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Production and Testing of *Baphia Ntida* Stem Dye on Cotton Fabric

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ABSTRACT

The application of natural dyes on fabric has witnessed growing interest due to environmental pollution. In this work, natural dye was extracted from heartwood of *Baphia nitida* and was tested on cotton fabric. The extracted dye was characterized using FT-IR spectroscopy, GC-MS and UV-Visible spectroscopy. Scouring, bleaching, rubbing fastness and wash fastness properties were evaluated on the dyed cotton fabric. The natural dye extract was applied on the cotton fabric without a mordant. The percentage yield of the dye obtained was 16.91. The colour of the dye extract is red. The results of the FTIR characterization suggested the presence of OH, C-H, CHO, C≡N, C≡C, C=O, N=O, C=C, C-Cl and C-Br functional groups in the dye extract. The GC-MS separated and identified 30 compounds. Among the compounds identified from the dye extract, Benz(a)anthracene, 7,8 - dimethyl, had the highest percentage area of (35.09%) and 1,3,5 - Triazine - 2,4,6 - triamine had the lowest percentage area. The chromophores in the dye extract were N=O and C=O. The colour fastness to washing showed that 2-3 and 4 were experienced for colour change and staining respectively while the colour fastness to rubbing showed that 3-4 and 2-3 colour changes were experienced for dry and wet rubbing respectively without mordant. The outcome of colour fastness of the dye showed a very good affinity to remain on the fabric when mordant was applied.

Keywords: *Baphia nitida*, Dye, Extraction, Cotton fabric

INTRODUCTION

Baphia nitida, also known as camwood, barwood, and African sandalwood (although not a true sandalwood), is a shrubby, leguminous, hard-wooded tree from central west Africa. It is a small understorey, evergreen tree, often planted in villages, and known as *Osun* in Yoruba [1]. Plate 1 shows *Baphia nitida* tree which is used as a medicinal plant. In particular, it has been used in traditional African medicine [2]. The leaves of *Baphia nitida* have anti-inflammatory activities, antidiarrhea effects, and analgesic activities. Powdered heartwood can be made into an ointment

with shea butter for treatment of sprains, swollen joints, and rheumatic pains [3]. The roots have medicinal properties as well.

Natural dyes are a non-toxic alternative to conventionally used synthetic dyes that are causing irreversible damage to the planet [4]. By using natural dyes, there is a direct cut back on the toxic chemicals being released that are associated with synthetic dye [5]. Synthetic dyes are not environmentally friendly as their synthesis involve extreme conditions such as high pH, high temperature, strong acids, and heavy metal catalysts. Synthetic dyes are toxic, carcinogenic and can cause skin and eye irritation [6]. Synthesis generates a large amount of effluent which contains toxic chemicals generated as side products. Synthetic dyes are not biodegradable; they accumulate on lands and in river causing ecological problems [7]. Many natural dyes have antimicrobial properties, making them safer for kids in particular [8]. Advantages of natural dyes such as its non-allergic, non-toxic, biodegradation and eco-friendly on textiles makes them suitable for consideration [9]. There are huge applications of natural dye on textile so it is clamorous to promote technology for extraction [10]

Dass *et al.* [11] studied the extraction and testing of natural dye from Dafara (C*issus populnea*) stem bark and is application on cotton fabric. It was observed that the effect of time on dye extraction showed increased intensity of dye as time increased. Ultra Violet-Visible spectrophotometer also showed that as the extraction time increased, the absorption wavelength (nm) also increased from 450 nm to 560 nm. This was ascribed to the high yield of extract and the subsequent evaporation of solvent. The effect of temperature on extraction showed that as the temperature increased from 40 °C to 100 °C, the dye intensity also increased. 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 worked best with a mordant at higher temperatures.

There are thousands of plants and animals which contain colourants that can be used as natural dyes. This present study is therefore focused on the extraction, characterization and application of dye extract from *Baphia nitida* on cotton fabric. The aim of this research work is to extract, characterize and apply the natural dye extract from the of *Baphia nitida* on cotton fabric.

Plate 1. Plant used for the research work *Baphia nitida* (camwood)**.**

MATERIALS AND METHODS

Sample collection and preparation

The heartwood of *Baphia nitida* stem was collected from Agbou, Adi-Etulo Buruku Local Government Area of Benue State, Nigeria. The stem sample was shade-dried and ground into powdered form using mortar and pestle, which was ready for extraction.

Soxhlet extraction

One hundred and fifty grams of the sample was measured using analytical weighing balance. The sample was partitioned into five portions each containing 30 g of the sample. Each portion was put into the thimble and 200 mL of absolute ethanol was added into the round bottom flask fitted into the thimble for extricable. The heating mantle was on and extraction was done for 6 hours on each of the samples and the temperature for extraction was 78.37 ˚C, which is the boiling point of ethanol. Rotary evaporator was used to remove excess solvent leaving the dye in dry state. The percentage yield and colour were determined for the purified dye samples [12].

FT-IR spectroscopy analysis

The dye extract was measured with FTIR spectrophotometer using a Shimadzu IR prestige 21 spectrometers in the wavelength range of 400-4000 cm^{-1}.

GC-MS analysis

The dye extracted was characterized on a Shimadzu GC-2010 connected with MS QP-2010. Column that was used was Restec Rtx-5MS measuring 60 mm × 0.25 mm ID thickness of 0.25

µm and packed with 95% dimethyl polysiloxane. Helium gas was used as carrier gas at a flow rate of 1ml/min and injection volume of 1 µl was utilized.

UV-Visible spectroscopy analysis

The dye extract was introduced in a quartz cell (1 cm pathway) and measured with UV-Vis spectrophotometer. A scan from 200 to 800 nm was performed in order to generate the characteristic absorption spectra of the sample.

Scouring process

The scouring process was carried out using 3M of sodium hydroxide. To each of the beakers labelled A, B and C, 50 ml of the prepared sodium hydroxide was used to carry out the scouring process for 1h at 100 ^{0}C [13].

Bleaching process

To each of the beakers labelled A, B and C, 3M of sodium hypochlorite was measured with measuring cylinder and 100 ml of the prepared solution was used to carry out the bleaching process for 1 hour at 100 °C [14].

Preparation of dye bath

The dye extract from the heartwood of *Baphia nitida* is insoluble in water but soluble in alcohol (ethanol). Therefore, to prepare the dye bath, 1 g of the dye extract, measured into three 250 ml beakers labelled A, B and C, was dissolved in 5 ml ethanol and this was diluted to 55 ml [15].
The ratio of the dye extract to ethanol to water is 1: 5: 50.

Dyeing the cotton fabrics

To the dye bath solutions above, 1g of the scoured-bleached fabrics was introduced. The dye-bath was heated gently, and the fabrics left until they have absorbed the colour. The dyeing process commenced and lasted 1 hour at 100 °C. When the dyeing process was completed, the materials were removed from the dye bath and allowed to air oxidize for 10 min; after which they were rinsed with cold water to remove loose dye particles that adhered to the surface of the dyed materials. The dyed fabrics were then air dried after which the fastness properties were tested [16].

Wash fastness properties

The dyed fabric was first cut (4x2 cm) after which the undyed white fabric was cut (4×2 cm) and were machine-stitched to give one piece of fabric. Exactly 100 ml of water was measured with measuring cylinder, 3 g of detergent (*Viva plus*) was weighed using weighing balance and dissolved in the 100 ml water contained in the beaker. The stitched fabric was introduced into the detergent solution and was stirred vigorously for 10 minutes, then rinsed and dried at room temperature [17].

Rubbing fastness properties

The dyed fabric was first cut (4x2 cm) after which the undyed white fabric was cut (4×2 cm) for both (dry and wet rubbing). For dry rubbing, the undyed white fabric was rubbed against the surface of dyed fabric for 10 minutes. For wet rubbing, the undyed white fabric was first immersed in water (wet) and was rubbed against the dyed fabric for 10 minutes [18].

RESULTS AND DISCUSSIONS

Production of dye from Heartwood of *Baphia nitida*

Plate 2 shows heartwood of *Baphia nitida* stem powder. Sample of pulverized heartwood of *Baphia nitida* stem is used for the dye extract. Soxhlet extraction method was used in the extraction of the dye from heartwood of *Baphia nitida.* Plate 3 shows the extracted dye from heartwood of *Baphia nitida* stem. The result of extraction of dye from the heartwood of *Baphia nitida* as presented in Table 1 showed that 150 g of the sample was used for the extraction. After the extraction, the weight of the sample was reduced to 124.64 g and the amount of dye extracted was 25.36 g. The percentage yield of the dye extract was 16.91% and the colour of the dye extract is red due to santarlins ($C_{15}H_{14}O_5$) and santarrubins ($C_{34}H_{28}O_{10}$) [12].

Plate 2. Sample of pulverized heartwood of Baphia nitida

Plate 3. Dye produced from heartwood of *Baphia nitida*

Table 1. Extraction of dye from *Baphia nitida*

Sample: *Baphia nitida*	**Value (g)**
Sample before extraction	150
Sample after extraction	124.64
Amount of dye extracted(g)	25.36
%yield	16.91
Colour of the dye extract	Red

FTIR Analysis of Dye extract from *Baphia nitida*

The FTIR spectrum of *Baphia nitida* as highlighted in Figure 1 showed abroad band at 3754.67 cm^{-1} due to the presence of O-H stretching vibrations (free hydroxyl). The band at 2933.79 cm^{-1} is assigned to the C-H stretching of alkane groups. In the spectrum of the dye extract, vibrations occurred at band 2620.04 cm^{-1} suggesting the presence of CHO group which is an aldehyde. A weak band at 2200.58 cm^{-1} is an indication of the presence of C≡N stretching vibrations. The band at 2033.72 cm^{-1} is assigned to C≡C stretching vibrations. The strong absorption band at 1745.43 cm^{-1} is assigned to R-CO-OR group and the absorption band at 1464.52 cm^{-1}, assigned to N-O group which confirms the presence of nitro compounds. The band at 1344.76 cm^{-1} is assigned to C-H rocking vibrations, while the absorption band at 1177.27 cm^{-1} is an indication of C-H wagging vibrations (CH_2X) and band at 993.37 cm^{-1} is assigned to =C-H bending vibrations. The absorption

band at 800.15 cm^{-1} and 713.53 cm^{-1} is assigned to C-Cl stretching vibrations and the medium band at 657.85cm^{-1} is an indication of the presence of C-Br stretching vibrations [19].

1

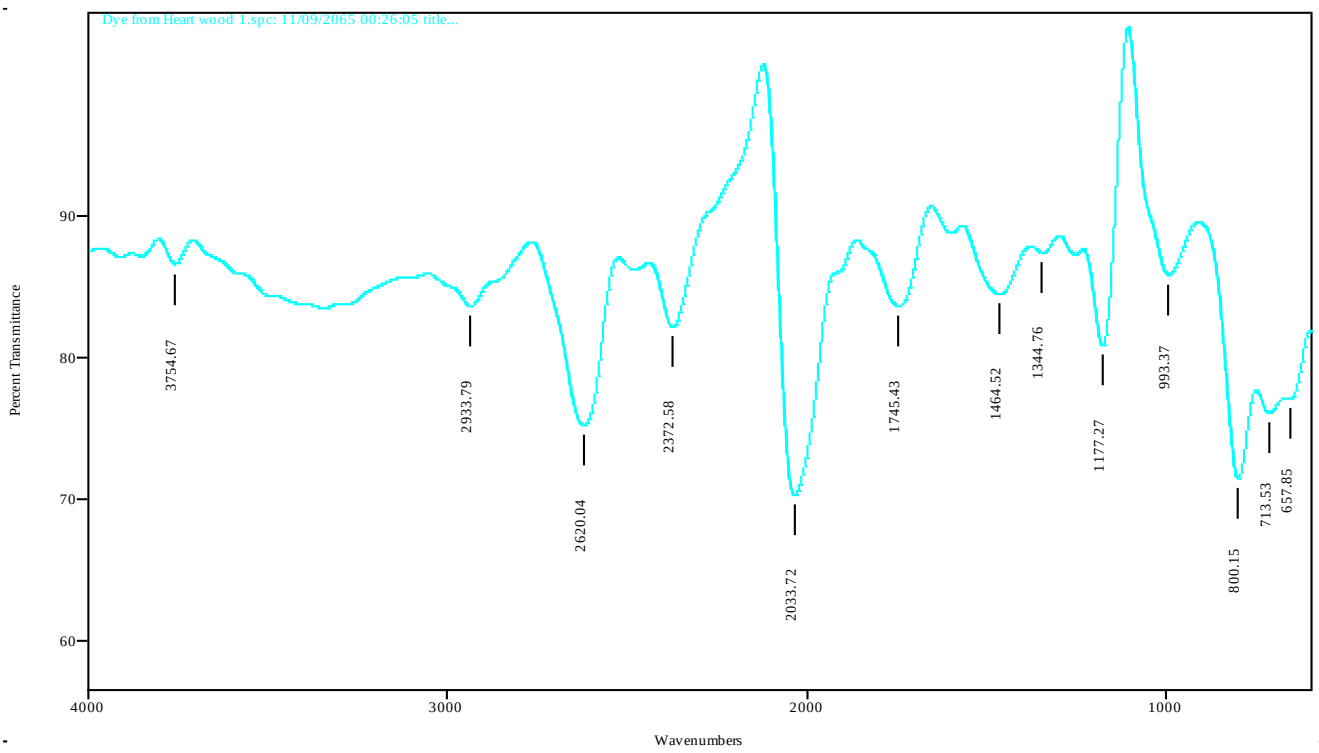

Figure 1: FTIR Spectrum of dye extract from heartwood of *Baphia nitida*

GC-MS analysis of Dye extract from *Baphia nitida*

The dye extracts obtained were subjected to GC-MS analysis for the determination of various volatile and semi volatile compounds in the heartwood of *Baphia nitida.* The GC-MS examination of the dye extract from heartwood of *Baphia nitida* led to the identification of 30 compounds. The retention time (RT), % of peak area, molecular formula, molecular weight and the characterized compounds are listed in Table 2 The prevailing compounds were 1,3,5-Triazine-2,4,6-triamine (0.01%); 5-Hydroxymethylfurfural (2.34%); 4-mercaptophenol (0.12%); 1,12-Bis (2-nitrophenoxy) dodecane (14.67%); 2-Dodecen-1-yl (-) succinic anhydride (0.47%); i-propyl -9-octadecenoate (0.53%); 17-pentatriacontene (3.31%) [14]. Note that, if two or more compounds have similar retention time it does not mean that the two compounds are the same, as several compounds have similar retention time. Also, the interaction between different compounds may also increase or decrease the time they spend inside the column [20].

The combination of GC with MS made GC-MS one of the ideal techniques for quantitative analysis of volatile and semi-volatile compounds [16]. Among the identified compounds of dye extract from the *Baphia nitida,* Benz (a) anthracene, 7,8 - dimethyl, has the highest percentage of (35.09%), 6H - Benzofuro [3, 2 - C] [1] benzopyran, 6a, 11a - dihydro - 3,9 - dimethoxy-, (6a R - cis) (homopterocarpin), 210Indeno [2,1 - C] pyridine, 3,7 - dimethyl - 9 - phenylimino, Coumaran -5 - ol - 3 - one, 2 - [4 -hydroxy - 3 - methoxybenzylidene] (27.99%), 1,12 - Bis (2-nitrophenoxy) dodecane, Cyclohexanepropanol, 2, 2 - dimethyl - 6- methylene and 2 - Dodecen - 1 - yl (-) succinic anhydride (14.67%) which implies that the heartwood of *Baphia nitida* constitute high quantities of these compounds and 1,3,5 - Triazine - 2,4,6 -triamine and 4H - pyran - 4 - one, 5 - hydroxy - 2 - methyl has the least percentage of (0.01%) which also implies very minute quantity of these compounds are present in the Heartwood of *Baphia nitida.*

Table 2: Chemical compositions of the dye extract from *Baphia nitida*

Peak	Retention Time	% of peak Area	Compound identified	Nature of Compound	Molecular Formular	Molecular weight
1	6.324	0.01	1,2,5 - Triazine -2,4,6- triamine	Heterocyclic compound	$C_3H_6N_6$	126
2	6.936	2.34	5 - Methyl - 2 Thiophenecarboxaldehyde	Heterocyclic compound	C_6H_6OS	126
3	7.669	0.08	5-Hydroxymethylfurfural	Heterocyclic compound	$C_6H_6O_3$	126
4	8.344	1.47	Benzene, 1,2,3 - trimethoxy- 5 - (2 - propenyl)-	Organic compound	$C_{12}H_{16}O_3$	208
	8.344	1.47	Benzenamide, N - cyano	Heterocyclic compound	$C_{11}H_{12}N_2O_4$	236
5	8.933	0.12	Furazanamine, 4 -azido-	Heterocyclic compound	C_2H_3NO	126
	8.933	0.12	Benzenemethanol, 3- fluoro	Organic compound	C_7H_7FO	126
6	9.620	0.18	Tetradecanoic acid, 12- methy l-, methyl ester	Fatty acid methyl ester	$C_{16}H_{32}O_2$	256
	9.620	0.18	Hexadecanoic acid, methyl ester	Fatty acid methyl ester	$C_{17}H_{34}O_2$	270
7	10.598	2.25	Methyl 5,9- Dimethyldecanoate	Fatty acid methyl ester	$C_{13}H_{26}O_2$	214
	10.598	2.25	Methyl 13- methyl Tetradecanoate	Fatty acid methyl ester	$C_{16}H_{13}O_2$	256
	10.598	2.25	N - [5 - Nitrosalicylidene] - d – cycloserine	Amino acid	$C_{10}H_9N_3O_5$	251

8	12.258	12.258	1,12 - Bis (2-nitrophenoxy) Dodecane	Organic compound	$C_{24}H_{32}N_2O_6$	444.5
	12.258	14.67	Cyclohexanepropanol, 2, 2-dimethyl - 6- methylene-	Organic compound	$C_{12}H_{22}O$	182
	12.258	14.67	2 - Dodecen - 1 - yl (-) succinic anhydride	Heterocyclic compound	$C_{16}H_{26}O_3$	266
9	12.429	4.36	Bis (2 - ethylhexyl) Phthalate	Organic compound	$C_{24}H_{38}O_4$	390
	12.429	4.36	Phthalic acid, 3- chlorobenzyl nonyl ester	Fatty acid ester	$C_{24}H_{29}ClO_4$	416.5
	12.429	4.36	Aspidofractinine-3-methanol(2.alpha.,3.beta.,5.alpha.)-	Heterocyclic compound	$C_{20}H_{26}N_2O$	310
10	12.950	27.99	6H - Benzofuro [3, 2 - C] [1] benzopyran, 6a, 11a- dihydro - 3,9 - dimethoxy-,(6a R - cis)-	Heterocyclic compound	$C_{17}H_{16}O_4$	284
	12.950	27.99	Indeno [2,1 - C] pyridine, 3,7 - dimethyl - 9- phenylimino-	Heterocyclic compound	$C_{20}H_{16}N_2$	284
	12.950	27.99	Coumaran -5 - ol - 3 - one, 2 - [4 - hydroxy - 3- methoxybenzylidene]-	Heterocyclic compound	$C_{16}H_{12}O_5$	284
11	13.803	5.90	4H - 1 - Benzopyran - 4- one, 2 - (3,4 – dimethoxy phenyl) - 7 – hydroxyl	Heterocyclic compound	$C_{17}H_{14}O_5$	298
	13.803	5.90	5 - pregnen - 3. beta. - ol-20 - one, trifluoroacetate	Organic compound	$C_{23}H_{31}F_3O_3$	412
12	14.049	35.09	Benz (a) anthracene, 7,8- dimethyl-	Aromatic compound	$C_{20}H_{16}$	256
13	16.109	1.21	i - propyl 11,12 -methylene-octadecenoate	Organic compound	$C_{22}H_{42}O_2$	338.6
	16.109	1.21	Ergosta - 4, 7, 22 -trien -3- one	Organic compound	$C_{28}H_{42}O$	394.6
	16.109	1.21	Tetracosapentaene, 2,6,10,15,19,23- hexamethyl-	Organic compound	$C_{30}H_{60}$	420.8
14	16.269	3.31	26, 27 - Dinorergosta- 5, 23 - dien-3 - ol, (3.beta.)-	Sterol	$C_{26}H_{42}O$	370
15	16.784	0.47	17 -pentatriacontene	Organic compound	$C_{35}H_{70}$	490.9
16	16.875	0.53	i - propyl 9 - octadecenoate	Organic compound	$C_{21}H_{40}O_2$	324.5

UV-Visible Spectrum of dye extract from *Baphia nitida*

The UV-visible of dye extract from heartwood of *Baphia nitida* as presented in Figure 2 appeared in the maximum wavelength of 277nm with the absorbance of 0.4592. These is attributed to $n \rightarrow \pi$. This suggests that the chromophores N=O and C=O may be present. [21].

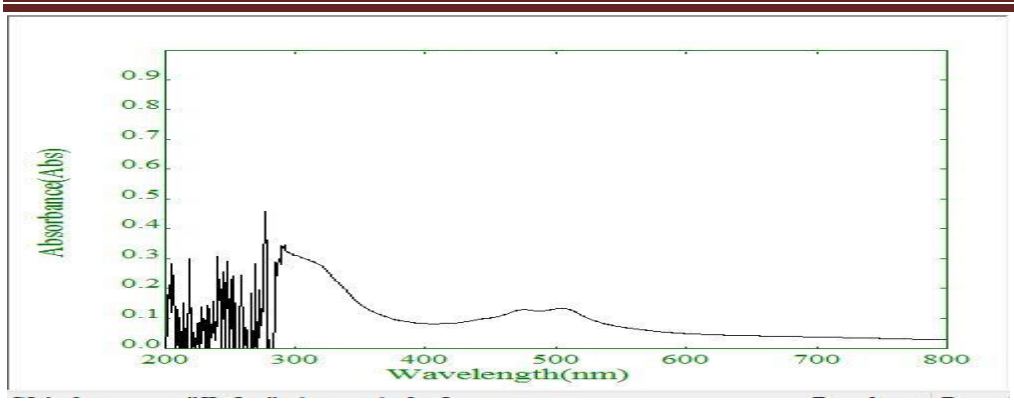

Figure 2: UV-Visible spectrum of *Baphia nitida*

Scouring and bleaching of cotton fabric

The scouring and bleaching of cotton fabrics as presented in Figure 3 showed that three cotton fabrics with different weights were scoured with sodium hydroxide (NaOH) and bleached with sodium hypochlorite (NaClO). The results showed that there is a progressive decrease in the weight of fabric after scouring and bleaching due to removal of natural wax and non-fibrous impurities, making the fabric white and brighter, the fabric and the weight is reduced [22].

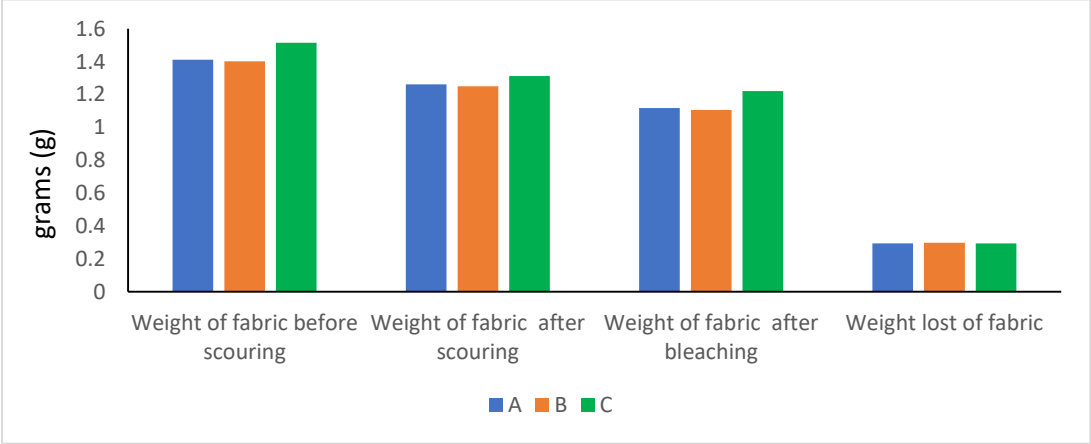

Figure 3: Scouring and bleaching of cotton fabric.

Dyeing of cotton fabric with the produced dye

The results of dyeing the cotton fabrics as presented in Figure 4 showed a progressive increase in the weight of the three different fabrics. This is due to increase in absorbency power of the fabric and this increases the dye affinity for the fabrics [23].

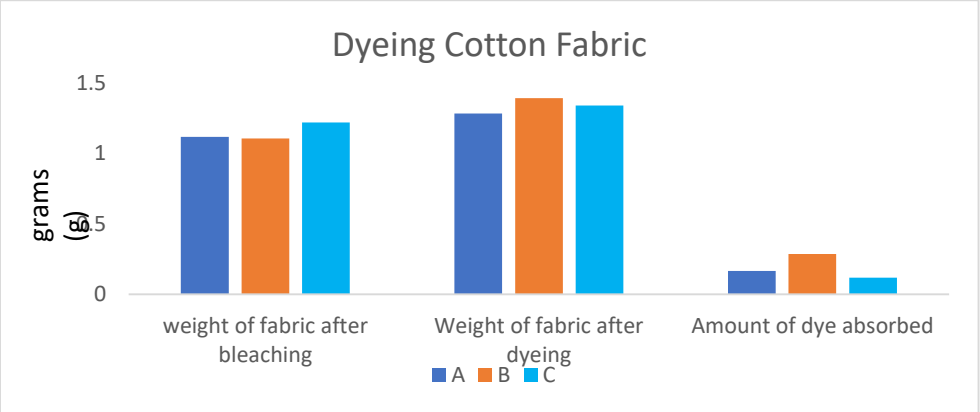

Figure 4: Dyeing of cotton fabric

Wash fastness properties of dyed fabric

The results of wash fastness properties of dye extract from *Baphia nitida* on cotton fabric as presented in Table 3 showed that when cotton fabric was dyed without mordants, a fastness grade of 2-3 was experienced which indicates poor to fair fastness.

The undyed fabric experienced a staining grade of 4 which indicates light staining [13].

Table 3: Wash fastness properties of dyed fabric

Fabric type	Colour change	Colour staining
Dyed fabric	2-3	4

Wash fastness:1- very poor; 2- poor 3-fair 4- good 5-excellent

Staining of adjacent white fabric: 1- deep staining; 2- significance staining; 3- moderate staining; 4- very light staining; 5- no staining

Rubbing fastness properties of dyed fabric

The result of rubbing fastness properties of cotton dyed sample with *Baphia nitida* dye extract as presented in Table 4. It showed that dyeing the fabric without a mordant (both dry and wet condition) gave rise to a rubbing fastness grade of 3-4 which indicates fair to good for dry rubbing and rubbing fastness grade of 2-3 which indicates poor to fair for wet rubbing fastness grade [17, 24].

Table 4: Rubbing fastness properties of dyed fabric

Fabric Type	Dry rubbing.	Wet rubbing
Dyed fabric	3-4	2-3

Rubbing fastness; 1- very poor; 2- poor: 3- fair; 4- good; 5-excellent

CONCLUSIONS

Natural colourant was successfully extracted from *Baphia nitida* using soxhlet extractor with ethanol as a solvent. The dye extracts were applied on cotton fabric without mordants. The results obtained in this study suggest that the red colour dye extracted from *Baphia nitida* possesses intrinsic affinity for cotton fabric. The observed affinity of the dye extracts for the textile substrates used for the study may be due to the presence of santalins and santarubins compounds. The FTIR characterization of the dye extract also suggested the presence of OH, CH, C≡N, C≡C, C=O, N=O, C=C, C-Cl and C-Br functional groups in the dye components. The chromophores in the dye extract were N=O and C=O.

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