

Effect of Temperature and Mordant on the Dyeing of Cotton Using Sodium Hydroxide Extract of *Whitfieldia lateritia* Dye

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ABSTRACT: The textile industry has intensified research to explore natural sources of dyes as opposed to synthetic dyes. As a representative effort in this direction, this paper describes the process of dyeing cotton with alkali extract of *Whitfieldia lateritia* plant. This plant is a green plant with several applications. Evaluations were made on the basis of the dyed fabric, different dyeing temperature, three mordants and different mordanting methods. Fastness properties of the dyed fabrics were tested. From the study, it was observed that the maximum dye exhaustion took place at B₅ (70°C). It was also observed that the colour strength increased with an increase in dyeing temperature and reached a maximum at 70°C before it then started to decrease with further increase in temperature. The fastness properties of the dyed fabrics increased with increase in temperature of dyeing. Of the three mordanting methods studied, post-mordanting method on the average, gave the most superior fastness property. In all cases however, the best results on wash, perspiration and light fastness were achieved using ferrous sulphate as mordant in the simultaneous mordanting method.

KEYWORDS: Natural dyeing, *Whitfieldia lateritia*, temperature, mordanting, colour fastness

I. INTRODUCTION

Plants are the major sources of natural dyes. Dyes are extracted from different parts of these plants such as roots, barks, stems, leaves and even flowers, for colouring textile fabrics. Some of these plants are also used for medicinal purposes for the treatment of different ailments. *Whitfieldia lateritia*, despite its numerous traditional applications such as; its wide use in folkloric medicine of Africa and Asia for the treatment of ailments such as inflammation, anemia, liver damage and boosting of blood [1], is one of the numerous plants that are under studied especially in the area of dye extraction. It belongs to the family of Acanthaceae which contains ten (10) species [2]. *Whitfieldia lateritia* is an evergreen plant with well-developed leaves which are arranged alternately along the stem [2]. It is native to Sierra

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Leone but recently has been confirmed to exist in several parts of Nigeria in places such as; Ivo, Ikwo and Izzi Local Government Areas of Ebonyi State; Owerri West Local Government Area of Imo State; and Isiala-Ngwa North and South Local Government Areas of Abia State [3]. The proximate and mineral compositions of *Whitfieldia lateritia* leaves have been evaluated using standard analytical protocols [2]. Reports on the qualitative and phytochemical analysis revealed the presence of flavonoids, alkaloids, saponins, cardiac glycosides and tannins in the leaves of *Whitfieldia lateritia*[1]. The UV-Vis and FTIR spectroscopic analysis carried out on the sodium hydroxide extract of *Whitfieldia lateritia* has also revealed the presence of flavonoids, polyphenols and tannins [3].

In the textile dyeing industry, excessive amount of synthetic dyes are used to meet the required global demands in colouration of textile goods due to their low cost, improved colour fastness and wider varieties of bright shades when compared with natural dyes. Synthetic dyes are produced from petrochemicals and so, some of them contain carcinogenic amines and their application causes serious health and environmental hazards, causing imbalance in the eco-system. In this regard, there is an increased demand in the greener alternatives or agricultural wastes. Natural dyes therefore, are among the viable options for developing a greener textile dyeing process. Plant leaves are potential sources of natural dyes because of their abundant nature and easy availability [4]. To overcome the problem of poor fastness and limited colour varieties associated with natural dyes, various metallic salts are being used as mordant to improve fastness and produce different shades with the same dye. Natural dye extraction process plays a vital role in the properties of the dyes obtained as they are affected by many factors such as time, temperature, pH and solvent used for the extraction. Hence, for proper technical extraction and application of natural dyes, efficient method of extraction has to be employed.

II. MATERIALS AND METHOD

Materials Used

Sodium hydroxide (NaOH) pellets, sodium chloride (NaCl), distilled water, potassium aluminium sulphate, tannic acid, potassium dichromate, ferrous sulphate, disodium hydrogen orthophosphate dehydrate, histidine monohydrochloride monohydrate, sodium dihydrogen orthophosphate dehydrate. Plain weave desized and bleached cotton fabric (60 ± 1 warp and 48 ± 1 weft per inch), white polyester fabric (55 ± 1 warp and 55 ± 1 weft per inch), *Whitfieldia lateritia* plant leaves were obtained from Ihiagwa, Imo State, Nigeria and identified by Mrs. M.N Osuagwu of the Department of Plant Science and Biotechnology, Michael Okpara University of Agriculture Umudike, Abia State, Nigeria. Thermostatic water cabinet Model: HH size 64x35x3 1cm, Cary 300 UV-Visible Spectrophotometer, Cary 630 FTIR machine.

Methodology

Preparation of samples

The *Whitfieldia lateritia* plant leaves were thoroughly washed and dried at room temperature until crisp. They were ground and then sieved to obtain $53 \mu\text{m}$ particle size. The required weight of the *Whitfieldia lateritia* powder for each test was weighed out using the Setra BL-410S electronic weighing balance.

Experimental

i. Study of the Effect of Temperature

Extraction of dye and dyeing of cotton fabric

The extraction was done at a MLR of 1:50 (g:ml) using 1% alkali solution at 70°C for 60 minutes in a thermostatic water bath. The hot solution was filtered and a clear solution which was used for dyeing cotton fabric was obtained.

The *Whitfieldia lateritia* dye extract was used to directly dye seven samples of cotton fabric each weighing 1g, to a 3% shade with MLR of 1:40 for 60 minutes at different temperatures viz; room temperature ($27 \pm 2^\circ\text{C}$), 40°C , 50°C , 60°C , 70°C , 80°C and 90°C . Dyeing was done in the presence of 30% (owf) of pure sodium chloride (NaCl) to promote exhaustion. This was done so as to be able to increase the rate of migration of dye molecules into the fabric at any slight change in temperature since increase in temperature favors the rate of absorption

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ii. Study of the Effect of Mordants and Mordanting Methods

Extraction of Dye and Preparation for Dyeing

The dye extract used for this study was extracted using a MLR of 1:25 at 70°C for 60minutes using a thermostatic water bath. Fabric preparation for dyeing with mordant was achieved by using a MLR of 1:40 for 40minutes (for mordanting), MLR of 1:40 for 60minutes (for dyeing), 30% (0.3g) of mordant and 30% (0.3g) of NaCl salt on the weight (1g) of the fabric (owf).

Dyeing with Mordants

For this study, three mordants (mixture of alum and tannic acid {in a 50:50 proportion}, ferrous sulphate and potassium dichromate) were used to treat cotton fabric. Pre-mordanting, Simultaneous mordanting and Post-mordanting methods were used.

i. Pre-mordanting

In pre-mordanting method, the fabrics were first immersed in an aqueous solution of the mordants- a mixture of alum and tannic acid in a 50:50 proportion, ferrous sulphate and potassium dichromate, in separate baths and mordanted for 40minutes at 70°C, followed by dyeing of the mordanted fabrics at 70°C for 60minutes using a MLR of 1:40 in the presence of NaCl salt. The dye bath was stirred at every 5minutes interval of dyeing. The fabrics were removed after dyeing and rinsed in cold distilled water before drying under shade.

ii. Simultaneous mordanting

In simultaneous mordanting method, the fabrics were immersed in dye baths containing the mordants, dye extract and NaCl salt. Dyeing took place at 70°C for 60minutes using a MLR of 1:40. The dye bath was stirred at every 5minutes interval of dyeing. The fabrics were removed after dyeing and rinsed in cold distilled water before drying under shade.

iii. Post-mordanting

In the post method, the fabrics were immersed in dye baths containing the dye extract and dyeing was carried out in the absence of a mordant at 70°C for 60minutes using a MLR of 1:40 in the presence of NaCl salt, followed by mordanting in separate baths containing the mordants. Mordanting was carried out for 40minutes using a MLR of 1:40 at 70°C. The fabrics were removed after dyeing and rinsed in cold distilled water before drying under shade.

iii. Colour Fastness Testing

Colour Fastness to Daylight

The resistance of the dyed textile fabrics to fading when exposed to daylight was ascertained through the colour fastness to daylight. In this regard, a small piece of the dyed cotton fabrics of mass 0.25g measuring about 3x3cm were cut and mounted on pattern cards (Blue Wool Scale). The test samples were then exposed to daylight for 72 hours. After 72 hours, the samples were removed and compared with the original unexposed samples. The changes in colour were then assessed using a Blue Wool Scale. The results of the test were then recorded.

Colour Fastness to Washing

The test was carried out for 30 minutes at a temperature of 60°C without any stainless steel balls using material to liquor ratio of 50:1 (ml:g) while following the procedure in [3].

After washing, rinsing and drying the grey scales for colour change were used to check for colour change in the washed fabric in relation to the unwashed fabric while the grey scales for staining were used to check the level of staining of the adjacent fabric.

Colour Fastness to Perspiration

The resistance of the colour of textile fabrics to perspiration was tested using the procedure in [3].

After the test, the colour change and the staining of the adjacent white cloth were assessed using a grey scale and the results obtained were carefully recorded.

III. RESULTS AND DISCUSSION

Effect of Temperature on dyeing of cotton fabric

Percentage (%) Dye Exhaustion and Colour Fastness

The results of the test on % exhaustion and colour fastness are presented in Table 1 and 2 respectively. The results showed that temperature has a great effect on the dyeing of cotton fabric with a direct natural dye. From the Table it is

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observed that the absorbance of the extract after dyeing initially decreased from B₁ to B₂ before increasing at B₃. The increase at B₃ could be due to the fact that impurities yielded more dyes thus making it difficult to have a steady percent dye uptake. Also, the decrease in absorbance with increase in temperature resulted to increase in the depth of the colour shade thus indicating that there is a reduction in concentration after dyeing which in turn shows that there was migration and absorption of the dye molecules into the cotton fabric. The further decrease from B₄ to B₅ and increase from B₆ to B₇ can be attributed to either the saturation of the available sites present in the fabric for bonding between the molecules of the fabric and that of the dye or that further increase in temperature caused a reverse movement of the dye molecules from the fabric into the dye bath [5]. Furthermore, diffusion of dye molecule in the fibre determines the rate of dyeing, therefore, following Fick's first law, diffusion coefficient D which is between 10⁻¹⁰ to 10⁻¹² cm²/s can only be increased by increasing temperature [6]. It then means that more dye molecules diffused into the cellulose fibre at increased temperature hence making the fabric absorb more dye molecules and have deeper colour shade than at room temperature [5].

From Table 1, it is clear that the maximum dye exhaustion took place at B₅ (70°C). It was also observed that the colour strength increased with an increase in dyeing temperature and reached a maximum at 70°C before it then started to decrease. This increase in dye uptake can be attributed to better dye exhaustion at higher temperatures. However, temperatures higher than 70°C resulted in decrease in colour strength which may be attributed to decrease in stability of dye molecules at higher temperatures. This result is in agreement with that obtained by Kannanmarikani et al [7].

Table 1: Percent Exhaustion of dyed samples at different temperatures

Sample Code	Dyeing Temperature, °C	Absorbance Before Dyeing	Absorbance After Dyeing	% Dye Exhaustion
B ₁	27± 2	0.55	0.52	5.45
B ₂	40	0.55	0.47	14.54
B ₃	50	0.55	0.48	12.72
B ₄	60	0.55	0.43	21.81
B ₅	70	0.55	0.32	41.81
B ₆	80	0.55	0.50	9.09
B ₇	90	0.55	0.51	7.27

The effect of temperature on the colour fastness properties of the dyed fabrics were also visible as the colour shade on the fabric increased with increase in temperature of dyeing B₁---B₇ as shown in Fig3 below. The deeper colour shade of fabrics B₅ and B₆ is an indication of having more dye molecules in the cellulose fibre hence these fabrics lost more dye molecules during washing leading to their lower colour change on the grey scale rating. This conforms to the statement "that for direct dyes wet fastness decreases as colour depth increases" [6]. The fastness however decreased slightly at B₇ as envisaged due to the poor interaction between the dye and fabric at high temperature.

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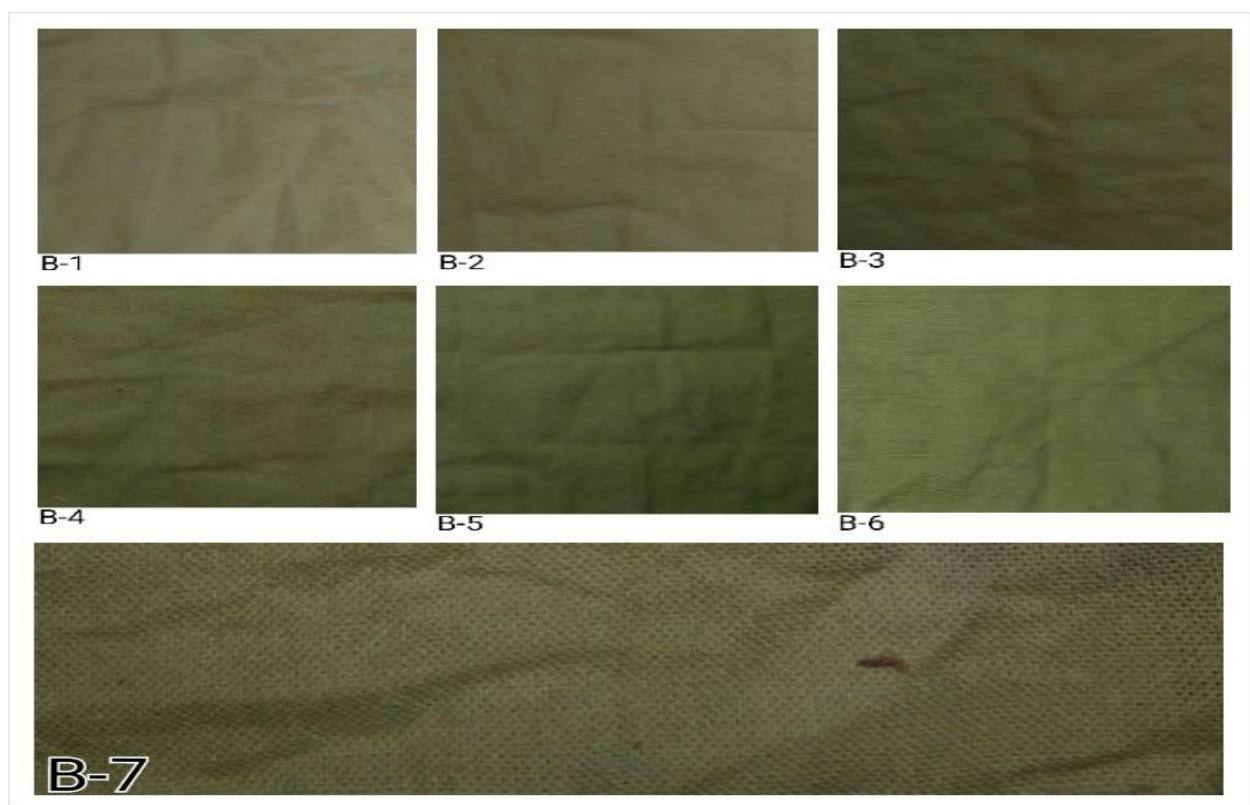


Fig 1: Cotton fabrics dyed with *Whitfieldia lateritia* dye at different temperatures.

Table 2: Results of colour fastness to washing, perspiration, and light of dyed samples at different temperatures

Sample Code	Dyeing Temperature, °C	Wash Fastness		Perspiration Fastness	Light Fastness
		Colour change	Staining		
B ₁	27± 2	2	5	3	4
B ₂	40	2	5	3	4
B ₃	50	2	5	3	4
B ₄	60	3	5	4	4
B ₅	70	4	5	3	4
B ₆	80	4	5	3	3
B ₇	90	3/4	5	3	4

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From Table 3, the fastness properties of the dyed fabrics increased with increase in temperature of dyeing. Samples B₅ and B₆ recorded very good (4) fastness results to colour change. Fabrics B₁, B₂ and B₃ however performed poorly in colour change assessment with ratings of 2. This can be attributed to low temperature of dyeing which was not enough to cause dye molecules to migrate into the cellulose pore and become adsorbed onto it. The excellent grey scale to staining ratings of 5 for the fabrics dyed with *Whitfieldia lateritia* dye is an indication that the adjacent fabrics were not stained. It then implies that *Whitfieldia lateritia* natural dye will not stain adjacent cotton fabric washed with them.

The fastness to perspiration of the dye studied showed good to very good result on the grey scale for colour change with fabrics B₁, B₂, B₃, B₅, B₆ and B₇ having 3 while B₄ recorded 4 on the scale rating.

Fabrics B₁, B₂, B₃, B₄, B₅, and B₇ had light fastness rating of 4 on the blue wool scale while fabric B₆ had rating of 3 on the blue wool scale. The reason for this result could be because of lack of metallic sites which will form good complexes with the fabric thereby protecting the chromophore from photolytic degradation [8].

Effect of Mordants and Mordanting Methods on Dyed Cotton Fabrics

Colour Fastness Properties

In Fig. 3, the effect of mordants and mordanting methods on cotton fabrics dyed with *Whitfieldia lateritia* dye extract can be seen clearly. The fabric shade shifted with a change in the method of mordant application for all the mordants used.

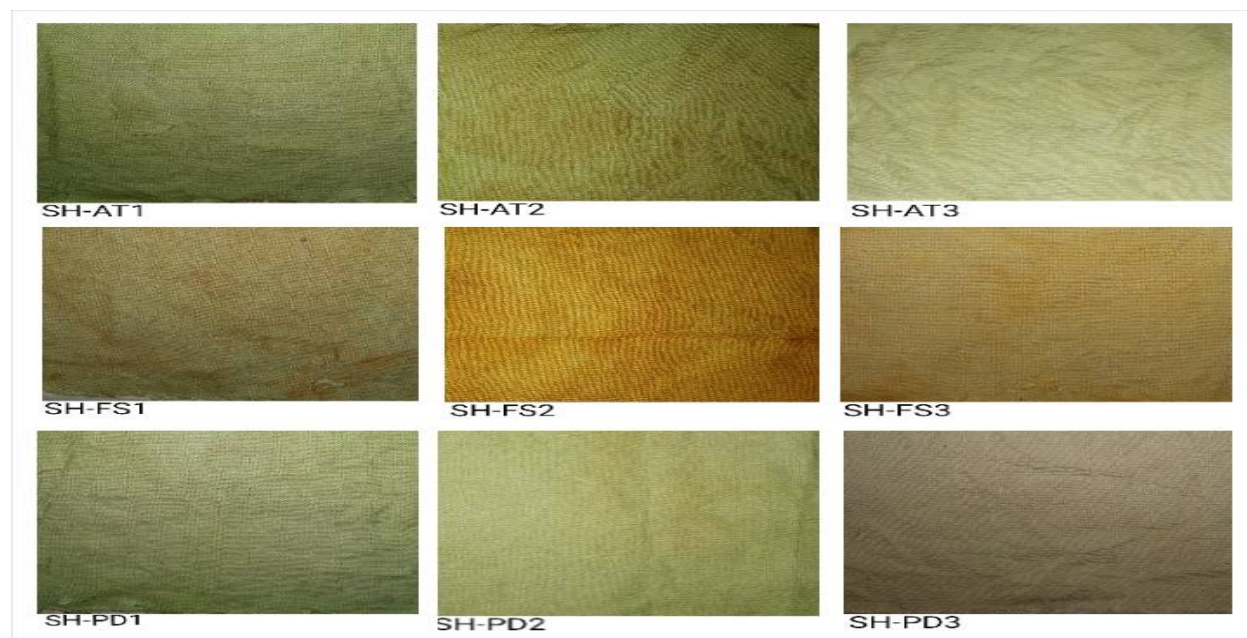


Fig. 2: Cotton fabrics dyed with *Whitfieldia lateritia* dye using different mordants and mordanting methods. Where: SH-AT designates dyed cotton fabrics mordanted with a mixture of alum and tannic acid. SH-PD designates dyed cotton fabrics mordanted with potassium dichromate. SH-FS designates dyed cotton fabrics mordanted with ferrous sulphate. The subscripts “1, 2 and 3” designates “pre-, simultaneous and post” mordanting methods respectively as used in this study.

Colour fastness to washing, as can be seen in Table 3 for all fabrics, did not show an all-round improved performance on the grey scale for colour change. Fabrics SH-AT₂, SH-PD₁ and SH-PD₂ had the least grade (2) while fabric SH-AT₁ had (3) on the grey scale rating, implying very noticeable colour change. Fabrics SH-FS₁ (3/4), SH-PD₃ and SH-FS₃ had ratings of (4), while fabrics SH-FS₂ and SH-AT₃ had rating of 4/5 implying very slight colour change. Colour staining assessment for all the fabrics at the three areas checked gave grades ranging from 3 to 5 for different fabrics.

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Fastness to perspiration gave a very good result as the least grade was 3/4 for fabrics SH-AT₂, SH-PD₁ and SH-FS₁. Fabrics SH-AT₁, SH-AT₃ and SH-FS₃ gave 4 on the grey scale. However, fabrics SH-PD₃, SH-FS₂ and SH-PD₂ were rated 4/5.

The light fastness for all the fabrics at the three areas checked showed poor, (3) for SH-AT₂ to excellent (7) for SH-FS₂, ratings on the blue wool scale. The rest are SH-AT₁ and SH-PD₂ (4); SH-PD₁, SH-PD₃ and SH-FS₁ (5) and SH-FS₃ and SH-AT₃ having the second best grade.

Table 3: Colour fastness properties of cotton fabrics dyed with sodium hydroxide (SH) extract of *Whitfieldia lateritia* dye using different mordants and mordanting methods

Sample code	Wash Fastness		Perspiration Fastness	Light Fastness
	Colour change	Staining		
SH-AT ₁	3	3	4	4
SH-AT ₂	2	3	3/4	3
SH-AT ₃	4/5	4	4	6
SH-PD ₁	2	4	3/4	5
SH-PD ₂	2	4	4/5	4
SH-PD ₃	4	4	4/5	5
SH-FS ₁	3/4	4	3/4	5
SH-FS ₂	4/5	5	4/5	7
SH-FS ₃	4	4/5	4	6

IV. CONCLUSION

Sodium hydroxide extract of *Whitfieldia lateritia* was used to dye cotton fabrics at different temperatures. It was observed that the dye exhaustion and colour strength increased with an increase in dyeing temperature and reached a maximum at B₅(70°C). Of all the fabrics dyed, fabrics B₅ and B₆ recorded the best wash, perspiration and light fastness property. Also, alkali extract of *Whitfieldia lateritia* yielded various colours on cotton fabric with the application of different mordants. Of the three mordanting methods studied, post-mordanting method on the average, gave the most superior fastness property. In all cases however, the best results on wash, perspiration and light fastness was achieved using ferrous sulphate as mordant in the simultaneous mordanting method.

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