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EXTRACTION, PHYSICAL CHARRACTERIZATION AND APPLICATION OF A NATURAL DYE FROM SORGHUM BICOLOUR SHEATHS ON COTTON FABRIC

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Sorghum Bicolour, Extraction, Application

Abstract

The physical parameter of a methanol dye extract of sorghum bicolour sheaths was analyzed by using standard method. A 24% yield was obtained with a melting point of 246 ^{0}C. The colour was maroon, and was found to be ionic in nature as seen in its inability to dissolve in nonpolar solvent. The λmax was found to be 430 nm. It is acidic in nature with a pH value of (4.47), and the crude extract was used to dye 100 % cotton in three dye-baths of 3 %, 4 % and 5 %.Percentage exhaustions of 67.8 %, 50.1 % and 25.1 % were observed on the dye-baths respectively. It has a moderate fastness and excellent stain fastness ability and therefore can be developed as an agent.

Natural Dye

Today, in the world of growing environmental consciousness, natural colourants have attracted the attention of everyone. Natural dyes have become a part of human life since time immemorial for example the alchemy of colours started its use from an early time (Vankar, 2000). Natural dyes used in food are screened for safety but the information is not known for most of the natural dyes used in craft dyeing and with potentially wider use. Colour is important in consumer acceptance of food products. There is a tendency to assume that consumable natural products are

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safer and better than synthetic product because they came naturally. The safety of natural dyes needs to be proved if they are used more widely and in commercial process (Kumar and Bharti, 1998).

Natural dyes produce an extraordinary diversity of rich and complex colours that complement each other. “Brian Murphy” said that natural dyes are vastly superior to synthetic dyes (Brian, 2006). The dye age well and develop a patina as the older textiles are exposed to sunlight and normal use (Brian, 2006). The eco-standards narrated by different environmental agencies such as environmental protection agency (EPA) and Food and agriculture organization (FAO) etc. have compelled the textile customers to choose the eco-label products (Shaheed *et al.*, 2014). Hence, there is a demand for natural dyes which are renewable, biodegradable and eco-friendly. Natural dyes showed better biodegradability and have higher compatibility towards environment due to which they are attracting attention around the globe.

Sorghum Bicolour

Sorghum bicolour, commonly called sorghum and also known as great millet, durra, jowari or milo, is a grass species cultivated for its grain, which is used for food for humans, animal feed, and ethanol production (Norton*et al.*, 2007).Sorghum is a genus of flavouring plants in the grass family *Poaceae*. Seventeen of the twenty-five species are native to Austria, with the range of some extending to Africa, Asia, Mesoamerica and certain islands in the Indian and pacific Oceans (Nwonye and Priscilla, 2017). One species is grown for grain, while many others are used as folder plants, either cultivated in warm climates worldwide or naturalized in pasture land (Folachode, 2018). Sorghum use less water compared to other grain crops. *Sorghum bicolour* leaves consist primarily of carotenoids, flavonoids and phenolic acids, chlorophyll, lycopene and beta-carotene, as well as palmitic, stearic, oleic and linoleic acid. Studies suggest that a diet prepared with these leaves would provide natural antioxidants and essential fatty acids that could fight cardiovascular related diseases.

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There are four main types of sorghum namely; *White Sorghum, Waxy Burgundy Sorghum, Sumac Sorghum, Black Sorghum.*

Materials and Methods

The dried sample was collected from a local market at Azare, Katagum Local Area council of Bauchi State, Nigeria. It was certified by a specialist from department of Crop Production Abubakar Tafawa Balewa University, Bauchi. The leaf stalks were ground to powder and preserved for analysis.

The 100% cotton material was collected from a local market "Kurmi" in Kano, Kano municipal area of Kano state and was certified by a specialist from the department of chemistry, polymer section, Abubakar Tafawa Balewa University, Bauchi.

Extraction

Method of Gahlot *et al.* (2018) was adopted with little modification. A 50 g of the ground sample was soaked in a glass fitted with cork for 48 hours in 500 cm^3 of methanol with intermittent shaking. It was filtered using a filter paper and was washed off using 200 cm^3 of methanol. The extract was allowed to dry – off under fan for two days. The dried filtered extract was purified in hexane by boiling, filtered, and air dried for future analysis.

Characterization of Extract

The dried sample was passed through series of analysis which include:

Mass and percentage yield analysis

Mass of the dried sample was determined using a weighing balance and was recorded as the actual yield and the theoretical yield was taken as the mass of the initial sample that was soaked. The percentage yield was calculated using the formula below (Bello, 2010).

$$\%\ Yield = \frac{Actual\ Yield}{Theoretical\ Yield} \times 100$$

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Melting point analysis

The melting point of the extract was determined using the Gallenkemp melting point apparatus. A certain amount of the dye was placed on the block, a thermometer inserted into the hole, and the block placed on a heating plate. The temperature at which the dye starts to melt or darkens (decomposes) was noted, and the value recorded (Fasoyiro, 2014).

Solubility

The solubility of the extract was determined using the following solvents: acetone, ethanol, dimethyl formamide, methanol, hexane, carbon tetrachloride and water. A small amount of the dye was placed in a test-tube and about 1- 5 cm^3 of the solvent was added till it either dissolves or not under room temperature. Same method was applied for all solvents. The solubility properties were recorded (Fasoyiro, 2014).

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pH Measurement

The method of Fasoyiro (2014) was adopted. About 1 g of the dye sample was boiled for 5 min in 250 ml of distilled water and was allowed to cool down before filtering with whatman filter paper. The pH of the filtrate was measured.

Determination of maximum wavelength of absorption

The maximum wavelength (λ max) of absorption for the extract (dye) was determined in distilled water using Jenway 6305 UV – visible spectrophotometer. A gram of the extract and 0.5 g of NaOH was dissolved in distilled water and made up to the mark in a 250 cm^3 volumetric flask to make a stock solution. A 1 % solution was made from the stock which was used in the

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determination of the maximum wavelength of absorption from 400 – 600 nm, at an interval of 20 nm.

Fabric Purification and Preparation

The fabric (cotton) was scoured, bleached and mercerized according to standard methods adopted from Omizegba *et al*. (2017) as described below.

Scouring

A 2 % NaOH solution was prepared and the 10 cm X 10 cm dimensions of the fabrics were immersed and were allowed to boil for 1 hour at a constant temperature. The fabrics were neutralized with a solution of 5 % acetic acid (CH_3COOH) and were washed off under a running tap and dried at room temperature (Omizegba *et al.,* 2017).

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Bleaching

The scoured fabrics were immersed in a mixture of a solution of a 0.5g magnesium sulphate ($MgSO_4$) and 0.1 g of sodium silicate ($NaSiO_3$) dissolved in a 10ml of 1 % NaOH and was diluted in a 5 % Hydrogen peroxide solution H_2O_2. It was boiled for 45 min and neutralized in a solution of 5 % CH_3COOH and washed with water. The substrate was dried at room temperature (Omizegba *et al.,* 2017).

Mercerization

The bleached fabrics were treated with a solution of 22 % NaOH at 0-5 ^{0}C for 45 min. The samples were neutralized in 5 % CH_3COOH solution and were washed in a 2 % detergent solution,

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completely rinsed in water and dried in the laboratory at room temperature (Omizegba *et al.*, 2017).

Preparation of Stock Solution

Dyeing process was adopted from Boryo *et al.* (2013) and Omizegba *et al.* (2017) with little modifications. The weight of the mercerized cotton fabric was taken, and the stock solution was made. The stock was made by weighing 1g of dye into 250 ml volumetric flask followed by the addition of sodium Meta bisulfite (2 g) and sodium hydroxide (1.25 g). It was pasted with little distilled water for proper dissolution and was made up to the mark in 250 ml volumetric flask (Boryo *et al.,* 2013). The quantity of the dye stock solution used was calculated or determined using the formula.

Volume of dye stock = $\frac{W \times P}{C}$

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Where:

W= weight of fabric sample

P = % shade (dyeing required)

C = % concentration of the dye solution

$= \frac{1}{250} \times 100 = 0.4$

Determination of percentage exhaustion

Dyeing was carried out according to Omizegba *et al.* (2017) with little modification in 3 different batches of 3 %, 4 % and 5 % respectively. The liquor ratio was 50:1 (that is 1g of fabric was dyed in 50 ml of dye liquor). Dyeing was carried out in a 500 ml beaker and the pretreated fabric was wet with distilled water, and was immersed completely in the beaker containing the dye bath solution. Dyeing was carried out at room temperature for 60 min.

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At 10 min interval, a sample of the dye was collected from the dye bath with the aid of a dropper. Each of these samples was later analyzed with the aid of an ultraviolet, spectrophotometer at a wavelength 430 nm.

The degree of percentage exhaustion was calculated using the formula

% Exhaustion = $\frac{C_o - C_t}{C_o} \times 100$ (Naser *et al.*, 2015)

Where:

C_o = Initial absorbance of dye bath

C_t = Absorbance at time

Determination of wash fastness

The dyed samples were subjected to ISO3 (International Organization for standardization) wash fastness test according to Boryo *et al,* (2013) and results were observed and recorded. The sample specimens were prepared by cutting the dyed samples into 2cm by 5cm dimensions. The solution was made by dissolving 2 g sodium carbonate and 5 g laboratory detergent (sodium lauryl sulphate SLS) in 1L of distilled water. The liquor ratio was 50:1 (1g of fabric in 50ml of solution) and the specimen immersed completely into the solution in a 100ml beaker. It was agitated for some time and was immersed in a water bath for 30 min at 50 ^oC (Boryo *et al.,* 2013).

Determination of Stain Fastness

The dyed samples were subjected to ISO3 (International Organization for standardization) wash fastness test according to Boryo *et al.* (2013) and results were observed and recorded. The sample specimens were prepared by cutting the dyed samples into 2 cm by 5 cm dimensions. These were made into composites by stitching test specimen made up of the dyed sample placed between un-dyed samples of same dimensions. The solution was made by dissolving 2 g sodium carbonate and 5 g laboratory detergent (sodium lauryl sulphate SLS) in 1L of distilled water. The liquor

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ratio was 50:1 (1 g of fabric in 50 ml of solution) and the specimen immersed completely into the solution in a 100 ml beaker. It was agitated for some time and was immersed in a water bath for 30 min at 50 ^{o}C (Boryo *et al.*, 2013).

Result and Discussion

Physical Characteristics

The physical characteristics of the dye were analyzed and the following results obtained;

Table 1: Physical properties of *Sorghum bicolour* sheath extract

Dye	Melting point (^{o}C)	Weight of extract (g)	Yield (%)	Color	pH
Sorghum leaf sheath	246	12	24	Maroon	4.47

Melting point

Normally, melting points of most naturally occurring compounds are high. A melting point value of 246 ^{o}C was observed for the methanol extract of *Sorghum bicolour* sheaths. Mahkam *et al.* (2014) obtained a temperature range of 192-195 ^{o}C for *Lawsone inermis*. Osabohien (2014) obtained 186 ^{o}C and 194 ^{o}C for dyes extracted from guinea corn and onion skin respectively. This value is lower than that obtained for this research. This could be due to the difference in solvent for extraction, nature of the environment where it was grown or the specie.

Mass

Among the yield naturally occurring compounds, sorghum stalk happen to have a high yield compared to other isolated compounds. A mass of 12g which is equivalent to 24 when expressed in % yield was obtained from 50g *Sorghum bicolour* sheath. Alam *et al.* (2007) obtained a 7.05 g yield for *Lawsone inermis* from 100 g dried henna leaves while Mahkam *et al.*, (2014) recorded 7.20 g yield from 40 g henna leaves. The two values are lower than that obtained for the present research.

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Time taken for extraction

The duration of extraction has an impact on the yield as given by Ajay *et al,* (2014) in four different media namely; alkaline, acidic, aqueous and alcoholic media. Acidic, aqueous and alcoholic media increase with increase in extraction time, except in the alkaline medium, which decreases with increase in time.

The two day extraction period observed on the sorghum stalk sheaths gave a very good yield (24 % from 50 g) which is even higher than that obtained by Babatunde (2017) over a 7-day period (13.4 % from 450 g sample).

Colour of the extract

A maroon colour was obtained from the methanol extract of *Sorghum bicolour* sheaths. A dark yellowish-brown dye was obtained from the red flowers of W. *fruticosa* by Grover and Patni (2011), *Sorghum bicolor* gave red-maroon-black colored dye and brown colour was obtained by Mirjali and Karimi (2013) for green walnut shells. Osabohien (2014) obtained a dye from the bark of whistling pine and root of African peach respectively giving reddish brown and brown colours too.

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pH Determination

The pH of the filtrate (filtered dye sample) was measured and was found to be acidic (4.47). *Lawsone inermis* was also found to be acidic Ashnagar and Shiri (2011). A value of 4.74 was also obtained for Henna leaves by Umadevi *et al.*, 2017.

Solubility

Table 2: Solubility of the Dye in various solvents

Dye	Acetone	Ethanol	DMF	Methanol	Carbon tetrachloride	Water
Sb	S	S	S	S	NS	NS

NS = Not soluble

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N = Soluble

DMF= Dimethyl formamide

Sb = *Sorghum bicolour* sheath extract

The dye is soluble in all polar solvents except water thus, signifying it may be an ionic compound. The dye is sparingly soluble in hot water and completely soluble in normal water at room temperature in the presence of some additives a method adopted from Boryo *et al.,* 2013. Osabohien (2014) obtained dyes from onion skin and guinea corn leaf which are soluble in organic solvents, sparingly soluble in cold water and readily soluble in hot water. The properties are similar to those of the sorghum stalk sheath.

Determination of maximum wavelength of absorption

Table 3: Maximum wavelength of absorption of Dye

Dye	Concentration (%)	Wavelength (nm)
Sorghum leaf extract	1	430

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The UV- Spectrophotometric analysis of the dye was analyzed at a concentration of 10,000 ppm (1 %) and its maximum wavelength of absorption was found to be 430 nm. A significant peak at wavelength of 482 nm was also observed which corresponds to that obtained for indicaxanthin and betanin which were found to be responsible for the red color in the specie (Folachode, 2018). Babatunde (2017) obtained a λ max of 535 nm (value associated to delphinidin dyes under bathochromic shift) using ethanol as a solvent for extraction. Mirjalili and Karimi (2013) obtained 360 nm as the maximum peak for absorption which is lower than that obtained for the present research. The total phenolic content and anthocyanin content of red colorant from dye sorghum was analyzed by Folachode *et al,* (2017) with 130.7 mg/g and 28.7 mg/g values respectively.

Degree of Percentage exhaustion

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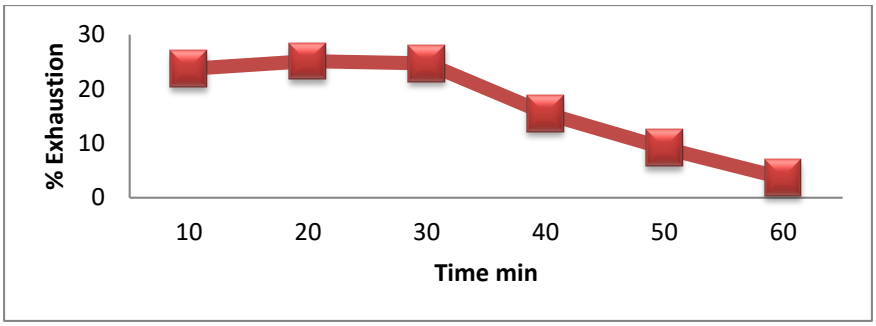

Figure 5: Percentage exhaustion of 5 % shade

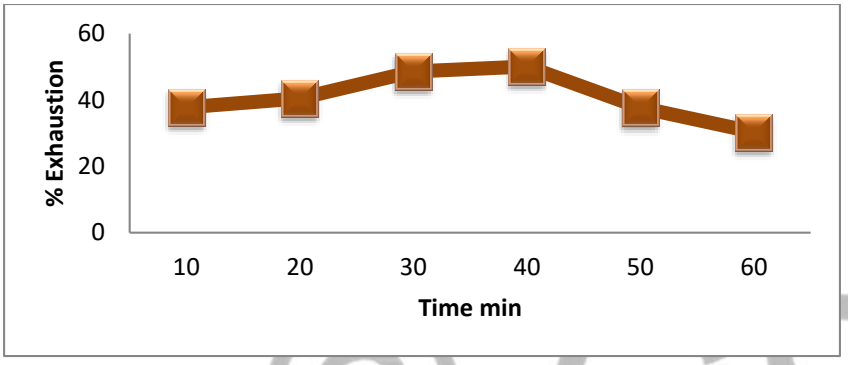

Figure 6: Percentage exhaustion of 4 % shade

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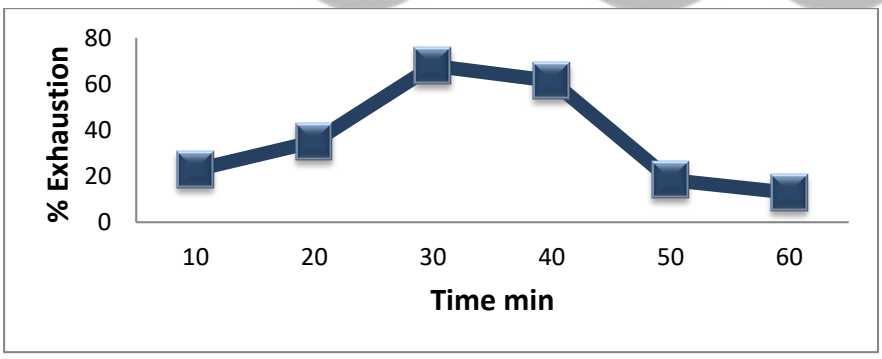

Figure 7: Percentage exhaustion of 3% shade

Figures 4, 5 and 6 Show the percentage exhaustion of *Sorghum bicolour* dye on a mercerized cotton fabric. The dyeing concentrations were varied and the time and temperature kept constant. The dyeing was observed at 3 %, 4 %, and 5 % dye baths. The 3 % dyeing has its peak of absorption (exhaustion) at time t = 30 min, 4 % at t = 40 min and 5 % t = 20 min.

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Higher maximum absorptions (% exhaustion) were observed for the dye bath concentrations as; 3 %, % E = 67.8, 4 %, % E = 50.1 and 5 %, % E = 25.1. The percent exhaustion decreases with increase in concentration. The intensity of the colours obtained increase with increase in percent concentration.

Grey Scale Assessments

The washed-dyed fabrics and prepared washed-composites were assessed for change in colour and staining respectively.

Wash Fastness

A direct dyeing process was observed (at room temperature in the absence of heat) and a dark Lilac colour was obtained. *Sorghum bicolour* sheath is poor to moderate when assessed for change in colour. The fabrics have affinity to the dye but the hue obtained after washing is moderate as was reported by Figueiredo *et al,* (2010). A direct dyeing process for Sorghum bicolour dye extract by Udeani and Milila (2016) on cotton fabric showed a moderate affinity, such was also observed by Shaukat *et al,* (2009) and Hassan *et al,* (2015). Udeani and Milila (2016) confirmed that great fastness ability was observed when dyed in the presence of a mordant (natural extract of *Pakia biglobosa*). Udeani (2015) stated that different investigation has revealed that cotton is less suitable for many natural dyes, although it cannot be ruled out completely as a substrate for carrying out investigations on natural dye sources as desirable stains can still be obtained for various dyeing activities.

Conclusion

The yield obtained (24%) was good compared to other isolated natural compounds (13.4%). The hot extraction method gave a higher yield than the cold based on the results compared. The structure of the colour conferring component was partially characterized and was found to be only soluble in polar organic solvents. It is acidic in nature with a high melting point value. Even though the colour is so intense, but it has moderate dye ability.

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