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Extraction and characterization of guinea corn plant extract for mordant assisted dyed cotton fabric

Mariam Taiwo Oloye-Akorede [1, *], Jacob Olalekan Arawande [2], Emeka Henry Obinwa [1], Jamiu Mosebolatan Jabar [3], Yekeen Olagunju Oderemi [4], David Adeniran Oyegoke [5] and Izzah Opeyemi Quadri [6]

[1] *Department of Chemistry, University of Medical Sciences, Ondo-City, P.M.B. 536, Ondo-State, Nigeria.*
[2] *Department of Science Laboratory Technology, University of Medical Sciences, P.M.B. 536, Ondo-City, Ondo-State, Nigeria.*
[3] *Department of Chemistry, Federal University of Technology Akure, Ondo-State, Nigeria.*
[4] *Department of Science, Laboratory Technology, Polytechnic Igbajo, Osun- State, Nigeria.*
[5] *Department of Chemical Sciences, Achievers University, Owo, Ondo-State, Nigeria.*
[6] *Department of Chemistry, University of Ilorin, Kwara-State, Nigeria.*

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Abstract

Synthetic dyes have been discovered to be harmful to the human body and also to the environment as they are non-biodegradable. Hence, recent researches and discoveries on the use of natural plants such as Guinea corn leaves (*Sorghum bicolour*) as colourants in the textile industry. Dye from *Sorghum bicolour* was extracted using both distilled water and ethanol (90:10 v/v) and (10:90 v/v) respectively. Dye standardization was done using UV-Visble spectrophotometer in the range between 375 nm to 800 nm. The wavelength of maximum absorbance was gotten to be 575 nm and used in determining the absorbance at various concentrations (100, 200, 300, 400, 500, 600, and 700 mg/L).

The physico-chemical properties such as pH level, dye colour, percentage yield and solubility of the aqueous extracted dye was found to be 6.86, reddish brown in colour, 15.66% and dissolves freely in warm water while that of the ethanolic extracted dye was found to be 6.68, wine in colour, 21.48% and partially soluble in warm water. Characterization of the dye was done using Fourier Transform Infrared Spectroscopy, Ultraviolet-Visible Spectroscopy, Scanning Electron Microscope, Brunauer-Emmett-Teller's theory, Thermogravimetric Analysis and Differential Thermal Analysis. The extracted dye was then used in dyeing pre and post mordant white cotton fabric using both alum and magnesium sulphate as mordants fabrics which improve the colour shades and fastness properties of the dyes thereby enhancing their aesthetic value.

Keywords: Colour; Extraction; Guinea Corn; pH; Dye; Fabric

1. Introduction

Natural dyes have been used as colourants for textile materials before the discovery of synthetic dyes. The recent discovery of the toxic nature of synthetic dyes and their harmful effects to human has renewed interest in the production, application, exploration, experimentation and use of natural dyes [1]. Sorghum (Sorghum bicolour) is an African crop known to be used as colourants in food and medicinal industries. The use of synthetic dye was so intense due to global industrialization and the obvious advantages of having brilliant and numerous colours not found in the colours from the nature among others. These conditions almost shelved off natural dyes to the verge of extinction [2].

* Corresponding author: Mariam Taiwo, Oloye-Akorede

Natural dyes extracted from plants, fruits and flower contain pigments such as anthocyanin, chlorophyll, betalains and many more which could interact with the wavelengths of visible light to either being reflected or transmitted by plant tissues. These dyes are derived from natural sources of plants-barks, roots, leaves and flowers, insect secretions and minerals. The most explored is plant, and *Sorghum bicolour* is one of such plants. *Sorghum bicolour* (guinea corn) has a red colour natural dye derived from sorghum shell [3]. Natural dyes can exhibit better biodegradability and generally possess higher compatibility with the environment compared to synthetic dyes [4, 5]. Round the universe, efforts are tailored toward promoting the cultivation of natural dye plants and their application in dyeing [6]. This has been possible because they are eco-friendly compared to synthetic dyes. Synthetic dyes are synthesized from petrochemical sources through hazardous chemical processes which pose a threat to the environment [3]. Researches have shown that synthetic dyes are suspected to release harmful chemicals that are allergic, carcinogenic and detrimental to human health [6]. Sorghum bicolour (guinea corn) is so named due to the inherent colours possessed by the plant. The reddish brown colour is physically seen on every part of the plant and these were used for the extraction except the seeds that is grown for food. This feature seem to be an evidence that sorghum corn is high in tannin [7]. The colour was extracted by boiling and the quantity to be boiled and water depend wholly on the amount of cloth to be dyed. The fact that the traditional practice of dyeing items with natural plant dyes is fast going extinct is the attraction to this experiment. To this fact, there is need to optimize the liquid extract of *Sorghum bicolour*, analyze its potentials as fabric dye and carry out dye characterization for its UV level of observance for easy handling and usage [13]. Natural dyes are renewable, environmental friendly, biodegradable and less allergic. They are abound in all parts of the plant when compared with other natural sources; the leaves are sustainable and provide the most yields of plant derived dyes [8]. Widespread uses of synthetic dyes have been associated with huge environmental issues with attendant health hazards as these chemicals are now known to have mutagenic, carcinogenic and other toxic effects on the global community [9, 10].

The chemical mordant agents, such as salts of Fe, Al, Co, Ni, Cu, and Sn are often used for enhancing shade, dye exhaustion and fastness properties of natural dyed textile substrates [11]. Environmental awareness has condemned the use of many of these electrolytes in mordant of natural dyed fabric due to their hazardous nature [8].

Cotton is most popular among the cellulosic fibres with consumers' desired properties, such as good moisture absorptivity, mechanical properties and dye ability. The dye ability of cotton fibers is facilitated through H–bond formation between natural dye molecules and amorphous region of the cotton polymer chain [12].

The aim of this study was to extract and characterize Guinea Corn plant extract for mordant assisted dyed fabric.

2. Materials and methods

2.1. Materials

The dried guinea corn leaves was purchased from Sokoti market, Ondo West local government area of Ondo State, Nigeria.

2.2. Methods

2.2.1. Preparation of Sorghum plant

The plant leaves were washed to remove dirt, cut into smaller pieces and placed in an oven at a temperature of 85 °C till it was fully dried. The dried samples were then crushed and ground into powder using a clean and dry electric grinding machine [8].

2.2.2. Dye Extraction

50 g of the ground sample was weighed into a beaker using an electronic balance and a mixture of ethanol and water (90:10 v/v) and vice versa were added to the beakers containing the plant sample at a liquor ratio of 1:10 and allowed to soak for thirty minutes while stirring intermittently using a glass rod [13]. The beakers containing soaked plant material were then heated on water bath (HH-6, PEC Medical USA) at 95 °C for 3 hrs, while stirring intermittently. After extraction period of 3 hrs, the extracted dye solutions were filtered off using a 75 nm mesh size sieve. The filtrates obtained were evaporated to dryness on a water bath at 97 °C leaving off the crude dye extracts. The crude dye extracts were then placed in an oven at a temperature of 65 °C for two hours to completely dry off the solvent mixtures [8].

2.2.3. pH Measurement

0.5g of the extracted dye was measured and dissolved in 100 ml of distilled water. The solution was stirred vigorously using a glass rod till the dye samples dissolved totally. A pH meter was inserted into the dye solution and the pH reading was recorded.

2.2.4. Solubility Determination

0.5 g of the extracted dye was measured and placed in a beaker. 100 ml of warm distilled water was poured into the beaker containing the dye sample and was shaking vigorously till the dye samples dissolved almost completely. The solubility was determined by checking if the crude dye extracts were completely dissolved in the warm distilled water with no residue present underneath the beaker. [8].

2.2.5. Percentage yield of dye

The percentage yield of dye was determined by evaporating the filtrate of the dye solution to dryness and then weighed.

2.2.6. UV/Visible Absorption Measurement

The absorbance was read from 375 nm to 800 nm and the wavelength of maximum absorbance was taken. Wavelength of maximum absorption obtained was used in measuring absorbance of the dye solution of various concentrations (100, 200, 300, 400 and 500 mg/L) [13].

2.2.7. FT-IR Analysis

The dye sample was crushed into a powder of 2 to 5 mg, it was mixed with potassium bromide in a mixing ratio of 1 to 100. Then, the mixed powder was pressed in a die at a load of 10 tons to form a pellet of 13 mm. The pellet was then inserted in the FT-IR chamber for analysis. The spectra were recorded within the frequency range of 4000 cm^{-1} to 500 cm^{-1} using an FT-IR spectrometer (Infrared spectrometer Varian 660 Mid IR Dual MCT/DTGS Bundle with ATR) with a detector at 4 cm^{-1} resolution and 200 scans per sample [10].

2.2.8. BET Analysis

0.3 g of the sample was weighed and loaded in to the BET glass sample tube; the weight of the tube before and after loading was recorded. The samples were degassed at 473 K for 3 hrs by connecting the tube to micromeritics flow prep 067 linked with Nitrogen gas to remove physically adsorbed water molecules. Degassed sample was reweighed and the analysis was carried out in micromeritics Tristar 3000 V4.02 under liquid nitrogen temperature.

2.2.9. TGA-DTA Analysis

TGA analysis provides information about physical phenomena, such as phase transitions, absorption, adsorption and desorption; as well as chemical phenomena including chemisorption, thermal decomposition, and solid-gas reactions. DTA curve provides data on the transformations that have occurred, such as glass transitions, crystallization, melting and sublimation. The samples were weighed and placed into the platinum cups and sealed. The temperature ranges from 0 – 900 ^{o}C under nitrogen atmosphere at a heating rate of 10 ^{o}C/min [14].

2.2.10. SEM Analysis

SEM analysis was done to determine the surface morphology of the extracted dyes. Powder samples of the extracted dyes were prepared for SEM analysis with a thin coating of colloidal carbon for electron conductivity. The surface morphological features of the samples were studied and revealed with a scanning electron microscope [14].

2.2.11. Mordant for Experiment

The mordant used for this experiment were 0.5 g each of magnesium sulphate ($MgSO_4$) and potash alum ($KAl(SO_4)_2 \cdot 12H_2O$). They were considered to be friendly to the environment [8].

2.2.12. Fabric for the experiment

White cotton fabrics were washed with clean water to remove impurities and were desized. The cotton fabric was cut into eight smaller pieces of 12 cm by 6 cm [8].

2.2.13. Preparation of dye solution

0.6 g of the dye was measured and diluted with 100 ml distilled water in a beaker. The solution was stirred continuously for thirty minutes till all the crude dye dissolved completely in the solvent [8].

2.2.14. Dyeing experiment

Pre mordant dyeing

The pre mordant dyeing was carried out by application of the mordant solution onto the fabric before the actual dyeing process.

Two pieces of 12 cm by 6 cm cotton fabric were placed each in the separate mordant solutions of potash alum and magnesium sulphate and stirred continuously for thirty minutes for even penetration of the mordant into the fabrics. The mordant fabrics were removed and placed in a separate aqueous and ethanolic dye solutions for thirty minutes with continuous stirring before drying.

Post mordant dyeing

Post mordant dyeing was carried out by application of the mordant solution to the dyed cotton fabric.

Two pieces of 12 cm by 6 cm cotton fabric were placed each in the separate aqueous and ethanolic dye extracts solutions and stirred continuously for thirty minutes. The dyed cotton fabrics were then removed and placed into the separate mordant solutions of potash alum and magnesium sulphate, stirred continuously for thirty minutes and allowed to dry [8].

2.2.15. Fastness properties of dyed fabrics

Light Fastness

Two pieces of 12 cm by 6 cm cotton fabric of the dyed cotton fabric, pre and post mordant were prepared. The fabric was exposed to sunlight for seven days, and was rated using a grey scale [6].

Wash Fastness

Two sets of 12 cm by 6 cm cotton fabric of the dyed cotton fabric, pre and post mordant were prepared. One set was washed/treated with soap solutions for 30 min. in a test wash machine according to ISO washing test number 3 [6].

3. Results and discussions

Table 1 shows the physico-chemical properties of the dye extract from *Sorghum bicolour* leaves**.** The ethanolic extract produced a wine solution with pH value of 6.68 which indicates a slight acidic condition. The aqueous extract produced a reddish brown solution with a pH value of 6.86 which indicates a slight acidic condition. The ethanolic extraction yield 21.48% of dye while that of aqueous extraction produced 15.66% of dye, an indication that heating at saturation temperature is a good method in extraction of natural dye. The ethanolic dye extract was found to be slightly soluble in warm distilled water which supports the fact that dyes are at least 70 percent soluble in ethanol. The aqueous dye extract was found to be readily dissolved in distilled water when heated and stirred continuously.

Table 1 Physicochemical Properties of the Extracted Dyes

Dye extracts	**Dye colour**	**Percent yield of dye (%)**	**pH value of dye**	**Solubility**
Ethanolic dye extract	Wine	21.48	6.68	Partially dissolves in warm water
Aqueous dye extract	Reddish brown	15.66	6.86	Dissolves freely in warm water

The absorption spectral analysis in Table 2 shows the absorbance range at various wavelengths of the aqueous dye extract using a UV/Visible spectrophotometer ranging from 375 nm to 800 nm. The wavelength of maximum absorbance was found to be 575 nm at an absorbance range of 2.322.

Table 2 UV/visible absorption spectral analysis

Wavelength (λ_{max} nm)	**Absorbance**
375	0.224
400	0.431
425	0.742
450	0.522
475	0.846
500	1.129
525	1.564
550	1.942
575	2.322
600	1.862
625	1.468
650	1.112
675	0.692
700	0.420
725	0.611
750	0.774
775	0.522
800	0.331

The Scanning electron micrograph image **(**SEM) Figure 1(a) of the ethanolic dye crude extract. It can be seen from the micrograph that the pore surface is closely packed. The pore spaces are reduced as compared to the water extracts dye which leads to a smaller pore diameter. It is deduced that there should be a reduced pore surface area as compared to water extracts of crude dye since the micrograph depicts a closer packed cluster.

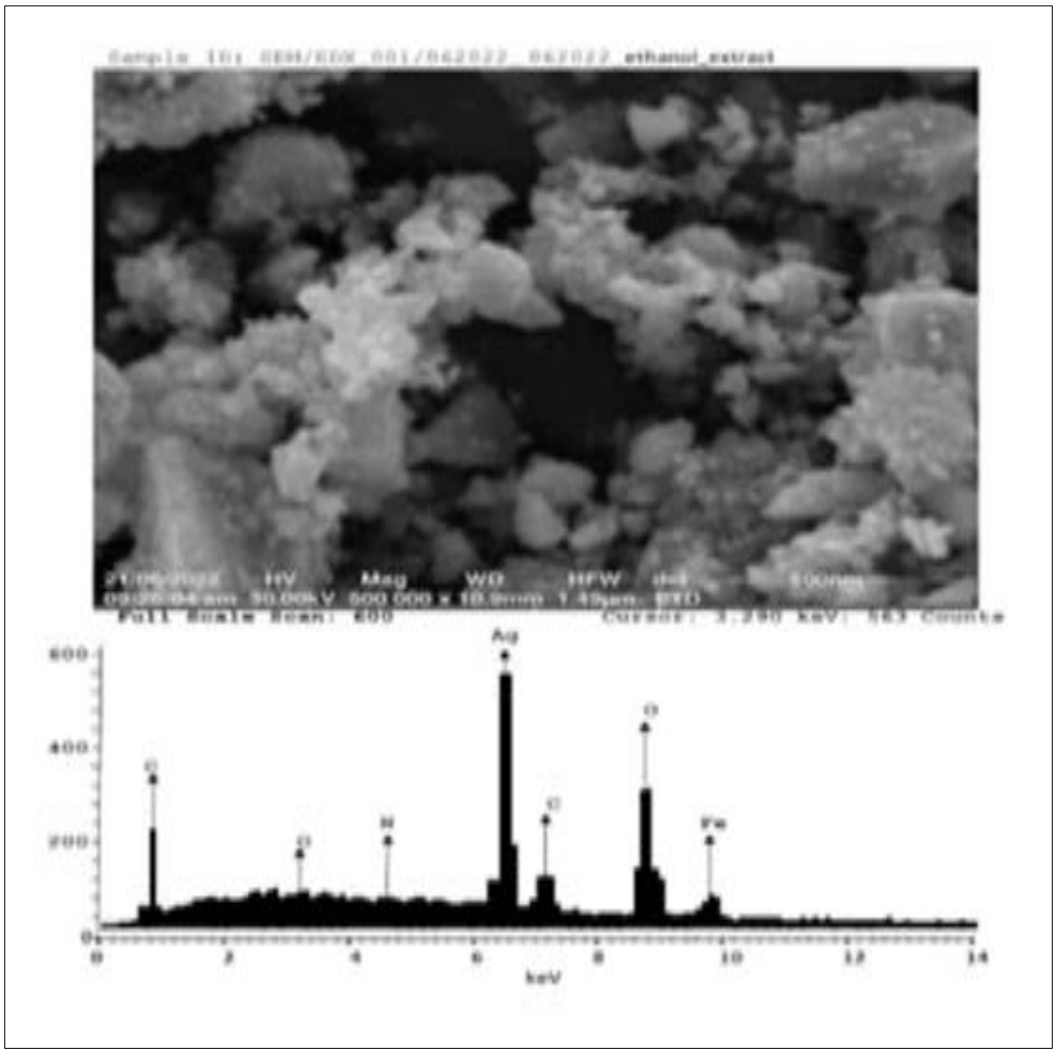

Figure 1(a) SEM/EDX Micrograph of ethanolic dye extract

The Scanning electron micrograph image **(**SEM) Figure 1(b) is the aqueous dye extracts. From the micrograph below, the pore particles are found to be loosely packed. Hence, an increased pore diameter as compared to ethanolic dye extracts and also an increased pore surface area. In the micrograph, there are more particle sizes as compared to ethanolic dye extracts.

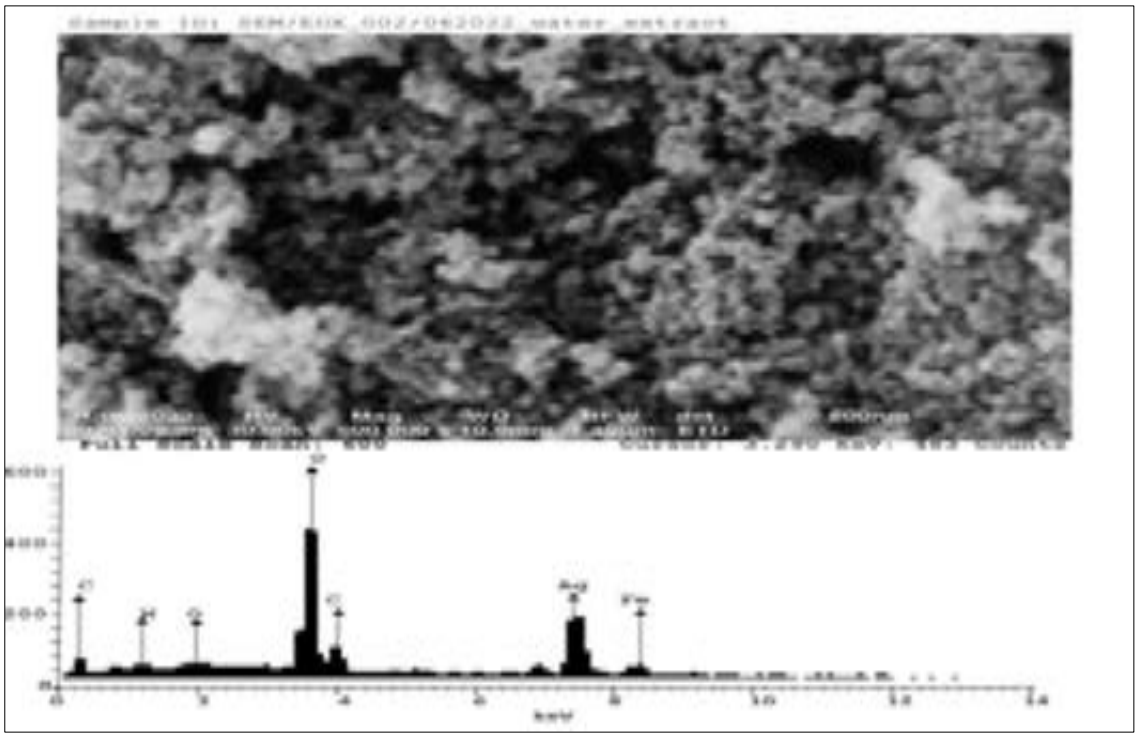

Figure 1(b) SEM/EDX Micrograph of aqueous dye extract

The elemental composition of the ethanolic and aqueous dye extract are shown in Table 3(a) and (b). Silver (Ag) has the composition of 42.92, 26.12 %, this is due to the microcidal properties of guinea corn leaves to the soil. The presence of iron (Fe), 2.67, 1.51% supports the fact that *Sorghum bicolour* leaves are rich sources of iron used in supplementing children meals for body development and growth. The presence of Carbon (C), 20.05, 38.00%, Oxygen (O) 30.34, 33.29% and Nitrogen (N) 3.27, 1.07% shows that the extracted dye is an organic compound.

Table 3 (a) Elemental composition of ethanolic dye extracts

Element	Composition (%)
Carbon	20.05
Oxygen (O)	30.34
Iron (Fe)	2.67
Nitrogen (N)	3.27
Silver (Ag)	42.92

Table 3 (b) Elemental composition of aqueous extracts

Element	Composition (%)
Carbon	38.00
Oxygen (O)	33.29
Iron (Fe)	1.51
Nitrogen (N)	1.07
Silver (Ag)	26.12

Table 4 (a) BET Result Analysis

Methods	Surface parameters		
	Sample weight (g)	**Saturated pressure (kPa)**	**Degassing Time (hr)**
N_2 Adsorption-desorption	0.30	95.00	3.00

Table 4 (b) BET Result Analysis

Samples	**Surface area ($m^2 g^{-1}$)**	**Pore volume ($cm^3 g^{-1}$)**	**Pore diameter (nm)**	**Average particle size (nm)**
Ethanol extracts	0.8154	0.1059	2.368	14.72
Water extracts	0.8830	0.0962	2.372	16.40

Table 4 (a, b) gives information on the particle pore sizes of both sample extracts. The surface area of the aqueous extract is much more larger than that of the ethanolic extract as the SEM micrograph depicts that the ethanolic extracts has closer and packed pore spaces hence smaller surface area as well as pore diameter and average particle size when compared to aqueous extract.

Table 5 (a) Colour and fastness properties of pre and post mordant dyed fabrics in aqueous

S. N	**Dye extract in Aqueous**	**Colour**	**Light fastness**	**Washing fastness**
1	Unmordant	Reddish Wine	1-2	1-2
2	Alum Pre-mordant $(KAlSO_4)_2 12.H_2O$	Dark red wine	3-4	4-5
3	Alum Post-mordant $(KAlSO_4)_2 12.H_2O$	Dark red-wine	3-4	3-4
4	$MgSO_4$ Pre-mordant	Blush wine	4-5	3-4
5	$MgSO_4$ Post-mordant	Blush wine	4-5	3-4

Note: 1-2 most colour change; 2-3 colour change; 3-4 slight colour change; 4-5 colour retained

Table 5 (b) Colour and fastness properties of pre and post mordant dyed fabrics in ethanol

S. N	**Dye extract in Ethanol**	**Colour**	**Light fastness**	**Washing fastness**
1	Unmordant	Reddish Wine	1-2	2-3
2	Alum Pre-mordant $(KAlSO_4)_2 12.H_2O$	Dark red wine	4-5	3-4
3	Alum Post-mordant $(KAlSO_4)_2 12.H_2O$	Dark red wine	4-5	3-4
4	$MgSO_4$ Pre-mordant	Blush wine	3-4	3-4
5	$MgSO_4$ Post-mordant	Blush wine	3-4	3-4

Note: 1-2 most colour change; 2-3 colour change; 3-4 slight colour change; 4-5 colour retained

It was observed in table 5 (a, b), various colour shades with different pre and post mordants, alum and $MgSO_4$ in the dyed cotton fabrics. The unmordant fabric showed a poor performance with light and wash fastness properties which may be due to the characteristics nature of natural dyes compared to synthetic dyes. The mordant fabrics both pre and post gave a better light and wash fastness properties in comparison to the unmordant fabrics [4, 6], [5].

3.1. FTIR result analysis

The various peak wave numbers of the functional group of the ethanolic dye extracts is shown in Figure 2(a). At peak 3300, it depicts O–H stretching vibration in hydroxyl group. At peak 2906, O–H stretching vibration in hydroxyl group is confirmed. Peak 2805 depicts –NH_2 stretching vibrations of amine. At peak 1226, C-C stretching vibration in aromatic group, and in an alkene. At peak 947, C-O stretching vibrations which represent the esters group. Peak 892, C=C stretching vibration due to aromatic ring deformation, an alkene. At peak 700, C-H bending vibration in alkene. Wavelength below 1000 cm shows the fingerprint regions in the spectrum.

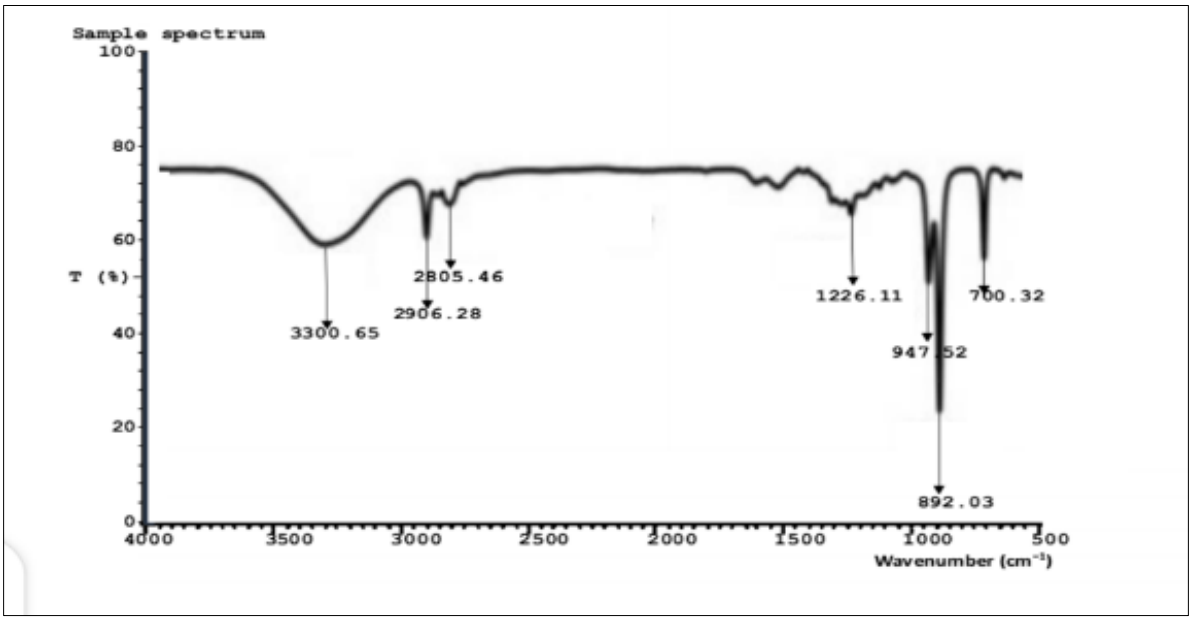

Figure 2(a) FT-IR Result Analysis of Ethanolic Dye Extract

Figure 2(b) shows the various peak wave number of the functional group of the aqueous dye extracts. At peak 3319, O–H stretching vibration which is the hydroxyl group of water. At peak 1499, –NH_2 stretching vibrations of amine. At peak 1321, C-O-C symmetric stretching mode of vibration representing the carbonyl group. At peak 1074, C-O stretching vibrations which represent the ester group.

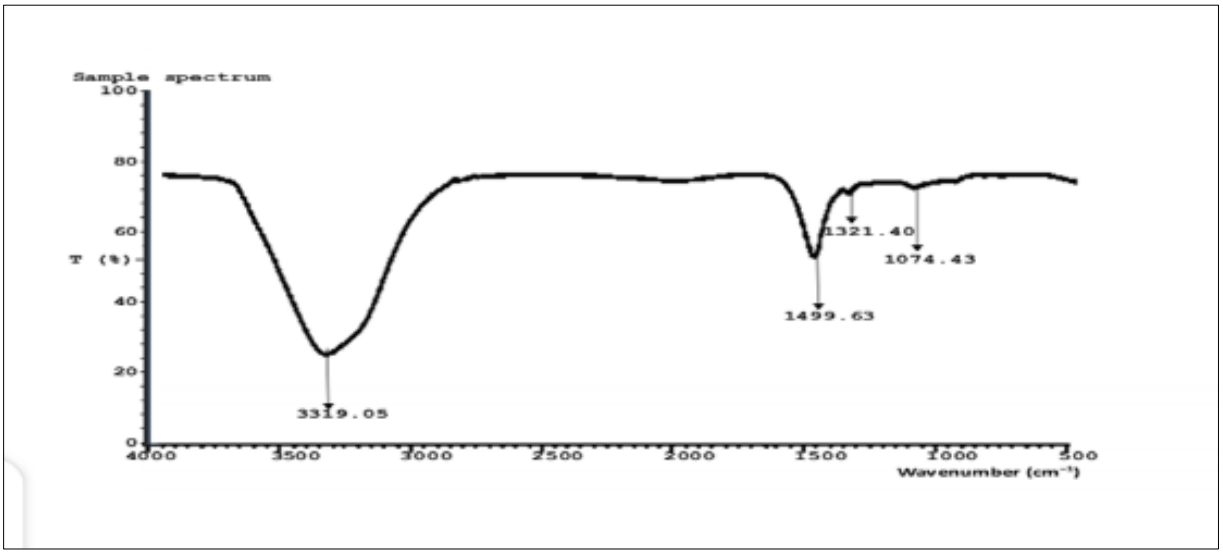

Figure 2(b) FT-IR Result Analysis of Ethanolic Dye Extract

The rate of decomposition of the sample when subjected to temperature for the ethanolic dye extracts is in figure 3. As the temperature increases, the sample mass decreases. DTA determines endo- and exothermic event temperatures, and

shows phase transitions. The DTa sharp peak shows it is melting at that temperature. At 800 °C, the sample was found to be thermally stable. This shows that the dye samples have the ability to withstand high temperature.

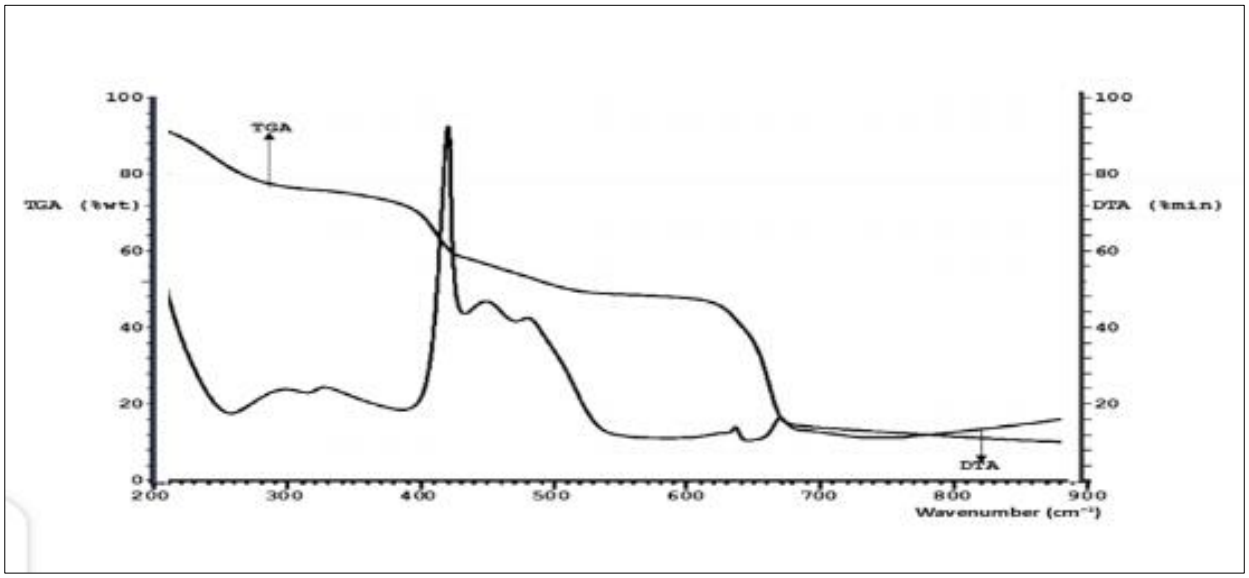

Figure 3(a) TGA - DTA Result of Ethanolic Dye Extracts

Thermogravimetric analysis in Figure 3(b) **s**hows the analysis result for aqueous dye extract. For endothermic reaction, ΔT is positive (sample temperature > reference temperature. For exothermic reaction, ΔT is negative (sample temperature < reference temperature. The sample reaction is exothermic.

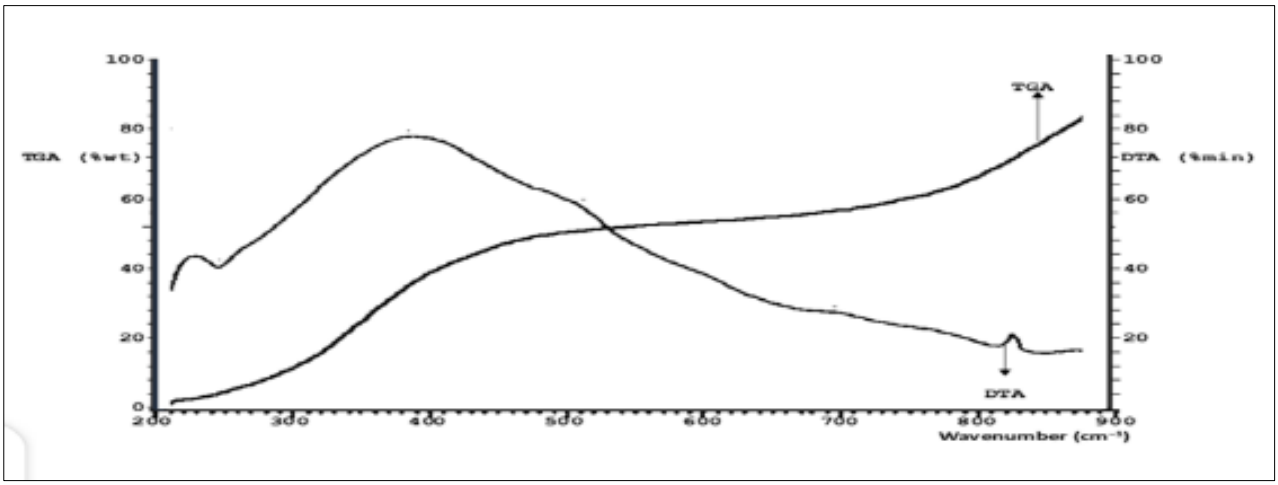

Figure 3(b) TGA- DTA Result of Aqueous Dye Extracts

4. Conclusions

This research study shows that ethanol is a more proper and suitable solvent than aqueous extraction this is because it produces more yield of dye. The extracted dye samples were found to be slightly acidic with almost being neutral at pH of 7 hence will have no effect on either acidic or basic substrates. The extracted dye samples contain vital minerals that are very useful for the body system if applied or used as food supplement. These plant dyestuffs are hereby recommended for use as colouration in foods, drinks and cosmetics industries.

The higher wave length of absorption is called wavelength of maximum absorption, which is seen at 575 nm at an absorbance range of 2.322. The presence of substituent in the dye confers water solubility and enhances colour formation on the dye which makes it applicable for use in staining techniques in the laboratory to enhance contrast in the microscopic image, as antiseptics and germicides, staining tissues and microorganisms and as stimulants of

epithelial growth. The application of mordants in fixing dyes into the fabrics improve the colour shades and fastness properties of the dyes thereby enhancing their aesthetic value.

BET and SEM studies have shown that water extracted dye have loose pore particles and easily dissolved in water for dyeing than ethanolic dye extracts. It also revealed that alum salts and Magnesium sulphate mordants are good for fixing natural dyes on natural fibers especially cotton, wool and in fire proofing fabrics. TGA studies proved that the extracted dye samples have the ability to withstand heat at high temperature and therefore will be suitable for industrial application without been degraded.

Compliance with ethical standards

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Disclosure of conflict of interest

No competing interests was declared.

Authors' contributions

Akorede-Oloye, M. T., Arawande, J.O design the research work. Obinwa, H. E. carried out the sampling procedure, research analysis, Quadri, I. O. did the data analysis. Oderemi, Y.O., and Oyegoke, D. A. did the interpretation of TGA and SEM analysis. All authors read, correct and approved the manuscript. Final version of the manuscript was revised and corrected by Jabar, J.M.

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