International Journal of
Development Strategies in Humanities, Management and Social Sciences | IJDSHMSS
ISSN Hard Print: 2360-9036 | ISSN Online: 2360-9044 | Volume 7, Number 1 May, 2017

Extraction and Utilization of Natural Dye Extract from Guinea Corn Leaf

[1]**Nwonye, N. U.** & [2]**Priscilla Nnenna Ezema**
[1]*Department of Home Economics, Nutrition & Dietetics, University of Uyo*
[2]*Department of Home Economics, Hospitality Management and Tourism, Michael Okpara University of Agriculture, Umudike*

Abstract

The leaves of guinea corn were bought from the market, chopped and the dye content was extracted using water and acetone respectively as solvents. The extraction of the dye was done using cold method (maceration) and soxhlet. This work was on guinea corn, a potential source of dye stuff for textile and food. The objective of the study was to assess the dye and colouring potential of the extracts from guinea corn, using different techniques of dye extraction: assess the light, washing and rubbing fastness properties and to impart colour on food using the extracted dye. To achieve the above objectives, guinea corn leaf, alum, cotton fabric and other materials were bought from the market in Aba. The experiment was carried out in chemistry laboratory in the University of Uyo, Uyo Akwa Ibom State. The sample was treated and the substrate was scoured and mordanted. Cold maceration and soxhlet extraction were employed using distilled water and acetone as solvents. The dye was produced in four states using distilled water and acetone plus sample in maceration and the yield were wine and slightly deep wine respectively while in soxhlet extraction, distilled water plus sample and acetone plus sample yielded slightly reddish brown and reddish brown colours. The extracted dyes were applied on cotton fabric treated with alum. The colours after dyeing yielded blush, deep blush, brick and deep brick respectively. The extracted dye was also used to impart colour on foods. It was therefore concluded that the process of the extraction was eco-friendly. The dye obtained has the dyeing potential which can be used as a source of textile dyeing and food colouring. It is therefore recommended that efforts should be made towards producing natural dyes as they are eco-friendly and can be produced in different shades.

Keywords: *Extraction, Natural dye, Acetone, Soxhlet, Maceration, Guinea corn leaf*

Corresponding Author: Nwonye, N. U.

Background to the Study

Natural dyes can be obtained from various parts of plants such as flowers, roots, leaves, barks, insects' secretion and minerals. These plants can be found in our environment. Although dyes abound in the natural environment, they can also be formed in different ways and used in different applications depending on their manufacturing processes (Obenewaa, 2010). Osobohien (2009) defined natural dyes as substances from plants and animals that impart colour to foods, cosmetics, drugs, hair, fibre, fur and polymers. The dyes are introduced to different items to upgrade their appearance and make them attractive for consumers to patronize. Irrespective of the pressing issue connected with use of natural dyes such as poor yield, poor adherence to textile materials, there is the need for sustainable processes which can be seen as the driving force for the development of new strategies to revive the use of natural dyes.

Natural dyes can exhibit better biodegradability and generally possess higher compatibility with the environment compared to synthetic dyes (Osabohien, 2009) and Otutu et al, 2010). Round the universe efforts are tailored toward promoting the cultivation of natural dye plants and their application in dyeing (Osabbohien, 2014). This has been possible because they are eco-friendly compared to synthetic dyes.

Synthetic dyes are synthesized from petro chemical sources through hazardous chemical processes which pose a threat to the environment (Ekong, 2016) Researches have shown that synthetic dyes are suspected to release harmful chemicals that are allergic, carcinogenic and detrimental to human health (Mansour, 2013). Textiles industries produce large amounts of polluted effluents that are normally discharged to surfaces of water bodies and ground water aquifers. This chemical waste contribute several damages to the ecological system of the receiving water surfaces, creating a lot of problem to ground water resources. In 1996, Germany became the first country to ban certain azo dyes. As the world demand for fibres and safety dyes increases, consumers became conscious of environmental hazards posed by synthetic dyes which causes skin cancer, disorders and allergic contact dermatitis. This condition of environmental consciousness had lead to the rebirth of interest in natural dye (Kultam and Gokhale, 2011). There has become the necessity to develop natural dyes as they have better bio-degradability with the environment. They are non-toxic, non-allergic to the skin, non-carcinogenetic, easily available and renewable (Thiyaparajan, Balakrishn and Tarnilaras, 2015). For proper technical extraction and application of natural dyes, efficient method of extraction and adequate use of solvents that will be able to extract the dye from the plant has to be employed. This has led to so many research works carried out across the world on the application of natural dyes as important alternatives to synthetic dyes (Acguah and Oduro, 2012).

The African continent is rich in different plant species with potential to produce novel natural products with dye-yielding properties (Wanyama et al, 2014). The authors reported that India alone has abundance of dye-yielding plants from whose different parts extraction of colour components for textile application and commercialization are gaining prominence in that part of the world. Nigeria has abundant resources in terms of plants which contain dyes in parts such as the roots, bark, leaves, seeds, fruits and flowers. One of such plant is guinea corn.

Guinea corn (sorghum bicolor) belongs to the grass family, Poaceae. The genus sorghum is one of about 100 genera of Poaceae. The genus is in the sub family, panicodeae and the tribe, andropogoneae. It is called kopo-mopo-re-corn in Yoruba, millomaize in United states (U.S), dura in Sudanm great millet and guinea corn in West Africa, kafir corn in South Africa, Mtama in Eastern Africa, and jowar in India (FAO, 1995 in Osabohieri, 2014). Sorghum is known by inflorescence (lead) and grain (Fruiter edible seed) in the fern of a panicle, spikelets borne in pairs and extensively branching roots. It is particularly more grown in warmer climates of the world. As grain, sorghum is used as food for people, livestock and poultry. It is the fifth most important cereal crop in the world, sorghum grass family have hollow stems (Culms) that are plugged at intervals (the nodes), with leaves arising at the nodes. The leaves generally are differentiated with a lower sheath hugging the stems from a distance and a blade. Sorghum bicolor originated in Northern Africa and can grow in arid soil and withstand prolonged droughts. It grows in clumps that may reach over four meters high. The grain (seed) is small, reaching about 3 - 4 mm in diameter. The colour of the special seeds varies from white through red and brown, pale yellow to deep purple brown etc.

The United states is the world producer of sorghum followed by India and Nigeria (FSD, 2007). In U.S, Australia and Argentina, Its growth habit is similar to that of maize, but with more side shoots and a more extensively branched root system. It is generally used as food, fodder, in the production of biodiesel, construction of brooms and brushes etc.

Guinea corn leaf is an important source of carbohydrate, protein and minerals such as calcium, selemum, manganese and iron in which the bioavailability depends on the level of interactions with various anti-nutrients (FAO, 2001). It is also rich in B-complex vitamins. Apart from its uses as food, it contains some chemical compounds that are protective against cancer, heart disease, heavy menstrual flow and tumor growth (Oyetaya and Ogunrotimi, 2012). Guinea corn leaves form an addition to the huge mass of environmental pollutants from crop harvest. Oyetaya and Ogunrotimi, (2012) discovered that guinea corn leaf is a potential source of nutrients and an essential antioxidant compound which could supplement human and animal diets instead of constituting a waste and source of environmental pollution. Therefore it is a good source for dye extraction.

In extracting dye from plants, different techniques can be used. Examples of such techniques are maceration and soxhlet extraction. Maceration is a technique used in extracting dye from plants that involves leaving the pulverized plant to soak in a suitable solvent in a closed container. Cold maceration is done at room temperature by mixing the plant with the solvent and leaving the mixture for several hours while occasionally shaking or stirring. Finally the extract in strained from the plant particles (Malidi and Altikriti, 2010, Neha and Vidya, 2011). Temperature in maceration can be cold or hot. This type of technique requires no special apparatus like the soxhlet.

Soxhlet extraction is a technique that places a specialized piece of glass ware in between a flask and a condenser. The refluxing soxhlet repeatedly washes the solid extracting the desired compounds into the flask. The technique is mostly carried out for colourant identification. The temperature of the instrument is always maintained well under the boiling point of the solvent used. Several cycles of the solvent is run as to extract all the compounds from the plant part for dye application.

Dye application is the widely used procedure in adding colour to fibres, yarns and fabrics. When a fabric is exposed or introduced into the dye bath the item absorbs the molecules of the dye. Any excess dye that remains on the outside of the fabric can bleed or become sensitive to surface abrasion, chemical additives or mordants such as salt and alum which most times are used to facilitate absorption of dye into cotton fibre. Colouring matters extracted from plants has diversified exceptions and have little or no colouring power by themselves except when used in conjunction with a mordant. Osabohien (2009) confirmed that many of the natural dyes have poor affinity for textile materials unless they are treated with a mordant.

Mordants are chemical agents which allow a reaction to occur between the dye and the fabric thereby aiding in fixing the colour to the fabric. Mordants fix the dye to a substrate by combining with the dye pigment to form an insoluble compound (wipperlinger, 2004). Obenewaa (2010) commented that mordant is an essential part of the dyeing process. It is very essential except for plants which contain a lot of tannin and do not necessarily require mordants. Not all dyes accept mordants. Natural dyes can be substantive and adjective. Substantive dyes like lichens and walnut need no mordant to help them adhere to the fabric. Adjective dyes do. The mordant joins with the fabric and the dye to set the colour permanently. It enters deep into the fiber, and when the dye is added they combine to form a good colour (Earth Guild, 2016).

Mordants that are regularly used are ALUM (potassium aluminum sulfate), IRON (fereous sulfate) TIN (stannous chloride) BLUE VERTICAL (Copper sulfate) TANNIC ACID, GLAUBER'S SALT, and CREAM OF TARTAR. Alum is the most common mordant. It does not affect colour. It can be used with cream of tartar which helps evenness and brightens slightly. Iron is also called copperas. It sadden or darken colours, bringing out green shades. Cotton and wool are dyed before mordanting with iron when darker shades are required and Tin blooms or brightens colour. Regular used with cream of tartar. Tin is poisonous. It is a good additive. Blue vitriol sadden the colour and brings out green. It is a good additive. It is also poisonous. Tannic acid is a good mordant for vegetable fibers. Cream of tartar is used in some cases as additives to help in getting evenness and brightens slightly. Apart from textile dyeing, natural dye extract can be used to impart colour on foods.

Food has always been assessed firstly by its colour when it comes to desirability. The act of eating is a multi-sensory experience, synthesizing perceptions of sight, taste, smell and touch, colour provides visual information about a food quality and condition, and influences the perception of its flavor. In nature, colour is determined by a food's inherent qualities, indicating types of flavor and degrees of sweetness, ripeness or decay (Obenewaa, 2010). Humans have continued to add or change the natural colour in food from very early times and for a variety of reasons such as aesthetic purposes to increase appetite and appeal, for symbolic effects, to make a less desirable food seem more desirable, and to mask defects. From ancient times, other varieties of food colourants have been derived from plant, animal, and mineral sources.

This study was based on using the guinea corn leaves for dye extraction that is environmentally friendly. The extraction was aimed at using different solvents and different methods of extraction to ascertain the most effective means of extracting the dye pigments from the plant. Furthermore, the study was also focused on mordanting the substrate with alum before dyeing. The study also used the extracted dye to impart colour on foods.

Materials and Methods

Materials

The dried leaves of the Guinea corn used for the work was purchased from New Market in Aba South, Abia State. The cotton fabric, baking soda, Aceton, and alum were obtained from old market in Aba South of Abia State. All chemicals used as solvent for extraction were of analytical grade and required no purification.

Equipment/instrument

Apparatus used for the experiments include: Volumetric flask (100ml/ 500ml),Beakers of varying sizes, Graduated measuring cylinder of volume size 10,20,25 and 100ml, Soxhlet apparatus, Heating mantle, Weighing balance, Pot, Bath, Rotary evaporator. The equipments were obtained from the chemical laboratory of the department of chemistry, University of Uyo, Uyo Akwa Ibom state, Nigeria. Water was provided in the chemistry laboratory.

Treatment of Sample

The plant sample was washed with distilled water to remove dirt and later checked for final spots on the leaves which may produce adverse effects on the extraction process of the dye stuff. The guinea corn leaves sample was reduced to smaller pieces using knife. See below.

Samples of guinea corn leaves

Treatment of Substrate

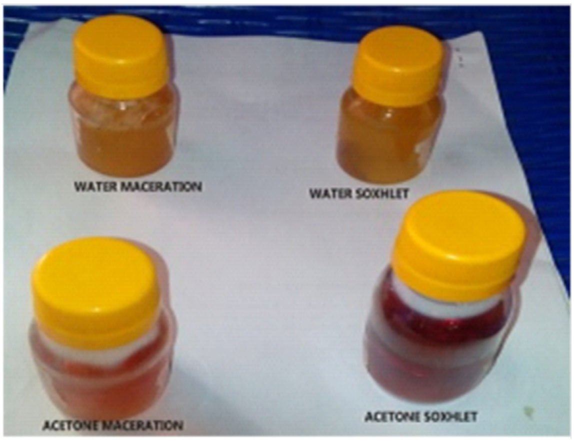
Scouring the substrate: The substrate (White Cotton) was simmered in a solution of dish soap. This removed the oil, wax or dirt that might interfere with dye adhering to the fiber. After, the substrate was rinsed properly.

Mordanting the substrate: In order to charge the substrate to be dyed, potassium Aluminum sulphate which is locally known as alum was used for the treatment. Two litres (2ls) of water was filled in a pot, 1 table spoon (1 Tb) alum and 1tsp baking soda were added to it plus the substrate. This was brought to a boiling point of 100°C with constant starring and simmered for two hours. The substrate (cotton Fabric) was allowed to cool off and soaked in the alum solution overnight.

Methods of Dye Extraction
Cold Maceration
100gs of the sample (guinea corn leaves) was added to a beaker (500ml) containing 300ml of distilled water and agitation was done at intervals for 48 hours for complete extraction. The resultant mixture was filtered using cotton wool and filter funnel. Similar experiment was conducted with acetone to compare the extraction power of the solvents. The temperature and the PH of each extract was taken and recorded.

Soxhlet Extraction
The dried leaves of guinea corn were gathered, chopped into smaller pieces and measured quantities of pulverized samples were fed into the soxhlet extractor. This method of extraction is carried out when a compound of low solubility needs to be extracted from a solid mixture. This technique places a specialized piece of glassware in-between a flask and a condenser. The refluxing solvent repeatedly washes the solid extracting the desired compound into the flask. The soxhlet extraction was carried out for colorant identification. Soxhlet extractor comprises of the condenser, thimble, siphon tube, solvent tube, round bottom flask and the heating mantle. 100g of each of the samples was measured into the thimble and 300ml of acetone 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 the sample and the temperature for extraction was 56.05˚c on the sample which is the boiling point of acetone. The same procedure was used for the extraction using water as solvent with a temperature of 100˚c. Several cycles of solvent were run so as to extract all the compounds from the leaves. Rotary Evaporator was used to remove excess solvent leaving the dye in dry state. The percentage yield, pH value and colour were determined for the purified dye samples. See below

Sample of Dye Extract from Guinea Corn Leaves



Extraction Yield Percentage
The extraction yield was measured. This was done by measuring the solvent's efficiency to specific components from the original material (Murugan and parimelahagan, 2014). This was defined as the amount of extract recovered in mass compared to the initial amount of whole plant. This was presented in percentage (%).

Dyeing Procedure

All the extracts were obtained in both aqueous and powdered form. 5g of the concentrated dye from the leaves of the guinea corn was mixed with 100ml of distilled water and acetone respectively for the dyeing process. The proportion of the fabric to be dyed with the dye mixture were constant in all the operations. The measurement of the fabric were 47cm by 25cm. 100ml of the liquid dye was measured into a 250ml beaker, the fabric was submerged into the beaker, the fabric heated between 40-60°c and stirred approximately for 20 minutes respectively. The dyed fabric was removed and aired for oxidation to take place for five minutes. It was rinsed separately in running water and dried under a shade. See below

Samples of Dyed Fabric

Water maceration	Acetone maceration	Water Soxhhlet	Acetone Soxhlet

Colour Imparted on Food Substrate

Macaroni Colouring

10g of macaroni was washed and put in the pot. 1.0g of dry dye sample was measured into a glass beaker, water was added and turned into the pot containing the pasta and stirred in order to obtain a uniform colour. The cooking was done for ten minutes and the pasta allowed to drain water very well. It was allowed for sometime to cool. See below

Yoghurt Drink Colouring

1.0g of the dry sample was mixed with 30 ml of water stirred very well and added to 170ml of homemade yoghurt in a container corked and shacked very well for 10 minutes. A homogenous mixture was formed with fine colour shade of blush as shown below

Batter

1.0g of dry sample was mixed with 10ml of water, stirred very well and added to the batter stirring vigorously so as to mix very well in it. A well dispersed colour of blush was obtained as shown below.

Result

Table 1: Cold Maceration Extraction of 100g of Material

Material	Technique	Temperature	PH	Time	Yield
Guinea corn leaves	Water Maceration	32˚c	5	48hours	38.6%
Guinea Corn leaves	Acetone Maceration	32˚c	5	48hours	49.8%

Table 2: Soxhlet Extraction of 100g of Material

Material	Technique	Temperature	PH	Time	Yield
Guinea Corn leaves	Water Soxhlet	100˚c	5	6hours	52.1%
Guinea Corn leaves	Acetone Soxhlet	56.05	5	6hours	61.8%

Table 3: Colour obtained with Different Solvents and method of Extraction

Material	Technique	Type of textile	PH	Temperature	Colour observed after filtration	Colour observed after dyeing
Guinea Corn Leaves	Water maceration	white cotton	5	32°C	Wine	Blush
	Acetone maceration	white cotton	5	32°C	Slightly reddish brown	Brick
	Water soxhlet	white cotton	5	100°C	Slightly deep Wine	Deep blush
	Acetone soxhlet	white cotton	5	56.05	Deep reddish brown	Deep brick

Table 1revealed that in water maceration the dye yield was 38.6% under 32°C with PH 5 and the time observed was 48 hours. For acetone maceration the yield was 49.8% maintaining the same temperature and PH.

Table 2 showed result on soxhlet extraction. The water soxhlet of the plant at 100°C within the PH of 5 gave the yield of 52.1% and the time observed for the experiment was 6 hours. For acetone soxhlet the temperature was 56.05 as the boiling point and the same PH and time was maintained. It gave dye yield of 61.8%.

Table 3 revealed the colour obtained from different solvents and methods. For water maceration, the colours observed after filtration and dyeing were wine and blush. In acetone maceration it was slightly reddish brown and brick. The water soxhlet gave slightly deep wine and deep blush after filtration and dyeing while acetone soxhlet gave deep reddish brown and deep brick.

Food Colouring

Table 4: Colour Imparted on Substrate

Dye extract	Technique	Substrate	Colour impart
Guinea corn leaf	Water soxhlet	Macaroni	Blush
Flour			
Batter	Blush		
Yoghurt	Blush		

Table 4 shows the different hues imparted on food such as macaroni, yoghurt, and batter. The colours improved the facial value of these foods and drink. The harmless nature of the dye as was extracted with distilled water and the nutrive values as having been researched by different scholars could allow for these applications (Osabohien et al, 2013, FAO, 1995). The colour obtained was blush in all the foods and drink. Table 9 answered research question 5.

Discussion

The dye sample from the heartwood of leaves of guinea corn was found soluble in distilled water and acetone. It was observed that the dye concentration was high in the soxhlet extraction. This supports the fact that soxhlet extraction is a very efficient form of extracting colour from solid materials (Vankar, 2016). It may also be as a result of the temperature as the heat of the solvent comes in contact with plant the extracting power was more efficient. Temperature is the main factor which affects the extraction efficiency of dye from natural plant

(Kannanmarking, Uma and Rajarathinam, 2015). At higher a temperature water and acetone was able to extract larger yield of natural dyes. The hues obtained by maceration using acetone as the solvent gave brick and deep brick compared to maceration using water as solvent which gave blush and deep blush . It also supports the fact that maceration is preferable used with volatile solvents (Hans-Jorg Bart, 2016)

See Appendix 1.
The temperature of the various extracts were taken during extraction and dyeing. The variation in temperature range was from 30-100°c at PH 5. The variation in temperature range for maceration was 32-60°c while for soxhlet was 56-100°c. The difference in temperature between the distilled water and acetone is that acetone is very high in volatility (Usoro, 2001).

The colour observed after filtration and dyeing revealed distilled water plus sample in maceration as blush, Acetone + Sample in maceration as brick, distilled water plus sample in soxhlet as deep blush and acetone plus sample in soxhlet as deep brick.(See Table 3).

The colour imparted on food such as macaroni, batter and yoghurt drink contributed in improving the appearance and nutritive values of the food and drink. This is in line with Oyetayo and Ogunrotimi, (2012) and Osabohien (2014) that natural dye can be used to impart colour on foods thereby improving the appearance for customers to patronize as well as enrich the nutritive value..

Conclusion

This work showed that different hues of dye can be extracted from the leaves of Guinea corn. The process of the extraction was eco-friendly. The dye hue obtained has the dyeing potential which can be used as a source of textile dyeing. The colour shade can be obtained using different methods and different solvents. The research work unveiled that their properties are preferable to synthetic dye as they are not toxic. The use of mordant in textile application is found to be fruitful to improving colour adherence and shades of the dyes. Guinea corn leaf is a very good plant part that serves as a source of raw material for obtaining shades of wine red dye that can be used for fabric dyeing in future. The study also unfolded the usefulness of dye obtained from guinea corn leaf using water soxhlet technique as food colouring product.

Recommendations

The advantages of researching natural dyes and their application on textiles will improve the art of creativity in the field of clothing and textiles. In view of the results of this study the following recommendations are made.

1. The clothing and textile sections in secondary schools, colleges of education and universities can use the result of the study as a resource material for teaching creative skills in dyeing fabrics, fibres, yarns, tie-dye, batik etc.
2. Instructors should use the study to educate the students on the use of dye that is eco-friendly.
3. The government should sponsor research work on natural dye exploration as this will help to safe guard our environment from the damage caused by the use of synthetic dyes.
4. It is recommended that dye obtained from guinea corn leaf should be used for food colouring.

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