved in water and then transferred to the fabric adjacent to the dyed fabric. This leads to poor wet rubbing fastness as shown in Table 4 (Samanta *et al.*,2009).

Table 4: Assessment of Fastness Properties of the Dyed Fabric

Dyed Sample	Fastness to et treatment	Fastness to rubbing
	Grade Fastness	
A	2	0
B	4	5
C	5	5
D	4	5

Where A represents sample dyed without mordant, B represents sample dyed with babool, C represents sample dyed with copper sulphate and D represents sample dyed with iron sulphate.

CONCLUSION

In this study, dye from Henna leaves was successfully extracted, and used to dye cotton fabric using different mordants. It was observed that cotton fabric dyed without mordant exhibited poor fastness to washing as expected, but showed moderate fastness to wet treatment, pressing and an excellent fastness to dry and damp pressing while the cotton fabric treated with mordants before dyeing showed very good fastness to washing as well as to damp and wet treatment with excellent fastness to rubbing and dry pressing (heat). The variation in hue and fastness properties of the pre-mordanted dyed fabric with babool seeds may be due to the presence of tanning acting as the mordant possessing phenolic hydroxyl group, which forms a complex with the dye molecules and leads to higher fixation of the dye on the fabric. While the colour change and enhanced fastness property of the fabric pre-mordanted with the synthetic mordants is possibly because iron sulphate and copper sulphate have the ability of forming metal-dye complexes.

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