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GUINEA CORN (*SORGHUM VULGARE*): LEAVES A POTENTIAL SOURCE OF TEXTILE DYE FOR NATURAL AND MAN - MADE FABRICS IN NIGERIA

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ABSTRACT

This study examined colour shades, dye quantity and fastness properties of dye extracted from guinea corn leaf, (*Sorghum vulgare)* for the dyeing of natural and man-made fabric. The dye component extracted from sorghum vulgare was tested for washing and light fastness. The leaves were dried at room temperature for two weeks before drying in the oven at 60°C for 2 hrs for easy blending to powder. Ethanol, Methanol and Acetone solvents were used for extraction at cold and hot conditions and Soxhlet methods. Three different yarn types were used as substrate: cotton, wool and polyester. The quantity of crude dye extracted using 200 gm of guinea corn leave powder and 250 ml of solvent for hot extraction is 1.53 gm and cold1.61 gm. with ethanol, methanol 0.98 gm (Hot) and 1.01 gm (Cold) and Acetone 0.92 gm (Hot) and 0.94 gm (cold).The sample colour was burgundy but with the solvent interaction and temperature the colours under hot condition (78°C) ranges from dirty red to dull brown and under cold (23°C) red brown to cartoon brown. Colour range test, L value under cold dyeing conditions, the real sample showed a lightness value of 10.62 and the controlled sample 11.12, indicating moderately dark shades. However, under hot dyeing conditions, these values dropped to 6.55 gm and 6.71gm respectively, at 40% reduction in lightness. Hot dyeing produced darker fabrics but, it diminished colour vibrancy. Redness values reduced by approximately 26% under hot conditions, while yellowness dropped by 58%. This inverse relationship suggests that *Sorghum vulgare* contains multiple colour components with different temperature requirements.

Keywords: *Sorghum vulgare; leaves:* dye; cotton; polyester; artificial fabric fastness properties.

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INTRODUCTION

Before the emergence of synthetic dyes, every dye used as colourant for food or fabrics were from nature, either plant, animal or non-living natural objects such as stones, mud or clay etc. Of all materials used, plants were the most prominent (Nkem *et al.,* 2018). The ease of application, the florescent nature of the colour and the availability gave the synthetic dyes an edge over natural dyes. However, with the world environmental health consciousness to "Save the mother earth" and her inhabitants from chemical pollution that is detrimental to the health of both man and animals, huge attention has now returned eco – friendly colourants (Akinboro 2015). However, studies have revealed that synthetic dyes emit a lot of chemicals into the air during use that pollute the environment thereby causing health hazards to both man and animals. The textile industries are regarded as one of the major of hazardous chemical consumers due to the incessant use of synthetic dye which is very toxic and have high effluent waste. This has been a great concern to consumers and stakeholders in this field. (The new awareness is an encouraging research on how best to extract colour from nature that is ecofriendly). In some developed countries, dyes extracted from nature has been commercialized, examples of such are:

For blue colouration of different shades, indigo (*indigofera tinctorial*) leaves have been used in Nigeria and United Kingdom (UK). The fastness to washing is average but the fastness to light is poor. Other colourants have been commonly used in various locations (Table 1).

Therefore, extraction of dyes from natural sources is hereby encouraged though it is believed by some scholars that natural dyes cannot replace synthetic dyes. The African continent is rich in different plant species with potential to produce novel natural products with dye-yielding properties. Wanyama *et al.* (2014) reinstated that Nigeria, with her abundance of natural resources, particularly thick vegetation should not seat on the fence. This study experimented with Guinea corn (*Sorghum vulgare*) leaves as a potential source of textile dye in Nigeria because Nigeria is the third leading world producer of Guinea corn (FSD, 2007). Although much research has been done on Guinea corn leaf as colourant for food or textile, their substrate seems to be limited to cotton material or linen which are of same family. This study has further experimented with polyester and nylon fibre.

Guinea corn (*Sorghum Vulgare)* leaf is a farm waste after harvest. It is a member of the grass family Poaceae which can thrive in hot areas with little rainfall, providing nutrients for millions of people (Oyetayo and Ogunrotimi, 2012). The genus Sorghum is one of about 600 genera of Poaceae. Sorghum is an important staple in the diet of Nigerians where it is a major food crop. It feeds both human and animals (Osagie 1998 cited by Olaoye and Ogunrotimi 2012). According to Osabohien (2014), in Yoruba, it is called Poro- poro baba though wrongly spelt and written in the reference, Milo maize in United states, Dura in Sudan, Great millet and Guinea corn in West Africa, Kafri in South Africa, Mtama in Eastern Africa and Jowar in India (FAO, 1995, Osabohiem, 2014).

Food and Agriculture Organization of United Nations FAO (2011) described *Sorghum Vulgare a*s the fifth most important cereal crop in the world. It has hollow stem and the leaves are plugged at intervals. It is a per-

Table 1: List of Natural Dyes in Commercial Use

Colour	Plant name and Part	Botanical name	Description/ observation
Blue	Indigo leaves – Europe, Nigeria.	Vat dye class Indigofera tinctorial	Popularly known as the king of natural dyes. It produces indigo blue of different shades. The fastness to washing is average but the fastness to light is poor.
Red	Madder/roots – India	Alizirin dye class. Rubia cordifoliquka	Queen of natural dyes. It produces red colour. It possesses all round good fastness quality.
Red	Morinda. Root and bark – Sri lanka	Morinda citrifolia.	The plant produces red colour and the fastness quality to both washing and light is average.
Yellow \ Scarlet red	Safflower/ Safflower florets. – Afghanistan		The plants produce shades of yellow and scarlet red. The washing and light fastness properties are poor.
Yellow	Tumeric/ rhizomes.	Substantive dye class	The colour produced is fast to washing but poor to light. The plant produces a yellow colour.
Orange	Saffron/ stigmas of flower – Iran & India.	Direct dye class Crocus Sativus.	The plant produces an orange colour. The fastness quality is low to both washing and light.
Red/ Orange	Annatto/ seeds. Fabric: wool, silk and cotton	Bixaorellano	It produces reddish, orange shades and the fastness property to washing and light is poor.
Crust red	Barberry/ roots, bark and stems.	Alkaloid dye type Berberus aristata.	The dye has good fastness quality and the plant exhibit crust red colour.
Yellow	Myrobalan/ fruits.	Terminalia chebula	Myrobalan produces a yellow colour. The colour is very fast to both washing and light.
Varieties of colours	Marigold.	Tagetus spp.	It has good fastness properties. It brings variety of colours
Varieties of colours	Flame of the forest/ flower – India	Butea Monosperma	Different colours can be derived from this plant. The fastness property is high.
Peach	Onion/ outed skin	Flavonoid dye class Allium cepa	The washing and light fastness is moderate. The plant produces a peach colour
Brown	Weld – Europe	Flavinoid dye class Reseda inteola	Very good fastness properties and a brown colour is produced
	Dolu/ Roots and Rhizomes.	Rheum emodi	The plant produces dull green colour with a low fastness property

ennial crop which can be harvested many times during the year. This may be one of the reasons why Oyetayo and Ogunrotimi (2012) labelled Guinea corn (*sorghum vulgare*) leaf as an addition of huge mass environmental waste derived from the crop harvest. The study explored, exploited and utilized the waste for wealth as mentioned by Osabohien (2014) by extracting colour from the intensive burgundy colour leaves of Guinea corn. Guinea Corn is a multi-purpose grain even as adhesive in the manufacture of plywood (Hojilla-Evangelister, and Bean 2011 and Olukemi *et al.,* 2005). Guinea corn varies in colour from yellow ochre to red brown and deep wine, depending on when it is cultivated (Rampho 2005). The physical observation of Guinea corn plant and the strong red wine colour on the leaves call for attention that it is a carrier of colour (Rampho 2005). Dye is a substance used to colour materials permanently in such a manner that the imparted colour becomes part of the materials and not merely applied to the surface as in printing pigments. (Halsey, *et al.,* 1968, cited by Braide, 2012).

The purpose and significance of the study is to reduce the dependency of fabric producers on imported synthetic dyestuffs, which according to Ekong (2016) poses a threat to environment because of its hazardous chemical processes. It will reduce or eradicate chemical air pollution, and environmental degradation that is dangerous to the health of man, animal and plants. The study will act as a spring board to induce other researchers to take advantage of Nigeria's thick vegetation and abundance of natural resources by extracting more colours other than the indigo blue from Elu (*Indigofera tinctorial*) leaves. It will also reduce post-harvest waste and the environmental nuisance the leaves constitute. The study may encourage the revisit of lost crops by stakeholders thereby rekindling farmers' interest to propagate them and this will facilitate employment.

MATERIALS AND METHODS

Study area

Guinea corn (*Sorghum vulgare*) leaves were collected from the University Farm, Federal University of Agriculture Abeokuta, Ogun State, Nigeria.

Materials used for extraction:

All the equipment used for the study were got from the Department of Chemistry, Ahmadu Bello University Zaria, Kaduna.

Substrate: White cotton material, Polyester and Nylon yarn

Methods: The dyestuff was extracted from the leaves of Guinea corn.

Laboratory Experiments

Preparation of Guinea corn leaves

The gathered Guinea corn leaves were left to dry at room temperature for two weeks before been dried in the oven at 60^{o}C for 2 hrs and blended into powder using a blender. The husks were kept away from direct sun and excess oven heat, so it may help in preserving the colour more since studies have shown that natural dyes do not have much affinity for heat or sunlight (Braide, 2012, Yusuf *et al.,* 2017). The samples were exposed to 2 hrs oven heat to aid the crispiness for easy blending into powder. The powdered sample was stored in an air tight bottle, and the moisture content was determined by oven drying method at 110^{o}C for 4 hrs as recommended by FAO (1998), cited by Oyebola and Ogunrotimi, 2012). Ash content was determined using muffle furnace at 550^{O}c and dye content was determined using Soxhlet extraction method. The sample was powdered as reported by Stahil (1965) and cited by Osabohien (2014) that it allows for most intimate contact with solvent.

Test for Colour and Dye Characteristic

The experiment was divided into three segments: Test for colour, Extraction process proper and test for Dye characteristics.

From the definition of dye by Halsey *et al* 1968, cited by Braide 2020, it can be deduced that not all coloured substances have the property of a dye. Therefore, the supposed dye material collected was subjected to colour and dye characteristics test.

Colour Test:
A little quantity of the prepared fine powdered leaves without measurement was put in small evaporating dishes and little drops of each of these solvents: Ethanol. (C_2H_5OH), Acetone $(CH_3)_2CO$ and Methanol (CH_3OH) were added to the powder to test for the presence of colour. It was kept for 30 minutes and stirred just once within this period, after which the colourless solvents changed colour and this confirmed the presence of colours in the sample. After the confirmation of colour, the extraction process commenced to test for dye characteristics.

Extraction Process: Two extraction methods were experimented upon:
i. Extraction with organic solvent
ii. Soxhlet extraction technique.

Extraction with organic solvent at room (23^{o}C) temperature
The materials used for extraction with solvents at room temperature were:
i. Bottles with Lids: for soaking the sample.
ii. Guinea corn leaf powder (*Sorghum vulgare*)
iii. Electric Shaker: to agitate the sample in order not to allow the guinea corn leaf powder to settle and to aid better extraction
iv. Solvents to soak the prepared *Sorghum vulgare* powder: Ethanol, Methanol and Acetone.
v. Weighing scale: to determine the weight of the plant powder to be soaked
vi. Measuring cylinder: to measure solvent into the plant powder
vii. Funnel with stands and filter papers: to filter the sample powder from the solvent
viii. Beaker; to collect the filtered solvent for evaporation
ix. Desiccator, to dry up the moisture in the dye and make it crispy for preservation.

The weight of the Guinea corn leaf powder for extraction was 6.735 grams, it was measured into bottles with lid filled with 250ml each of the three solvents; Ethanol, Methanol and Acetone. The neck of the bottles was hung on the handle of a shaker and was left on for at least 16 hours every day but not over-night. The reason for this was to avoid damage and spillage. This state was maintained for three days for the first extraction, six days for the second extraction and nine days for the third extraction. At the end each soaking period, the solvent was filtered from the crumb. The filtered solvent was dried using desiccator to evaporate the solvent away from the dye, living it in a crystal form.

Extraction with Organic Solvent at Boiling Temperature
The same process of cold extraction was adopted for the hot extraction but with little variation based on temperature and the change on extraction time. For hot extraction, the soaked powder in the flat bottom flask were left on the water bath to boil for only four hours after which it was left to simmer before filtering. Temperature in maceration can be cold or hot, this type of technique requires special apparatus like the Soxhlet (Nwoye and Ezema 2017).

Soxhlet extraction is a technique that places a special glass between a flask and a condenser. The technique is mostly carried out for colour identification and quantity record. The temperature was well regulated and the solvent was well circulated with the powdered plant over and over again.

Materials Used:
i. Stand: For holding the set
ii. Clamp: To hold the condenser
iii. Thimble: To keep the herb
iv. Solution bottle: To pour the solution for boiling
v. Rubber Tubes: to transfer water into the condenser.
vi. Weighing Scale: To weigh the crumbs
vii. Measuring cylinder: To measure solution
viii. Heating mantle: to boil the solution

The boiling of the prepared *Sorghum vulgare* powder in the solution was for four hours at 100⁰C. The amount of powder that can be extracted at a time with this Soxhlet method is limited because the thimble has a readily made size. The powder was poured in the thimbles after measurement and the extraction solution was measured into the solution bottle and was placed on the heating mantle. On the solution bottle was placed the thimble that had already been filled with the *Sorghum vulgare* sample powder, on this condenser was placed another condenser referred to as the top condenser. It was from this condenser that water dripped into the solution in order not to allow excess evaporation of solution and it is from the condensers on the solution bottle that the steam of the solution dropped into the thimble. With the simple dripping of solution, the guinea corn leaf powder gets wet and the dye solution filtered out of the thimble and dripped back through a different pipe on the same condenser into the solution bottle. After four hours of boiling, a saturated coloured solution was formed, ready for evaporation.

RESULTS AND DISCUSSION

Different solvents reacted differently to temperature during extraction based on their colour output. Visually at the liquid stage before evaporation the colours varied by temperature per solvent, the hot extraction shows values (faint) in their hue while under cold condition the colour intensity (thickness) is more (Table 2).

Table 2: Colour Variation in dye solution with Different Solvents and Method of Extraction

S/N	Local Names	Extraction Solvent	Sample Colour	Liquid Extraction Colour (Hot)	Liquid Extraction Colour (Cold)
1	Poro poro-baba	Ethanol	Burgundy	Dirty Red	Red Brown
2	Poro poro-baba	Methanol	Burgundy	Yellow Ochre	Cartoon Brown
3	Poro poro-baba	Acetone	Burgundy	Dull brown	Red Brown

With an extraction condition and time of 78⁰C for 4 hours for hot extraction and room temperature of 23°C for 3 days for the cold extraction, the cold extraction process yielded more dye compared to hot extraction method (Table 3). Considering the time difference in the extraction processes of each method, the number of days (3) for cold and hours (4) for hot extraction, the quantity margin may be termed insignificant. Ethanol, of the three-extraction solvents yielded the highest quantity of dye at both hot and cold condition (Table 3). This study negates the findings of Nkem *et al.* (2018) which state that hot water extraction is a better method of extraction. The hot extraction time is faster than the cold. Mordants tone the hue of the colour from intensive to valuable or pastel (Table 3). According to Wan Yunis *et al* (2017) that normally natural dyes are extracted by boiling but not to say there can be no other methods of extraction, the result of this study buttressed that statement because cold and hot extraction was used and the cold yielded more dye than the hot extraction.

Table 3: Extraction Process with Ethanol, Methanol and Acetone using same Ex Condition (200 gm sample/250 ml solvent)

Solvent	Hot	Cold
Ethanol	1..53	1.61
Methanol	0.98	1.01
Acetone	0.92	0.94

Under cold dyeing conditions, the real sample showed a lightness value of 10.62 and the controlled sample showed 11.12, indicating moderately dark shades (Table 4). However, under hot dyeing conditions, these values dropped to 6.55 and 6.71 respectively, at 40% reduction in lightness. While hot dyeing produced darker shades, it diminished colour vibrancy. Redness values reduced by approximately 26% under hot conditions, while yellowness dropped by 58% (Table 4). This inverse relationship suggests that *Sorghum vulgare* contains multiple colour components with different temperature requirements.

Table 4: Colour Range Test. Unmordanted Cotton Sample Dyed with *Sorghum vulgare*

Sample	L	a*	b*
Real sample	10.62 (cold)	2.10	3.87
Controlled	11.12 (cold)	2.93	4.05
Real sample	6.55 (hot) 40% reduction	1.56 (26%)	1.63
Controlled	6. 71`(hot)	1.69	1.72 (58%)

The observations corroborate the findings of Saraja and Rajab (2014) that expressed that polyester and wool has more affinity for natural dyes even more that the cotton fabric that is from the natural fibre (Table 5). The dye extract without mordant performed better on polyester and nylon than on cotton fabric. It also had good level of fastness quality without mordant to washing and heat (Table 5). This corroborates the findings of Udeani and Milila (2018), but with mordant as revealed by Osabhein (2013); Zubairu and Mshelia (2015), it produces a more radiant colour based on their different types.

Table 5: Unmordanted Cotton Blend (Polyester Plus Cotton) and Nylon (Cold) Dyeing

Sample	L	A	B	L	A	B
R S	15.27	3.27	5.85	22.43	11,51	11.44
C S	17.26	5.89	7.70	17.46	10.71	9.42

Under cold dyeing conditions, the two samples exhibited distinctly different colour parameters (Table 6). The Real Sample (RS) recorded a lightness value of 8.63, while the Controlled Sample (CS) registered 7.58. This represents a difference of 1.05 units on the L* scale, meaning the Controlled Sample was 12.2% darker than the Real Sample under identical cold dyeing conditions with mordant. The chromatic values revealed more substantial differences. For redness (a), the Real Sample measured 5.43 compared to the Controlled Sample's 4.57, yielding a difference of 0.86 units (Table 6).

Table 6: Mordanted Cotton Fabric under Cold and Hot Condition

	L	A	B	L	A	B
Sample	Cold			Hot		
RS	8.63	5.43	4.16	11.58	3.54	4.39
CS	7.58	4.57	3.30	11.55	3.52	4.37

The unmordanted cotton fabric demonstrated distinct colour changes in lightness (L^* =8.75) with relatively low redness of a^* = 0.46 and high yellowness of b^* =8.39 (Table 7). The lightness decreased slightly from b^* = 8.39 after washing to b* = 7.24 and light exposure, indicating moderate stability of the yellow component under light.
The mordanted fabric exhibited different behavior patterns under the two test conditions. The yellowness (b*= 3.87) was considerably lower than the unmordanted washed sample (b* = 8.39). Under light exposure, the mordanted sample showed the highest lightness value across all conditions (L*= 12.84). This represents a 78% increase in lightness compared to the unmordanted light -exposed sample, indicating that the mordanted fabric experienced significantly more washing (Table 7).

Table 7: Light Washing Fastness Table of Unmordanted and Mordanted Cotton Fabric

Sample	L	A	B	L	A	B
Washing	8.75	0.46	8.39	10.79	1.66	3.87
Light	7.21	3.12	7.24	12.84	1.72	4.75

CONCLUSION

The study has achieved some level of success in enlightening stakeholders including local textile dyers, designers, traders and users, on the abundance of plants and other farm waste that can be converted to wealth through exploratory research.

Guinea corn leaves are capable of producing textile dye for cotton, polyamide and nylon and have a good affinity for man-made fibres.

The study negates the assumption that natural dyes only have affinity for natural products.

The dye extract without mordant performed better on polyester and nylon than on cotton fabric, it also has good level of fastness quality without mordant to washing and heat.

Radiance in colour as observed with mordant brings reduction in tint or intensity.

Guinea corn (*Sorghum vulgare*) leaf without mordant exhibits a good-quality hue.

In polyamide and nylon, application of dye extracted from Guinea corn leaf (*Sorghum vulgare*), requires no mordant. When mordanted it does not add any value to colour hue, bleeding or heat.

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