



Extraction and Testing of Natural Dye from Dafara (*Cissus populnea*) Stem Bark and Its Application on Cotton Fabric

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Authors' contributions

This work was carried out in collaboration between all authors. Author PMD designed the study, wrote the protocol and wrote the first draft of the manuscript. Author MAA managed the literature searches, analyses of the study and performed the spectroscopy analysis. Author DH managed the experimental process and author JJ identified the species of plant. All authors read and approved the final manuscript.

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ABSTRACT

Extraction and testing of natural dye from *Cissus populnea* stem bark and its application on cotton fabric was studied. The maximum absorbance of crude dye from *Cissus populnea* stem bark extract at different temperature was measured by Ultra Violet-Visible spectrophotometer in a wavelength range of 495 nm-535 nm. It was observed that, the effect of time on dye extraction showed increase in intensity of dye as time increases. Ultra Violet-Visible spectrophotometer also showed that as the extraction time increases the absorption wavelength (nm) also increases from 450 nm to 560 nm. This was ascribed to the high yield of extract and the subsequent evaporation of solvent. Effect of temperature on extraction showed that as the temperature increases from 40°C to 100°C the dye intensity also increases. This was credited to the gradual increase in the removal of the dye components vis a viz increase in dye concentration. Fixed dye test on cotton fabric revealed that the dye works best with a mordant at higher temperatures. This was attributed to

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interaction between N-H functional group of dye and the O-H functional group of the cotton fabric. Infra-Red determination of unmodified dye extract of *Cissus populnea* stem bark showed absorption at 1636.3 cm^{-1} which confirmed the presence of amide group (R-NH₂), with a medium-strong intensity of N-H bending. The dye extracted could be of amino-anthraquinone group. Its brightness and improved wash fastness on cotton fabric suggested that it could be used on clothes by the garment making industries when mordant added.

Keywords: *Cissus populnea*; cotton; dye; extraction; fabric; mordant; textile.

1. INTRODUCTION

Dye from natural resources especially from plants are increasingly becoming important alternatives to synthetic dyes for use in the textile industry [1]. Unlike synthetic dyes which have been found to be toxic and harmful to the environment, natural dyes are biodegradable, non-toxic and generally have higher compatibility with the environment when comparable to synthetic counterparts [2].

The use of non-allergic, non-toxic and eco-friendly natural dyes on textiles has become a matter of importance due to the increased environmental awareness in order to avoid some hazardous synthetic dyes. However, worldwide the use of natural dyes for the colouration of textiles has mainly been confined to artisan / craftsman, small scale / cottage level dyers and printers as well as to small scale exporters and producers dealing with high-valued eco-friendly textile production and sales [3]. Recently, a number of commercial dyers and small textile export houses have started looking at the possibilities of using natural dyes for regular basis dyeing and printing of textiles to overcome environmental pollution caused by the synthetic dyes. Natural dyes produce very uncommon, soothing and soft shades as compared to synthetic dyes. On the other hand, synthetic dyes are widely available at an economical price and produce a wide variety of colours; these dyes however produce skin allergy, toxic wastes and other harmfulness to human body.

Cissus populnea is a plant associated with a myriad of medicinal uses in different parts of the world. Its extracts have been credited with antibacterial properties as components of a herbal anti-sickling Nigeria Formula [4].

The plant *Cissus populnea* Guill & Perr belongs to the family *Amplidaceae* (*Vitaceae*). It is readily found almost in every forest area of tropical Nigeria especially the middle belt area and in other African countries (Senegal, Sudan,

Uganda, Abyssinia) most of the plants grow wildly while some are cultivated. It is commonly known as 'Uro Okoho' by the Idomas, Igbo and Igala tribes of Nigeria; 'Dafara' (Kano, Zaria); 'latutuwa' (Katsina) by the Hausa language of the indicated towns of northern Nigeria [5]; Ethno-medicinal uses include treatment of sore breast, indigestion, venereal diseases, intestinal parasites, oedema and eye problems resulting from attack of black cobra (*Naja nigricollis*) [6]. The plant is also used as cathartic, aphrodisiac and antidote to arrow wounds. The stem bark has been reported to contain carbohydrates, tannins, cyanogenic glycosides, anthraquinones, saponins, cardiac glycosides and flavonoids [7].

Cissus populnea stem is a potential dye producing plant which has not been exploited. In this research, dye was extracted from the dried stem bark powder of *Cissus populnea* plant and then applied on a cotton fabric.



Fig. 1. Dafara (*Cissus populnea*) 'white stem' plant

2. MATERIALS AND METHODS

2.1 Chemicals/Reagents

Cissus populnea stem bark, distilled water, sodium chloride, cotton fabric, methanol, hydrochloric acid, aluminiumsulfat-18-hydrate.

2.2 Instrument /Apparatus

UV-Visible spectroscopy, IR spectroscopy, 400ml beaker, Funnel, Mortar & Pestle, Sieve, electronic compact scale Weigh meter, stirrer, 25 cm³, 250 cm³ MBL measuring cylinder, 250 ml beaker, LABSMAN 24 cm filter paper, thermometer 200°C, Jenway 400 pH meter, spatula.

2.3 Sampling and Sample Preparation

2.3.1 Sampling of plant

Potential dye-yielding plants namely *Cissus populnea* stem of the family *Amplidaceae* (*Vitaceae*) was collected in the month of August 2015 in Yola-Adamawa state, Nigeria, dried and kept.

2.3.2 Sampling preparation

The dried stem bark of the plant was grind and kept in a properly sealed and well labeled plastic container in the chemistry laboratory of Modibbo Adama University of Technology, Yola-Nigeria.

2.4 Preparation of Standard Reagents for the Modification of Dye [ASTM D1696, 2011]

- A 10% Sodium Hydroxide** was prepared by dissolving 50 g of the compound in water, and dilute with water to 500 ml.
- A 10% Hydrochloric Acid** was prepared by diluting 118 mL of the compound with water to 500 mL.

2.5 Extraction of Dye from *Cissus populnea* Stem

Hot method of extraction was used to extract the dye from the stem bark. The powdered stem bark of *Cissus populnea* (9 g) was detanned with acetone by percolation. The absence of tannins was tested with lead subacetate. The detanned crude stem extracts from *Cissus populnea* was added to 250 ml of distilled water in a 400ml Pyrex beaker. The mixture stirred, heated to boiling for 3 hours, allowed to stand for 20 minutes and then filtered using a 24 cm filter paper. The coloured filtrate (200 ml) was used for dyeing the fabric in the presence of 10% (o.w.f) selected mordant [8].

2.6 Measurement of the Absorption of the Dye Using UV-Visible Spectrophotometer (UV-Visible D4951)

Identification of natural dyes in textiles involves selective extraction of dyes and comparison of each dye by various testing techniques, viz. UV-Visible and IR spectroscopy.

The colour values of the dyed fabrics with selected mordants was obtained. The amount of the light reflected at each wavelength by each dyed fabric sample was plotted as a percentage of the amount of light falling on the coloured surface at each wavelength. The curve obtained provides a detailed description of the colour properties of dyed samples with mordant evaluated using standard procedures.

2.7 Treatment of Cotton Fabric (ASTM D 2259)

Cotton fabric was treated with sodium hydroxide. In this dye-fixed test, 1.70 g cotton fabric was immerse into 50 ml of 10% sodium hydroxide solution for 30 minutes then dried for use in the dyeing process.

2.8 Dyeing Process

Dyeing of cotton fabrics (1.70 g) was carried out using the simultaneous mordanting method at boiling for 1 hour with constant stirring using a fixed amount of liquor ratio (1:200). The dyed samples was thoroughly washed with cold and hot water to remove any unfixed dyed material and finally dried in open air [8].

2.9 Fixed-Dye on Cotton Fabric Test

In this dye-fixed test, the treated cotton fabric was weighed (W_p), immerse into 50 ml of the dye bath containing 0.2 g of aluminum sulfate and is heated at 100°C for 1 hour, cooled and dried. It was wash with cold water then hot water to remove the unfixed dye, the cotton fabric was dried and weighed (W_f). The purpose and scope of this is designed to measure the amount of fixed-dye on the cotton fabric [1]

$$\text{Weight of fixed dye} = \frac{w_f - w_{\text{pure}}}{w_{\text{pure}}} \times 100$$

Where

$$W_{\text{pure}} = \text{weight of Pure cotton fabric}$$

W_f = weight of dyed cotton fabric

This process was repeated at different temperature interval of 80, 60, 50 and 40°C, values obtained are recorded

2.9.1 Effect of temperature on wash fastness test

Wash fastness without Mordanting agent was done by immersing 1.70 g (W_{pure}) of the treated cotton fabric into 50 ml of the dye in the absence of a mordant (Alum) and heated to 100°C for 30 minutes [9], cooled and dried. It was wash with cold and later hot water to remove any unfixed dye. The cotton fabric was dried and weighed (W_f).

$$\text{Weight of fixed dye} = \frac{W_f - W_{pure}}{W_{pure}} \times 100$$

Where

W_{pure} = weight of Pure cotton fabric
 W_f = weight of dyed cotton fabric

This process was repeated at different temperature interval of 80°C, 60°C, 50°C and 40°C.

Note; this whole process was repeated using aluminum sulfate as a mordant [1].

2.9.2 Effect of concentration on wash fastness test

Wash fastness test was carried out by dyeing 1.70 g of cotton fabric in 5/40 ml of the dye bath in the absence of a mordanting agent. The purpose and scope of this is designed to measure the amount of fixed-dye on the cotton fabric.

$$\text{Weight of fixed dye} = \frac{W_f - W_{pure}}{W_{pure}} \times 100$$

Where

W_{pure} = weight of Pure cotton fabric
 W_f = weight of dyed cotton fabric

This process was repeated at different concentration of 10/40, 20/40, 30/40 and 40/40 ml [1]. This process was repeated using aluminum sulfate as a mordant and values recorded.

3. RESULTS AND DISCUSSION

3.1 Effect of Time on the Extraction Crude Dye from Stem Bark of *Cissus populnea* Plant

Extraction is a separation process which involves the removal of a substance from its matrix. In this extraction process the stem bark of *Cissus populnea* was initially detanned using acetone by percolation to remove tannins which have a yellowish color. Dye was extracted from the detanned substance (Red color) at different time interval and the different samples were analyzed using UV-Visible spectrophotometer.

The result from Fig. 3 shows that, the intensity of the dye increases with increase in time. As the extraction time increases the absorption wavelength (nm) increases (490-560 nm). This may be due to the high yield of extract and the evaporation of solvent. Also, from the absorbance, the intensity of color increases with increase in extraction time. The IR shows (Fig. 4) that radiation in this region is utilized by the organic molecule for structural elucidation due to inter-atomic bonds vibrations. Chemical bonds in different environments will absorb varying intensities and at varying frequencies.



Fig. 2. Dye extract from *Cissus populnea* stem bark powder using hot method of extraction

3.2 IR Determination of Unmodified Dye Extract of *Cissus populnea* Stem Bark

Fig. 4 shows the IR chart of pure dye extract of *Cissus populnea* stem bark. Absorbance at

3268.9 cm^{-1} corresponds to a strong C-H of weak-medium intensity of N-H symmetry and asymmetry stretch. The absorption at 1636.3 cm^{-1} confirms the presence of amide group (R-NH₂) with a medium-strong intensity of N-H bending. Stretching frequencies are higher than corresponding bending frequencies. This is because it is easier to bend than to stretch or compress a bond. This absorption also indicates the presence of an amine group due to N-H bending with a weak-medium intensity. Thus, N-H is the functional group that may likely react with the OH functional group of the cotton fabric. Therefore, the amount of available N-H and or O-H groups could be responsible for the intensity of fixed on the fabric.



Fig. 3. *Cissus populnea* stem bark powder

3.3 Effect of Temperature on Dye Extraction

The maximum absorbance of crude dyes extracted at different temperature was measured by UV-Visible spectrophotometer in a wavelength range of 495-535 nm. Fig. 5 shows the λ -max of each extract observed in the curve recorded perfectly matched to that of standard anthraquinone dyes [10] possibly amino-anthraquinone.

Temperature is the main factor which affects the extraction efficiency and selectivity in natural anthraquinone. It could influence the physicochemical properties of water whereby at high temperature, polarity of water decreasing and thus enhancing the solubility of less polar compounds in water. Results in this study (Fig. 6) show that the temperature increases gradually from 50°C up to 100°C, the maximum

absorbance of *Cissus populnea* extracts was obtained at λ -max 535 nm which correspond to that of standard anthraquinone. This explains that at higher temperature, water was able to extract larger amount of less polar compound [11].

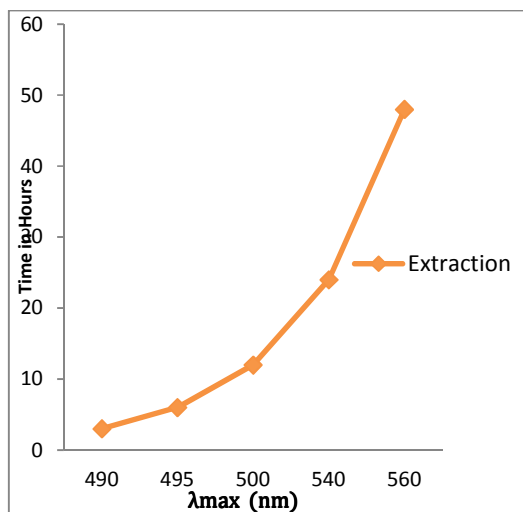


Fig. 4. Effect of time extraction of the crude stem bark of *Cissus populnea*

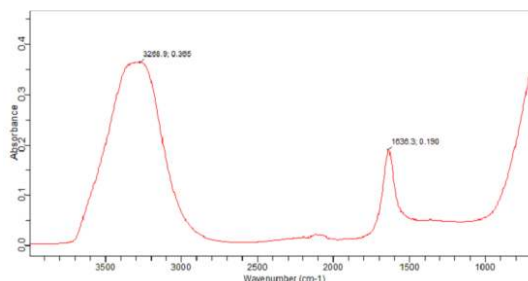


Fig. 5. IR chart of crude dye extract from *Cissus populnea* stem bark

3.4 Wash Fastness Test for the Dyed Cotton Fabric

Wash Fastness Test of dyed fabric at different Temperature is shown in Fig. 7, dye-fixation on the cotton fabric was favoured at higher temperature i.e. as the temperature increases the dye-fixation also increases, this implies that temperature increase affinity for dye-fixation on cotton fabric [12]. This may be due to collisions between the cotton fabric (Cell-OH) and the dye (Dye-NH₂) was more violent at higher temperatures. The higher temperatures mean higher molecular velocities this means there was less time between collisions. The frequency of

collision was increase. The increased number of collisions and the greater violence of collisions results in more effective collisions. The rate for the reaction increases.



Fig. 6. Cotton fabric dyed

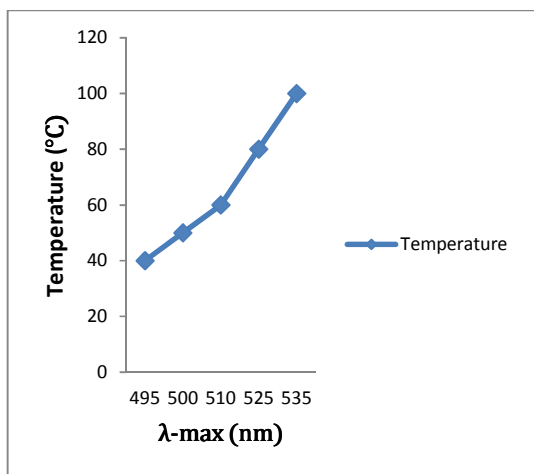


Fig. 7. Effect of temperature (°C) on dye extraction of *Cissus populnea* stem bark

3.5 Effect of Temperature on Fixed-dye “with” a Mordant and “without” Mordant

Fig. 9 shows the effect of temperature on Dye-fixed with a mordant and without a Mordant. Those without mordant (aluminum sulfat-18-hydrate) shows a lower dye-fixation on the cotton fabric compared to the one having aluminum sulfat-18-hydrate. Although there was a bit increase of dye-fixed on the cotton fabric as the temperature increase (1 – 2.2%). Temperature effects on rates of kinetic theory says that molecules are in constant motion. The velocity of the molecules is directly proportional to the

Kelvin temperature. The kinetic energy and molecule velocity increase with temperature. $KE = [1/2] [mv^2]$. The mass of the molecules is “m” and the velocity is “v”. Covalent bonds are the strongest chemical bonds and are formed by the sharing of a pair of electron. Cotton fabric dyed with a Mordant shows a high Dye-fixation on the cotton fabric, this may be due to the metallic salt forming complex [13]. This indicates that the dye works better with Aluminum Sulfat-18-hydrate as mordant.

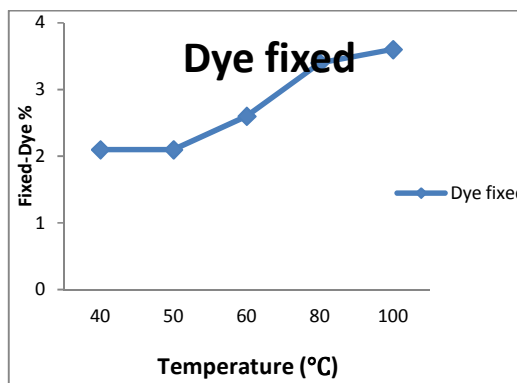


Fig. 8. Wash fastness test for dyed cotton fabric at different temperature

3.6 Effect of Concentration on Fixed-Dye “with” a Mordant and “without” a Mordant

Fig. 10 shows the effect of concentration on fixed-dye with aluminum sulfat-18-hydrate as mordant and without a mordant. Concentration is the amount of solute per volume of solvent. An increase in concentration produces more collisions. The chances of an effective collision goes up with the increase in concentration. The exact relationship between reaction rate and concentration depends on the reaction “mechanism”. This is the process involving elementary reaction steps. There was a decrease in dye active bond sites as the concentration reduces. The effect of concentration on fixed-Dye with aluminum sulfat-18-hydrate as mordant shows a higher fixed-dye compared to dye-fixed without mordant. Effect of fixed-dye on cotton fabric without aluminum sulfat-18-hydrate as a mordant show a little dye fixation on the cotton fabric [14,15].

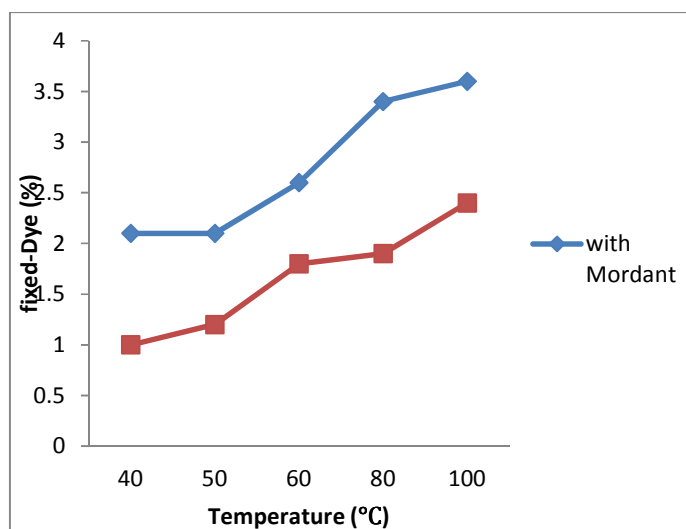


Fig. 9. Effect of temperature on fixed-dye “with” a mordant and “without” mordant

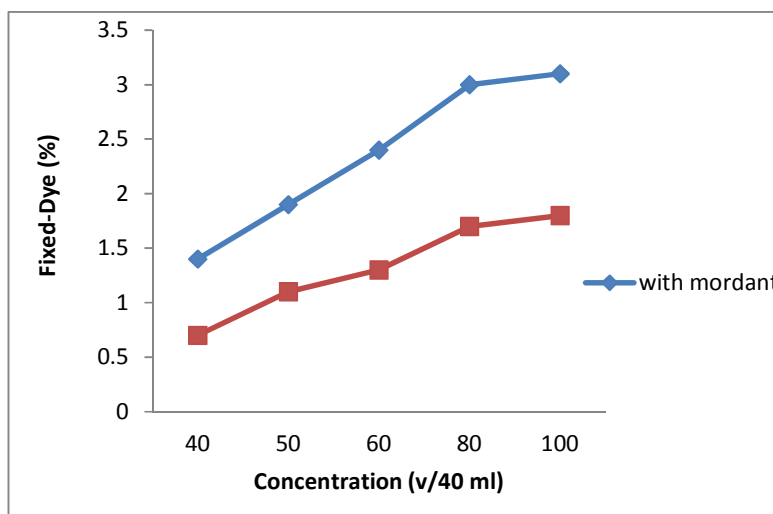


Fig. 10. Effect of concentration on fixed-dye “with” a mordant and “without” mordant

4. CONCLUSION

The dye obtained displays fairly good saturation on cotton with medium to good fastness properties. Fixed-Dyeing properties these include; good wash fastness also favours the mordants. The amount of available N-H and or O-H groups could be responsible for the intensity of fixed on the fabric. The dye works best with a mordant and this mordant favours the uptake. It also provides means of increasing the brightness of natural dyes of anthraquinone, hence improving the wash fastness of cotton fabric used for cloth making.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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