

Study on the Application and Colour Fastness Properties of Aqueous Dye Extract from *Whitfieldia Lateritia* Leaves

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Abstract

The application and colour fastness properties of aqueous dye extract from *Whitfieldia lateritia* leaves were studied. The UV-Vis and FT-IR spectra of the dye extracts showed the presence of polyphenols such as flavonoids and tannins. The extract was used to dye cotton fabrics using different mordants viz- alum/tannic acid, ferrous sulphate and potassium dichromate. Pre-mordanting, simultaneous mordanting and post-mordanting methods were also used to dye the cotton fabrics in the presence of sodium chloride as an electrolyte. The cotton fabrics showed a total or slight change in colours with different mordants and mordanting methods. The dyed fabrics were evaluated for Colour fastness to Washing, perspiration and light. The general assessment of the colour fastness tests showed that the aqueous extract of *Whitfieldia lateritia* dye applied under the influence of mordants irrespective of the method of application used showed good ratings.

Keywords: *Whitfieldia lateritia* • Natural dye • Cotton fabric • Mordants • Colour fastness

Introduction

The application of dyes gotten from natural sources for the dyeing of textile goods has become a possible choice of interest. The selection of local species as a natural dye can make its use practicable, thereby enhancing local biodiversity. Before the development of synthetic dyes, natural dyes were used to colour textile goods. The discovery of synthetic dyes solved the problem of poor colour fastness, lack of variety of colour shades and inadequate technical knowhow on the extraction and application techniques experienced with the use of natural dyes [1]. Synthetic dyes are inexpensive, available in a variety of colours and have excellent fastness properties. However, they are reported to have allergic, toxic and even carcinogenic properties, which are harmful to humans and the environment [2]. Environmental interest and consciousness have, therefore, favoured the development of sustainable products and processes that are less injurious to man and his environment at large [3]. Natural dyes are found to be less toxic than synthetic dyes and generate waste effluents that can be treated via biodegradation. Thus, researchers are focusing their interest to this family of colourants as seemingly alternatives to certain synthetic dyes, especially those that pose a threat to human health and the environment [4,5]. Understanding the potential of vegetable-based materials for natural dye extraction is significant in enabling their application and value from local species.

Natural dyes have some shortcomings in terms of stability and affinity for substrates. Therefore, they require dye procedures or fixatives, such as mordants. Most of these mordants are metal salts, which could form coordination complexes with the natural dye molecule [6]. Also, biomordants, often tannin-rich compounds, which are responsible for the formation of hydrogen bonds, can be used in place of metallic mordants. The use of

these mordants improves the stability of the colour produced by natural dyes on substrates [7].

Natural dyes and ways of improving their affinity on textiles have been studied. For instance, the extraction, dyeing and colourfastness properties of dye extracted from the bark of *Croton urucurana* Baill have been investigated [8]. The extract was found to be a potential natural dye source for textile dyeing as good color fastness ratings of the dyed fabrics were obtained [8].

A study on the potential of anthocyanins from blueberries as a natural dye for cotton: a combined experimental and theoretical study, have been undertaken. The study explored the capabilities of anthocyanins as a natural dye on cellulose-based materials. The aim is to investigate the potential of different types of biomordants, which might be equally or more effective compared to metal mordants amongst others. It was reported that the use of metallic mordants was successful for stannous chloride with pre-mordanting only. It further revealed that biomordants showed no sufficient intermolecular interactions to replace this metal salt but endured the wash and light tests [9].

Extraction, characterisation and application of natural dyes from selected plants in Uganda for dyeing of the cotton fabric have been studied. The study used simultaneous mordanting method with 10% (o.w.f) of four selected mordants to dye cotton fabric. The mordanting process resulted in different ratings of colour depth, chroma (C) and hues (H) with noticeable changes in lightness rating (L), and mordant dependent. Light and wash fastness ratings were good, between 3 and 4, suitable for textile application [10].

Effects of mordanting methods of dye from *Veronia amygdalina* on cotton fabrics colouration have been studied. From the study, cotton fabrics when dyed in the absence of mordants showed a good wash fastness of (4), an excellent dry fastness of (4-5), wet rubbing fastness of (4) and good light fastness of (5). On the application of mordants, various colours were produced with different mordants, and the shades also varied with mordanting techniques though there was no significant improvement on the rubbing and wash fastness. Light fastness was improved from (5) to (6) when mordants were applied especially iron water, ferrous sulphate and potassium dichromate. Still, there was no significant increase in light fastness when alum was used as mordant. Furthermore, they observed that the application of mordants and the method of mordanting enhanced the colour characteristic of the dye on cotton fabrics. They reported that post mordanting technique gave the best colour fastness and strength on the fabrics for most of the mordants used [11].

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The dyeability and fastness qualities of dye from a henna plant on linen and cotton fibres in the presence of some selected mordants have been investigated [12]. The results of the experiment showed that cotton had a high rate of dye intake than linen. The report further suggested that the colours obtained depended mostly on the solvent used. However, hot water extraction appeared to be more efficacious [12].

An investigation has also been carried out on the extraction of natural dye from green chili (*Capsicum annum*) plant [13]. The dye was extracted using the solvent extraction method while the dyeing was done with two different mordants; copper sulphate and ferrous sulphate. The study reported that the principal colouring agent in green chili is oleoresin. The mordants produced different yellow shades of the chili dye. They gave good light fastness, good rub fastness and moderate wash fastness when the dye was applied on a 100% scoured cotton material [13].

Similarly, the effect of potassium aluminum sulphate (PAS) on dye concentration, hue, and fastness to wash and light for selected regional dyewoods (black walnut, Osage orange, and eastern red cedar) on woolen yarn has been investigated [14]. The use of a PAS mordant influenced the dye concentration, hue, and colour fastness to light and laundering of woolen yarn dyed with sawmill waste with implications for the triple bottom line. Regardless of the type of mordant used, Osage orange changed from a golden yellow to a tan/brown on exposure to light. PAS mordant caused an enhanced colour fastness to light when used for black walnut and eastern red cedar. In contrast, a significant difference for laundering but not for light, was observed for Osage orange [14].

A similar study compared aluminium mordants on the colour fastness of the natural dye on cotton fabric. The result of the study carried out clarifies that the type of aluminium mordant used had much influence on the colour fastness to washing whereas the type of dye used had much impact on colour fastness to light [15].

In another research work, ramie fabrics were dyed with four different natural dyes using rare earth as a mordant. The effect of pre-mordanting, simultaneous mordanting and post mordanting methods were studied. The result indicated that the highest dye uptake by the fabrics was obtained with the post mordanting method. It was also observed that the colour fastness to washing, light and rubbing of the ramie fabrics dyed with the natural dyes were improved by the use of rare earth as mordant. The study also was able to prove that rare earth can be an environmentally friendly mordant in natural dyeing [16].

Natural dye extracted from tea leaves have been used to dye cotton fabric. Use of ferrous sulphate following a pre-mordanting method increased the depth of shade of cotton much more than that obtained for the presence of (Copper sulphate, Tannic acid and Alum) in the dye bath [17].

The extraction of dye from Dapap tree (*Erythrina*) particularly *Erythrina fusca* L. and its application on cotton and silk in the presence of fixer solutions (FeSO_4 and CaCO_3) have been studied. From their experiment, they reported that the dye is a red colour dye and that FeSO_4 was a better fixer solution (mordant) than CaCO_3 [18].

In Nigeria, *Whitfieldia lateritia* is among the most understudied plants as a source of natural dye. *Whitfieldia lateritia* is an evergreen flowering plant belonging to the family of acanthaceae, having well-developed leaves alternately arranged along the stem. It has been found in large quantities in Sierra Leone and Nigeria, both in Africa [19,20].

Study on the proximate and mineral compositions of *Whitfieldia lateritia* leaves have been reported [21]. Analysis of the plant extract has shown that it contains flavonoids, alkaloids, saponins, cardiac glycosides and tannins in the leaves of *Whitfieldia lateritia* [20,22]. In a recent study, dyeing of cotton fabric with sodium hydroxide extract of *Whitfieldia lateritia* dye has been reported to be most efficient at 70°C for the range of temperatures studied, at a dyeing time of 60 min. Also, different mordants and mordant application techniques have been used to improve the affinity between the dye and cotton fibre thereby resulting in an increase in its colour fastness properties as well as a change in colour of the dyed fabric [23].

This work attempts to study the application and colour fastness properties of cotton fabrics dyed with aqueous dye extract from *Whitfieldia lateritia* in the presence of mordants.

Materials and Method

Materials

Whitfieldia lateritia plant leaves were obtained from Ihiagwa, Owerri, Nigeria. It was authenticated by Mrs. M. N Osuagwu of the Department of Plant Science and Biotechnology, Michael Okpara University of Agriculture Umudike, Nigeria. A desized and bleached plain weave cotton fabric (60 ± 1 warp and 48 ± 1 weft per inch) and white polyester fabric (55 ± 1 warp and 55 ± 1 weft per inch) were obtained from Woolen, and Synthetic Textile Manufacturing Ltd, Ikeja, Lagos Nigeria. Other materials used include perspirometer, blue wool scale, grey scale, distilled water. Sodium hydroxide, sodium chloride, potassium aluminium sulphate, tannic acid, disodium hydrogen orthophosphate dehydrate, histidine monohydrochloride monohydrate, sodium dihydrogen orthophosphate dehydrate were analytical grade products of Loba Chemie PVT Limited.

Sample preparation

The leaves of *Whitfieldia lateritia* plant were washed and kept under room temperature ($27 \pm 2^\circ\text{C}$) for 14 days to dry until crisp. The dried leaves were then pulverized using a mill to reduce particle size and passed through a micro-sieve of 53 μm mesh size and stored in polyethylene bags for further use. Different quantities of *Whitfieldia lateritia* powder were obtained using an electronic weighing balance to be used for extraction.

Dye extraction using distilled water as solvent

The extraction was done at material to liquor ratio (MLR) of 1:25 at a temperature of 70°C for 60 min using a thermostatic water bath. The methodology employed in Okonkwo et al. [23] was used. The aqueous dye solution was prepared by adding 1gm of *Whitfieldia lateritia* leaves powder in a beaker containing 25 ml of water and heating the solution at 70°C for 60 min. The dye solution was filtered to obtain a clear solution which was further used to dye cotton fabric. Figure 1 shows the images of the *Whitfieldia lateritia* leaves and aqueous extract used in this study.

UV-Vis spectroscopy and FT-IR spectroscopy of the dye extract

Cary 300 UV-Visible spectrophotometer was employed to determine the wavelength of maximum absorption (λ_{max}) of *Whitfieldia lateritia* dye extract. A blank was prepared for the extracting solvent. An automatic pipette was used to put the blank and the dye extract in their respective cuvettes. The sample was then scanned between the region 300-800 nm wavelength and the value for maximum absorbance, and its corresponding wavelength was determined [20]. FT-IR of the extract was conducted using the Cary 630 FT-IR spectrophotometer, with 16 scans within the spectral range of 4000-650 cm^{-1} at a resolution of 8 cm^{-1} .

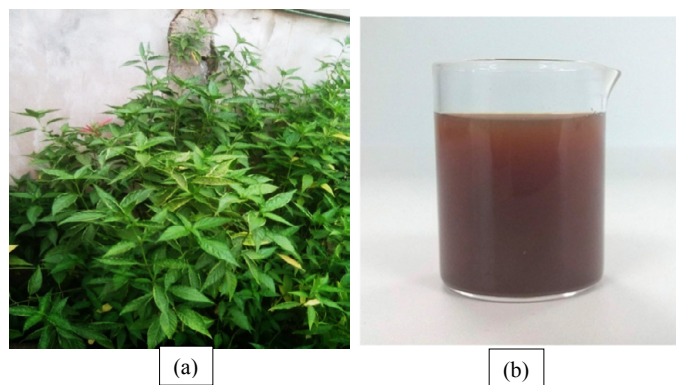


Figure 1. Images of the *Whitfieldia lateritia* (a) leaves and (b) aqueous extract.

Mordants and mordanting methods

Potassium dichromate, ferrous sulphate and a mixture of alum and tannic acid (50/50) were the mordants used for the fabric dyeing. The Pre-mordanting, simultaneous mordanting and post-mordanting methods were employed. The weight of mordant and sodium chloride salt (NaCl) used was 30% (0.3 g) each, on the weight (1 g) of the fabric. The methodology used in this present study for mordanting of fabrics was as reported in Okonkwo et al. [23].

Pre-mordanting

This involves the immersion of the fabrics in an aqueous solution containing the mordants at a temperature of 70°C for 40 min using a material to liquor ratio (MLR) of 1:40. After pre-mordanting, the dyeing of the mordanted fabrics was done for 60 min, in the presence of NaCl salt. The dye bath was stirred at 5 min interval during dyeing. After dyeing for 60 min, the fabrics were removed and rinsed in cold distilled water before drying under shade.

Simultaneous mordanting

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

Post-mordanting

In the post method, the fabrics were immersed in a dye bath containing the dye extract only. Dyeing was carried out at 70°C for 60 min at an MLR of 1:40 in the presence of NaCl salt. Intermittent stirring was maintained at every 5 min. After dyeing, the fabrics were then mordanted in a bath containing the mordant. Mordanting was carried out for 40 min at an MLR of 1:40 at 70°C. The fabrics were removed after mordanting and rinsed in cold distilled water before drying under shade.

Colour fastness assessment

Colour fastness of dyed fabrics was evaluated for washing, perspiration and light fastness properties, according to the test procedures used by Okonkwo et al. [23].

Results and Discussion

UV-Vis spectroscopy analysis

The UV-Vis Spectroscopy analysis of the aqueous dye extract of *Whitfieldia lateritia* is shown in Figure 2. The figure shows that the maximum absorption

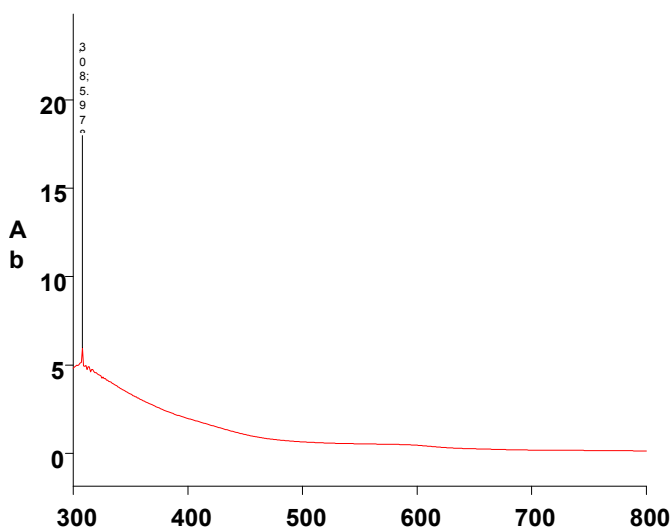


Figure 2. Absorption spectrum of aqueous dye extract of *Whitfieldia lateritia* leaves.

lies within the UV region, specifically at a wavelength of 308 nm and this falls within the spectral range of flavonoids. This result is in line with the findings of Okonkwo et al. [20] and Charu et al. [24]. Phenols and phenolic acids have been reported to show absorption bands between 250 and 290 nm. In comparison, flavones and flavonols exhibit two absorption bands of approximately the same intensity at about 250 and 350 nm [6]. The maximum absorption of the present extract may be associated with the presence of flavonoids.

FT-IR was used to identify the functional groups of the components in the dye extract. Figure 3 shows that the absorbance spectrum is pure since it has less than five absorption bands. This occurrence suggests that the extract components may be a low molecular weight compound [25]. The absorption band of O-H stretching vibrations around 3257.7 cm⁻¹ was observed. This suggests the presence of polyphenols, such as flavonoids and tannins [6]. The broad absorbance seen is due to the intramolecular hydrogen bonding between the OH group and adjacent oxygen atom [26]. This observation is in agreement with the result reported in Okonkwo et al. [20]. The peak at 2120.9 cm⁻¹ is an indication of the presence of C≡C stretch that is an alkyne. This is most likely because the intensity of the peak is weak, which is also why it may be symmetrically substituted. Furthermore, an absorption band around 1638.3 cm⁻¹ belonging to the C=C bond stretching vibrations was observed and is typical of a conjugated alkene.

Effect of mordant and mordanting methods on cotton fabrics dyed with aqueous dye extract of *Whitfieldia lateritia*

The effect of mordants and mordanting methods on dyed cotton fabrics are presented in Figure 4. It can be seen that the colour shade of the fabric

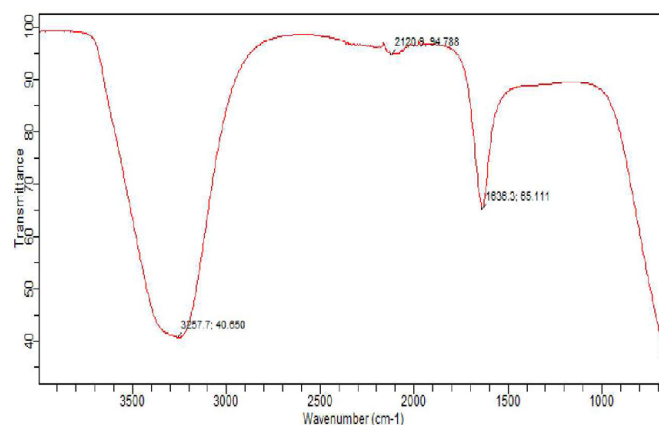


Figure 3. Infrared spectrum of *Whitfieldia lateritia* dye extracted with distilled water.

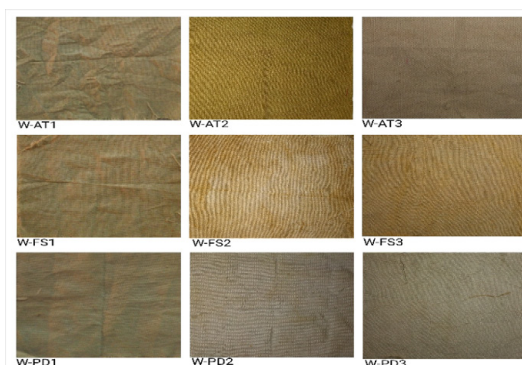


Figure 4. Cotton fabrics dyed with aqueous dye extract of *Whitfieldia lateritia* using different mordants and mordanting methods. Where: W-AT designates dyed cotton fabrics mordanted with a mixture of alum and tannic acid. W-PD designates dyed cotton fabrics mordanted with potassium dichromate. W-FS designates dyed cotton fabrics mordanted with ferrous sulphate. The numbers “1, 2 and 3” represent “pre-, simultaneous and post-” mordanting methods respectively as used in this study.

shifted with a change in the mordant application method for all the mordants used. From the figure, it is further observed that the use of mordant on *Whitfieldia lateritia* dye extracts for dyeing of cotton fabrics causes a shift in colour. The shift could be due to the interaction between the molecules of the mordant, dye extract and the fabric. This observation is consistent with the findings of Okonkwo et al. [20] and Okonkwo et al. [23].

Colour fastness to washing

Table 1 describes the results for the colour fastness properties to washing. Fastness to the colour change of the dyed fabrics after washing gave a good result as none of the samples was below 3; the highest grade being 4/5 for W-AT₂ followed by W-FS₃ (4). The least grade was 3 for W-AT₁, W-PD₁, W-PD₂ and W-PD₃ on the grey scale for colour change. From the result, there was a colour change after washing of the fabrics indicating that some dye molecules which were not firmly attached to the fabric left the fabric during washing. The adjacent cotton fabrics were not noticeably stained as the fastness to staining was 4 except for W-FS₃ which recorded 5.

Table 1. Colour fastness properties to washing of cotton fabrics dyed with aqueous dye extract of *Whitfieldia lateritia* using different mordants and mordanting methods.

Sample code	Wash Fastness	
	Colour change	Staining
W-AT ₁	3	4
W-AT ₂	04-May	4
W-AT ₃	03-Apr	4
W-PD ₁	3	4
W-PD ₂	3	4
W-PD ₃	3	4
W-FS ₁	03-Apr	4
W-FS ₂	03-Apr	4
W-FS ₃	4	5

Colour fastness to perspiration

Table 2 displays the results for the colour fastness property to perspiration to cotton fabric dyed with the aqueous dye extract of *Whitfieldia lateritia* using different mordants and mordanting techniques. The grey scale is used to rate the performance of fabrics assessed for colour fastness to perspiration to colour change of the specimen and staining of the adjacent fabric. From **Table 2.** Colour fastness properties to light and perspiration of cotton fabrics dyed with aqueous extract of *Whitfieldia lateritia* using different mordants and mordanting methods.

Sample code	Perspiration Fastness	Light Fastness
W-AT ₁	4	6
W-AT ₂	4	7
W-AT ₃	4	6
W-PD ₁	3	4
W-PD ₂	3	5
W-PD ₃	4	6
W-FS ₁	2	7
W-FS ₂	3	7
W-FS ₃	03-Apr	6

the results obtained, the cotton fabrics dyed with this dye extract recorded excellent performance with the pre-mordanted fabric W-FS₁ having the least grade (2). From the result, it shows that fabrics dyed with aqueous extract of *Whitfieldia lateritia* dye will perform moderately well under conditions that promotes perspiration.

Colour fastness to light

The results of the colour fastness properties to light of cotton fabric dyed with the aqueous dye extract of *Whitfieldia lateritia* using different mordants

and mordanting methods is shown in Table. The blue wool scale is used to rate the performance of fabrics assessed for colour fastness to light to the extent of fading of the test specimen. In Table 2, it can be seen that the ratings ranged from 4 for W-PD₁; 5 for W-PD₂; 6 for W-AT₁, W-AT₃, W-PD₃ and W-FS₃; and 7 for W-AT₂, W-FS₁ and W-FS₂. These results indicate a slow rate of fading of the dye colours on fabrics when used at areas having a moderate intensity of sunlight. The high ratings obtained can be attributed to the fact that when metallic mordants are used, a complex is formed with transition metals. The complex protects the chromophores from photolytic degradation. The chromophoric group absorbs the photons which by resonating within six-member ring dissipate their energy, thereby protecting the dyes [27]. Hence, the metal mordants used in the present study protect the dye from photolytic degradation, thus, giving excellent light fastness.

Conclusion

This work has explored the capabilities of aqueous dye extract of *Whitfieldia lateritia* in dyeing cotton fabric in the presence of mordants using different mordant application methods. The UV-Vis spectroscopy and FT-IR spectroscopy, both confirmed the presence of polyphenols such as flavonoids and tannins. The extracted dye can be used in combination with mordants such as the ones used in this work to colour cotton fabric. The different methods of the mordant application were seen to cause a change in the colour of the fabric. The general assessment of the colour fastness tests showed that the aqueous dye extract of *Whitfieldia lateritia* irrespective of the mordant and method of mordant application followed has good ratings.

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