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Extraction of Natural Dyes from *Whitfieldia lateritia* Plant and Its Application on Cotton Fabric

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Abstract

Extraction of natural dye from *Whitfieldia lateritia* plant and its application on cotton fabric was studied. In this research, natural dye was extracted from the leaves of *Whitfieldia lateritia* using 1% alkali as solvent. The dye extract was analyzed in the area of fabric dyeing using a mixture of alum and tannic acid as mordant, and sodium dithionite as reducing agent. UV-Vis spectroscopy, FTIR spectroscopy and colour fastness to washing, perspiration and daylight were also carried out. It was revealed that *Whitfieldia lateritia* dye is a direct dye since it dyed cotton fabric without the aid of a mordant. However, the application of mordant improved the colour fastness of the dyed cotton fabric. UV-Vis and FTIR spectroscopy confirmed the possible presence of some percentages of flavonoids, polyphenols and tannins in the dye.

Keywords: Natural dye; UV-Vis spectroscopy; FTIR spectroscopy; Mordanting; Colour fastness; *Whitfieldia lateritia*; Cotton

Introduction

The development of natural dyes which have better biodegradability with the environment was necessitated by the increase in the world's demand for safety fibers and dyes. Amidst growing environmental and health concerns eco-friendly nontoxic natural dyes re-emerged as a potential viable 'Green chemistry' option and a co-partner to some synthetic dyes [1]. These natural dyes are non-toxic, non-allergic to the skin, non-carcinogenic, easily available and renewable [2].

Whitfieldia lateritia is a flowering plant belonging to the family of *acanthaceae* [3]. It is native to Sierra Leone but recently has been observed in several parts of the world like Nigeria [3]. In Nigeria, it can be found in large number in places like 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. It is usually called by different names in several regions where they are found such as; "Ogwu obara" in Igbo; "Ogu`n eje" in Yoruba; and "Magani jinni" in Hausa language [3].

There have been reports of the medicinal values of *Whitfieldia lateritia* plant as it is widely used in folkloric medicine of Africa and Asia for the treatment of ailments such as inflammation, anemia and liver damage and boasting of blood [3]. The proximate and mineral compositions of *Whitfieldia lateritia* leaves have been evaluated using standard analytical protocols [4]. Reports on the qualitative phytochemical analysis revealed the presence of flavonoids, alkaloids, saponins, cardiac glycosides and tannins in the leaves of *Whitfieldia lateritia* [3]. Despite the use of *Whitfieldia lateritia* leaves for the treatment of various diseases, there is a dearth of documented data available regarding the presence of dyes or pigments in it and their methods of extraction. The chemical composition and structure of the *Whitfieldia lateritia* dye is not known since no research work has been done in this area. Its interaction with cotton fabric is not known. The major objective of this research is to extract natural dyes from *Whitfieldia lateritia* leaves, to identify and classify *Whitfieldia lateritia* dye extract.

Materials and Method

Materials

Distilled water, Sodium hydroxide pellets, Sodium chloride, Sodium dithionite, Potassium aluminium sulphate, Tannic acid, 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) and White polyester fabric (55 ± 1 warp and 55 ± 1 weft per inch). *Whitfieldia lateritia* plant leaves were obtained from Ihiagwa, Imo State, Nigeria. Thermostatic water cabinet Model: HH size 64 x 35 x 3 1 cm, Cary 300 UV-Vis machine, Cary 630 FTIR machine (Figure 1).

Method

Preparation of samples: The *Whitfieldia lateritia* plant leaves were

Figure 1: *Whitfieldia lateritia* plant.

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

Dye Extraction using 1% Sodium Hydroxide as solvent: The extraction was done at a material to liquor ratio of 1:50 (g:ml) using 1% alkali solution. Aqueous dye solution was prepared by adding 1 gm of leaves of *Whitfieldia lateritia* in 50 ml of water. The extraction was done at 70°C for one hour. The hot solution was filtered and a clear solution was obtained which is used for dyeing cotton fabric.

Identification of the chemical composition of the extracted dye: In textiles, natural dyes are identified through selective extraction of dyes and comparing each dye by various testing techniques such as UV-Vis and IR spectroscopy amongst others.

UV-Vis spectroscopy and fourier transform Infra-red spectroscopy: The UV-Vis spectroscopy of the extracted dye was scanned within the wavelength of 300-800 nm using a Cary 300 UV-Vis machine. FT-IR of extracted dye was traced on Cary 630 FTIR machine within a wavenumber range of 4000-650 cm^{-1}.

Classification of the extracted dye: For this study, three tests on dyeing of cotton fabric with and without certain dye additives were carried out. The additives used here are sodium dithionite (reducing agent) and equal mixture of alum and tannic acid (mordant). The reducing agent was added to determine if the dye was a Vat dye while the mordant was added to determine if the dye was a mordant dye. 30% (owf) of pure sodium chloride and the mordant were used. Dyeing was done at a material to liquor ratio of 1:50 for 30 minutes at a temperature of 70°C. The material was dyed at 70°C to avoid possible denaturation or degradation of the dye molecules at high temperatures.

Dyeing of cotton fabric without a reducing agent or mordant: 50 ml of *Whitfieldia lateritia* dye extract was poured into a 100 ml beaker (dye bath). 0.3 g (30% owf) of pure sodium chloride (NaCl) was added and stirred. The dye bath was then put over a water bath with temperature set at 70°C. When the temperature of the dye bath got to 70°C, 1 g of already wetted cotton fabric was then entered into the dye bath. The dye bath was stirred at every 5 minutes interval of dyeing and the fabric was removed after 30 minutes of dyeing. The fabric was rinsed in cold distilled water and dried under a shade. Fabric designation is A1.

Dyeing of cotton fabric with a reducing agent: 50 ml of *Whitfieldia lateritia* dye extract was poured into a 100 ml beaker (dye bath). 0.3 g (30% owf) of pure sodium chloride (NaCl) and 1.0 g of sodium dithionite ($Na_2S_2O_4$) was added and stirred. The dye bath was then put inside a water bath with temperature set at 70°C. When the temperature of the dye bath got to 70°C, 1 g of already wetted cotton fabric was then entered into the dye bath. The dye bath was stirred at every 5 minutes interval of dyeing and the fabric was removed after 30 minutes of dyeing. The fabric was rinsed in cold distilled water and dried under a shade. This fabric has the designation A_2

Dyeing of cotton fabric with a mordant: The dyeing was done at a material to liquor ratio (MLR) of 1:50 in the presence of 30% (owf) of mordant in a pre-mordanting method. 30% (owf) (0.3 g) of mordant (a mixture of alum and tannic acid in the ratio of 50:50) was dissolved in 50 ml of distilled water. The solution was put in a water bath with temperature set at 70°C. When the temperature of the solution got to 70°C, 1.0 g of cotton fabric was then entered into the solution and stirred. After 30 min, the fabric was removed and excess water squeezed out. Fabric was then ready for dyeing.

50 ml of *Whitfieldia lateritia* dye extract was poured into 100 ml beaker and 0.3 g (30% owf) of pure sodium chloride (NaCl) added and stirred. 1.0 g of the mordanted fabric was entered into the dye bath and stirred. The dye bath was stirred at every 5 minutes interval of dyeing. The fabric was removed after 30 minutes of dyeing and rinsed in cold distilled water before drying under shade. The fabric designation is A_{31}.

Colour fastness testing

Colour fastness to washing: The test was carried out for 30 minutes at a temperature of 60°C without any stainless steel balls using a liquor ratio of 50:1 (ml:g).

Soap solution was prepared by dissolving 5 g of bar soap in 1 litre of distilled water. The fabric samples were prepared by sewing the dyed cotton fabric of size 100 ± 2 mm × 40 ± 2 mm and weight 1 g, to a plain weave bleached cotton fabric of same size and weight at their shorter ends. The washing was done in a 100 ml beaker containing soap solution and other additives as prescribed above under the specified conditions of liquor ratio, temperature and time.

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

Colour fastness to perspiration: This test measures the resistance of the colour of textile fabrics to perspiration in all forms. The methodology followed was as reported in Saville with modifications [5]. Perspiration was carried out under acidic and alkaline solutions.

The alkaline solution contains 5 g/L sodium chloride, 2.5 g/L disodium hydrogen orthophosphate dehydrate, 0.5 g/L histidine monohydrochloride monohydrate brought to a pH 8 with 0.1M sodium hydroxide while acidic solution contains 5 g/L sodium chloride, 2.2 g/L sodium dihydrogen orthophosphate dehydrate, 0.5 g/L histidine monohydrochloride monohydrate brought to a pH 5.5 with 0.1M sodium hydroxide. A mixture of the prepared solutions in a v/v ratio of (25:25) was then made and used for further studies.

A composite sample was made by sand-witching the dyed sample measuring (5×5) cm between two pieces of un-dyed bleached cotton fabric of the same dimension. The composite specimen was thoroughly wetted in the prepared solution (acidic/alkaline) at room temperature for 30 minutes. At the end of 30 minutes the composite specimen was removed from the solution and wiped off of excess liquid and then placed between two glass plates of the Perspirometer measuring about 7.5 x 6.5 cm under a pressure of 12.5kPa. The Perspirometer containing the treated composite sample was then placed in an oven at 37°C for 4 hours. After 4 hrs, the specimens were removed and dried at room temperature.

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

Colour fastness to daylight: This test was done following measures the resistance of a textile material to fading when exposed to daylight. A small piece of the dyed cotton fabrics of mass 0.25 g measuring about 3 × 3 cm were cut and mounted on pattern cards (Blue Wool Scale). The test samples were then exposed to daylight for 72 hours, after which the sample was removed and compared with the original unexposed samples. The changes in colour were assessed using a Blue Wool Scale. The results of the test were then recorded.

Results and Discussion

This research was carried out to ascertain the chemical constituents of the alkali extract of *Whitfieldia lateritia* which will serve as a guide during the classification of the dye. FTIR and UV-Vis spectroscopy analysis were useful in this regard. The classification was done after dyeing cotton fabric with certain additives such as mordant and reducing agent and subjecting the dyed fabric to wash, perspiration and light fastness tests.

Chemical composition of the colourant

The alkali extract of *Whitfieldia lateritia* dye shows maximum absorption at 416 nm which falls within the spectral range of flavonoids and this result is in line with the findings in Figure 2 [6].

From the IR spectroscopy analyses, IR- peak at 1602.8 cm^{-1} and 1654.9 cm^{-1} for sample A shows the presence of alkene (C=C) stretching typical of flavonoid-based compounds (Figure 3). The peak at 1729.5 cm^{-1} in sample A could be due to the stretching of carbonyl group (C=O). This region is attributed to the presence of CO stretching of esters of hydrolysable tannins, especially derivatives of gallic acid [7]. The broad peak at 3250.2 cm^{-1} for sample B indicates the presence of alcohols or phenols (Figure 4). It can therefore be inferred that the extract of *Whitfieldia lateritia* plant contains flavonoids, polyphenols and tannins.

Colour fastness

The colour fastness property of cotton fabrics dyed with *Whitfieldia lateritia* dye extract in different areas which includes: dyeing without a mordant and reducing agent; dyeing with reducing agent only; and dyeing with mordant only, was evaluated.

Colour fastness to washing: The data presented in Table 1 and Figure 5 shows that fabric dyed with *Whitfieldia lateritia* dye showed good fastness to colour change 3 for A_1 and 4/5 for A_{31}. However, A_2 showed the least rating of 2. The grey scale rating of 4/5 implies that there was a negligible colour change of the fabric after washing. Fabric A_2 despite its deep shade failed to give a good value on the scale. This is indicative that the dye is not a vat dye since there was poor fixation of the dye on the cotton fabric hence the fabric lost more dye molecules during washing leading to its poor grade on the grey scale rating. The colour staining assessment for all the fabrics at the three areas checked gave a grade of 4 for all fabrics meaning that the adjacent cotton fabrics were not obviously stained by the dye.

Colour fastness to perspiration: The colour fastness property to perspiration of the dye studied showed good to excellent result on the grey scale for colour change with fabrics A_1 and A_2 having the least rating of 3 and 4 respectively. This result is an indication that fabrics dyed with alkali extract of *Whitfieldia lateritia* dye will perform well under conditions where perspiration is imminent.

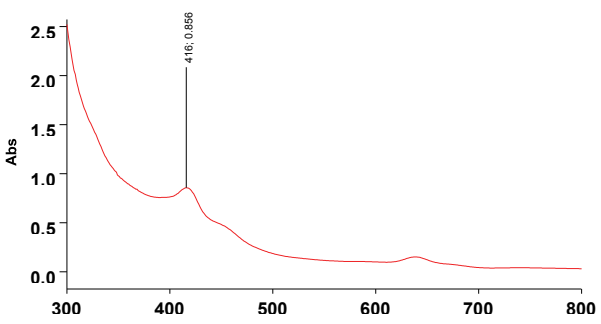

Figure 2: UV-Vis Spectroscopy analysis showing maximum absorbance and wavelength of alkali extract of *Whitfieldia lateritia* dye.

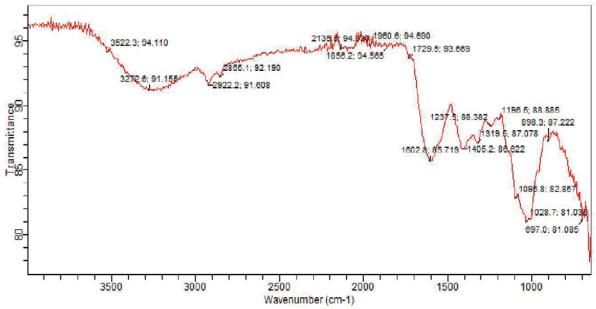

Figure 3: Infrared spectrum of *Whitfieldia lateritia* plant leaves dry powder (Sample A).

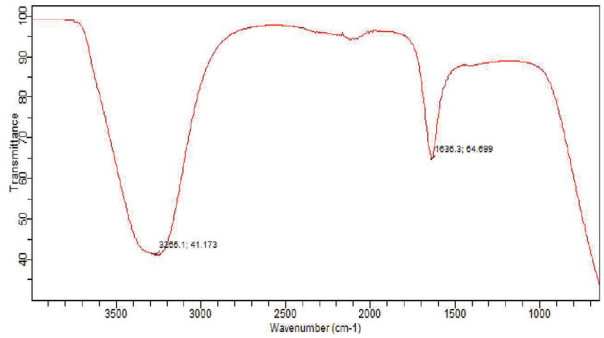

Figure 4: Infrared spectrum of the alkali extract of *Whitfieldia lateritia* plant (Sample B).

Sample code	Wash fastness		Perspiration fastness	Light fastness
	Colour change	Staining		
A_1	3	4	3	4
A_2	2	4	4	4
A_{31}	4/5	4	4/5	6

Table 1: Results of Colour fastness to washing, perspiration and light of dyed Samples A_1, A_2 and A_{31} on cotton fabrics.

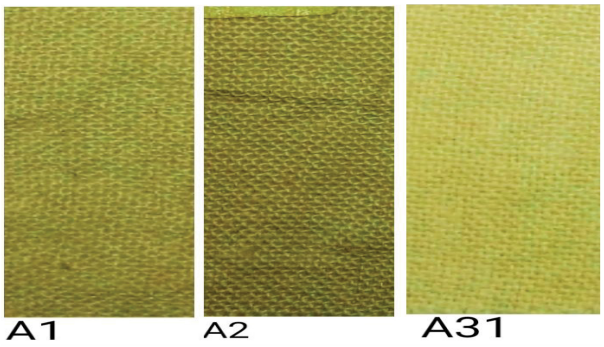

Figure 5: Cotton fabrics dyed with *Whitfieldia lateritia* dye.

Colour fastness to light: Colour fastness test to daylight of the dyed fabrics was also carried out. From the result the fabrics recorded somewhat fair values on the blue wool scale except for A_{31} having a

rating of 6 which is an indication that the fabrics can be used under areas of moderate sunlight intensity.

From Figure 5 it can be seen that the cotton fabric was dyed by the extract in all the three areas considered. However, the colour depth on the fabrics differed based on the additive used. Comparing the colour strength of fabrics dyed, it was observed that fabrics dyed with only reducing agent (A_2) had a deeper/stronger colour shade than fabrics dyed without any additive (A_1) and fabric dyed with mordant (A_{31}). The colour strength of A_1 and A_2 fabrics are almost similar. It is very likely that the lighter shade of the dyed pre-mordanted fabric is due to the presence of the mordant. Such compounds are able to produce insoluble colour complexes with certain azo and anthraquinone dye derivatives [8]. The use of mordant in dyeing also leads to bathochromic shift in colour, notwithstanding the duller hue and improved washing and light colour fastness [8]. The colour depth of the dye extract on the dyed fabrics is in this order from highest to lowest; $A_2 \rightarrow A_1 \rightarrow A_{31.}$

Classification of dye extract

From the tests carried out, it was found that the dye extract is a direct dye since it dyed the cotton fabric to a reasonable colour depth and fastness without a mordant or reducing agent. The dye extract when applied on cotton fabric under the influence of a mordant showed improved colour fastness and change in hue when compared with the fabric dyed without any additive and this agrees with the findings of that the application of mordants improves colour fastness and causes a bathochromic shift in colour [8]. The chances of being a vat dye was eliminated when dyed with a reducing agent as the dye extract liquor did not change colour when the reducing agent was added meaning that there was no reduction reaction. When left in the open, just after dyeing, fabrics dyed with vat dyes change to the actual dye colour as a result of oxidation but this was not the case with fabric dyed with *Whitfieldia lateritia* dye extract when it was left in the open for oxidation before washing. Again the dyed fabric colour did not change when it was left in the open for oxidation before washing. All these are indications that the dye extract used was not a vat. It can therefore be inferred that the dye extract is a direct dye but the application of mordant can cause increased affinity for cellulose fibre through the formation complexes.

Conclusion

The present work showed that, dyes can be extracted from the leaves of *Whitfieldia lateritia* and these dyes can be used as a dye for cotton fabric. Good colour fastness property can be obtained by incorporating a mordant such as alum/tannic acid in a pre-mordanting technique as studied. On the overall, the mordanted fabric (A_{31}) gave the best colour fastness property to washing, perspiration and daylight whereas the effect of reducing agent such as sodium dithionite was not so pronounced. The UV-Vis and IR spectroscopic analysis revealed that alkali extract of *Whitfieldia lateritia* dye contains flavonoids, polyphenols and tannins.

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