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Extraction and Application of Natural Dye from *Nauclea Diderrichii* Heartwood

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Abstract

The study extracted dye from *Nauclea diderrichii*; ascertained which method and solvent extracts the dye more efficiently. The extracted dye was applied to cotton fabric treated with the mordant, alum. The experiment was carried out in chemistry laboratory University of Uyo. Uyo Akwa Ibom State. Cold maceration and soxhlet extractions were employed using distilled water and acetone as solvents. The dye was produced in four states, samples in maceration yielded yellow and slightly deep yellow respectively while in soxhlet extraction, samples yielded deep yellow and highly deep yellow. The extracted dyes were applied on cotton fabric treated with alum. It was therefore concluded that the process involved in the extraction was eco-friendly. The yellow dye obtained has the dyeing potential which can be used as a source of textile dyeing.

Keywords: Extraction, Natural Dye, Soxhlet, Maceration, *Nauclea Diderrichii*.

Introduction

Natural dyes are substances from plants and animals that impart colour to foods, cosmetics, drugs hair, fibre, fur and polymers (Osobohien, 2009). The dyes are introduced to different items to upgrade their appearance and make it attractive for consumers to patronize. The use of natural colour for dyeing fabrics has been in practice since ancient time (Ibrahim *et al,* 2013). Our forefathers have been extracting dye from plants. These dyes can be obtained from plant leaves, flowers, root, bark, insect secretions and minerals. They were the only dye available to mankind for colouring textiles as far back as 1856 (Sujata and Raja, 2016). The roots, stem, leaves, flowers and fruits of various plants supplied vegetable dyes. Certain mollusks found on the shores of Mediterian sea supplied animal dye as the famous tyrian purple. The invention of synthetic dyes during 1856 – 1900 negatively affected the market of natural colorants as synthetic dyes were cheaper and possess the quality of excellent fastness and obtainable in varity of shade (Samanta and Konar 2012). Before 1856 primitive dyeing techniques included sticking plants to fabric or rubbing crushed pigment into cloth. This methods became more sophisticated with time and techniques using natural dyes from crushed fruits, plants which were boiled into the fabric and which gave light and water fasteners were developed (Mansour, 2013).

Furthermore, in 1856 after the accidental synthesis of manveine by perkin in Germany and it's subsequent commercialization. Coal-tar dyes began to compete with natural dyes. As chemical techniques advances the manufacture of synthetic dyes became possible, leading to greater production efficiency in time of quality, quantity and the potential to produce low-cost raw materials. This graduated to the progressive replacement of natural dyes by synthetic dyes.

Synthetic dyes are synthesized from petro chemical sources through hazardous chemical processes which pose 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 huge amounts of polluted effluents that are normally discharged to surface water bodies and ground water aquifers. This chemical waste contribute several damages to the ecological system of the receiving surface water, 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 hazzards of 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 (Kulkama, 2011). There became 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 solvent 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 to synthetic dyes (Acgnah 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 is gaining prominence in that part of the world. Nigeria has abundant resources in terms of plant which contains dyes in parts such as the roots, bark, leaves, seeds, fruits and flowers. One of such plant is Nauclea diderrichii.

Nauclea diderrichii is an indigenous plant to moist tropical forest of West Africa like Nigeria, Ghana, Ivory coast, Liberia and Equatorial Guineas. It also grows in central Africa like Cameroon, central African Republic, Congo and Gabon. In some parts of eastern Africa like Uganda and Mozambique, Nauclea diderrichii is found and thrive best in humid tropical rainforest forest (Jonathan, et al, 2016). In Nigeria this plant is indigenous. It was one of the 22 trees species identified in the early stage of forestry practice in Nigeria as a tree species of major economics importance until the 1960's and several attempts has been made towards regenerating it through artificial means. Nauclea diderrichi (De wild) merr. belongs to the family of Rubiaceae. Some of its indigenous names are opepe in Nigeria, Badi in Ivory coast, kusia in Ghana and Bilinga in Gabon (Wagenfuhr, 2000). The fruit are about 3-4 cm in diameter and are covered with polygonal honeycomb. The fruits are edible by both men and animals and contains small seeds in it. Nauclea diderrichii is a forest species which can reach 35-40 of height (Pitekelabou Aidam and Kokou, 2015).

According to the classification of the Nigeria standard Organization, it is one of the strongest wood in Nigeria (Onyekwelu, 2007). It is used for drums, poles, doors, chairs, tools and mortars. The wood traded as bilinga, opepe or badi is suitable for heavy construction of bridges, heavy flooring, boat building, railway sleepers, sporting goods, toys, agricultural implements, draining boards, fuel wood and charcoal production. The pulp of the infructesence is edible, but not much eaten. In Nigeria the leaves are fed to livestock. Furthermore it is widely used in local traditional medicine in the treatment of anemia, stomachache, malaria, fever jaundice, appetizer and diueretic, gonorrhea menstruation problems, hepatitis and as wash for treatment of measles (Opuni et al, 2012). The heartwood is orange or golden yellow and can be used in producing dye. In extracting dye from plant, different techniques can be used. Examples of such techniques are maceration and soxhlet extraction.

Maceration is a technique used in extracting dye from plant 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 with occasional shaking or stirring. Finally the extract in strained from the plant particles (Mahdi and Altikriti, 2010). Temperature in maceration can be cold or hot. The type of technique require 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 compound 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 compound from the plant part.

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 fibre can bleed or become sensitive to surface abrasion, chemical additives or mordents such as salt and alum are most times used to facilitate absorption of dye into cotton fibre. Colouring matter extracted from plant have diversified exception 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 mordant.

Mordants are chemical agents which allows a reaction to occur between the dye and the fabric thereby aid 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 (wipplinger, 2004).

Obenewaa (2010) commented that mordant is an essential part in 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 sulphate), 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 our green shades. Cotton and wool are dyed before mordanting with iron when darker shades are required Tin blooms or brightens colour. Regular used with cream of tartar. Tin is poisonous. It is a good additive. Blue vitriol sadden colour and bring's 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.

This study was based on using the heartwood of *Nauclea diderrichii* for dye extraction that is environmental friendly. The extraction was aimed as using different solvents and different methods of extraction to ascertain the most effective means of extracting the dye pigment from the plant. Furthermore, the study was also focused on mordanting the substrate with Alum before dyeing.

Research questions:

1. Which solvent is more effective in extracting the dye from the plant?
2. Which method is more effective in extracting the dye from the plant?
3. What are the color shades obtained after dyeing with mordanted fabric?

Materials and method

Materials: The dried yellow heartwood of Nauclea diderrichii 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. Equipment used for the work were volumetric flask (100ml, 500ml), Beaker (500ml) graduated measuring cylinder of volume size 10, 20, 25 and 100ml, Soxhlet extractor, pot and bath. The equipment 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's and later checked for final spot on the heartwood which may produce adverse effect on the extraction process of the dye stuff. The heartwood sample was reduced in size using knife.

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 was 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 soak in the alum solution over night.

Method of dye extraction

Cold Maceration

100gs of the sample 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.

Maceration process

General procedure: -cut plat material into small pieces.
- Place in vessels
- Add whole of the selected solvent and cover the vessels
- Allow to stand for 48 hours and shake occasionally.
- Strain off liquid (dye) into a suitable container
- Press solid residue for more liquid
- Filter the liquid using muslinet

(Adapted from Mahdi & Altikriti, 2010)

Soxhlet Extraction

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 sample 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 sample and the temperature for extraction was 56.05˚c on the sample which is as the boiling point of acetone. The same procedure was used for the extraction using water as solvent with a temperature of 100˚c which is the boiling point. Several cycles of solvent were run so as to extract all the compound from the heartwood. Rotary Evaporator was used to remove excess solvent leaving the dye in dry state.

Extraction yield percentage

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

Dyeing Procedure

All the extracts were obtained in both aqueous and powder form. 5g of the concentrate dye from the heartwood of Nauclea diderrichii 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 and 32˚C as the temperature 100ml of the liquid dye was measured into a 250ml beaker, the fabric was submerged into the beaker, the fabric heated between 30-39˚c and stirred approximately for 20 minutes respectively. The dyed fabric was removed and aired for oxidation to take place for five minutes. They were rinsed separately in cold water and dried under a shade.

Identification of color shade

The extracted dye and dyed fabric were subjected to physical examination. The dye extract labelled WM (Water Maceration), AM (Acetone Maceration), WS (Water soxhlet) and AS (Acetone soxhlet). To achieve this, a twenty man panel consisting of post graduate students from fine Arts anh Home economics department of the university of Uyo was used. The panelist were randomly selected to evaluate the dyed fabrics. The sensory analysis instrument was developed using a 5 point scale of 1=Yellow, 2=slightly deep yellow, 3=Hue not decided, 4=Deep yellow and 5=Highly deep yellow.

Statistical Analysis

The obtained scores from the sensory evaluation were subjected to descriptive statistical analysis using mean and standard deviation. The acceptability of each measured attribute were based on a 5 point scale rating. Attribute means score greater than 3.00 was considered acceptable while attribute mean score less than or equal to 3.00 was considered unacceptable.

Result

Table 1: Maceration extraction of 100g of material

Materials	Technique	Temperature	PH	Time	yield
Nauclea diderrichii Heartwood	Water Maceration	24˚c	5	48 hours	32.4%
Nauclea diderrichii Heartwood	Acetone Maceration	24˚c	5	48 hours	42.8%

Table 2: Soxhlet extraction of 100g of material

Material	Technique	Temperature	PH	Time	Yield
Nauclea Diderrichii	Water Maceration	100˚c	5	6hours	52.1%
Nanclea diderrichii Heartwood	Acetone Soxhlet	56.05	5	6hours	61.8%

Table 3: Colour obtained with different solvents and method of extraction

Material	Tedluiquel	Type of textile	PH	Temp	Colour observed after filtration	Colour observed after dyeing
Macerat ion	Water maceration	white cotton	5	24˚C	Yellow	Yellow
Diderric hii	Acetone maceration	white cotton	5	24˚C	Slightly deep yellow	Slightly deep yellow
	Water soxhlet	white cotton	5	100˚C	Deep yellow	Deep yellow
	Acetone soxhlet	white cotton	5	56.05	Highly deep yellow	Highly deep yellow

Table 1 revealed that in water maceration the dye yield was 32.4% under 24˚C with P^H 5 and the time observed was 48 hours. For acetone maceration the yield was 42.8% maintaining the same temperature and P^H.

Table 2 showed result on soxhlet extraction. The water soxhlet of the plant at 100˚C within the P^H 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 P^H and time was maintained. It gave dye yield of 61.8%. Therefore Table 1 and 2 answers research questions 1 and 2.

Table 3 revealed the colour obtained from different solvent and method. For water maceration color observed after filtration and dyeing was yellow. In acetone maceration it was slightly deep yellow. The water soxhlet gave deep yellow after filtration and dying while acetone soxhlet gave highly deep yellow. These are shown in the pictures above.

The mean ratings of color shade of fabrics dyed using WM,AM,WS and AS and thee standard deviations are presented in Table 4 below.

Table 4: Mean ratings of color shades of WM, AM, WS, and AS after dyeing

S/N	color shades	solvent/method	mean	SD	remark
1	yellow	WM	4.15	0.93	Accepted
		AM	1.15	0.37	Not accepted
		WS	1.30	0.47	Not accepted
		AS	2.75	0.97	Not accepted
2	Deep yellow	WM	2.35	1.98	Not accepted
		AM	3.60	0.75	Accepted
		WS	1.80	0.70	Not accepted
		AS	1.75	1.21	Not accepted
3	Hue not decided	WM	1.70	0.98	Not accepted
		AM	1.15	0.37	Not accepted
		WS	1.30	0.47	Not accepted
		AS	1.15	0.37	Not accepted
4	Slightly deep Yellow	WM	3.00	0	Not accepted
		AM	3.00	0	Not accepted
		WS	3.65	0.75	Accepted
		AS	2.75	0.97	Not accepted
5	Highly deep Yellow	WM	3.00	0	Not accepted
		AM	3.00	0	Not accepted
		WS	3.00	0	Not accepted
		AS	4.65	0.49	Accepted

Mean score > 3.00 is acceptable; attribute mean score < 3.00 is not acceptable.

Table 4 answers research question 3 which unfolded the fact that extraction carried out with water maceration yielded yellow hue with the acceptable mean of 4.15 and standard deviation of 0.93. For acetone maceration, water and acetone soxhlet mean were below 3.00 which informed unacceptability. For deep yellow color shade acetone maceration had an acceptable mean of 3.60 while water maceration, water soxhlet and acetone soxhlet had means below 3.00 depicting their unacceptability.

For undecided hue, their means were below 3.00 showing the unacceptability from the respondent. The result for slightly deep yellow water soxhlet was accepted with a mean of 3.65 and it is within the range of acceptability while water maceration, acetone maceration and acetone soxhlet had means below or equal to 3.00respectively revealing the unacceptability of the color shades.

Furthermore, for highly deep yellow, Table 4 showed that acetone soxhlet had the acceptable mean of 4.65 indicating that the color shade was highly deep yellow. Water maceration, acetone maceration and water soxhlet means were 3.00 respectively indicating that the color shades were not accepted as highly deep yellow.

Discussion

The dye sample from the heartwood of Nauclea diderrichii was found soluble in distilled water and acetone. It was observed that the dye concentration was high on 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 affect the extraction efficiency of dye from natural plant (Kannanmarking, Uma and Rajarathinam, 2015). At higher temperature water and acetone was able to extract larger yield of natural dyes. The yellow dye extracted by maceration using acetone as the solvent gave a slight deep yellow compared to maceration using water as solvent. It also supports the fact that maceration is preferable used with volatile solvent (Hans-Jorg Bart, 2016). The temperatures of the various extracts were taken during extraction. The variation in temperature range from 20-100˚c at P^{H} 5. The variation in temperature range for maceration was 22-24˚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, E.S., 2001).

The colour observed after filteration and dyeing revealed distilled water plus sample maceration as yellowish, Acetone plus Sample maceration as slight deep yellow, distilled water plus sample soxhlet deep yellow and acetone plus sample soxhlet as highly deep yellow.

Conclusion

This work showed that yellow natural dye can be extracted from the heartwood of *Nauclea diderrichii*. The process of the extraction was eco-friendly. The yellow dye 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 works unveiled that their properties are preferable to synthetic dye as the are not toxic. The use of mordant in textile application was found to be fruitful in improving colour shades of the dyes. Nauclea diderrichii heart wood is very good plant part that serve as a source of raw material for obtaining yellow dye that can be used for fabric dyeing in future.

Recommendations

The advantages of researching on 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 fabric, fibres, yarns, tie-dye, batik etc.
2. Instructors should use the study to educate the student on the use the study to educate the student 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 use of synthetic dyes.

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