The Utilization of *Pentaclethra Macrophylla* Dyes: A Promising Approach to Environmental Conservation Through Extraction, Characterization and Application in Textile

Onofuevure, V.[1*], Salubi, L. O.[1], Oyagbarha, E.[2], and Egbai, R. I.[3]

[1]Department of Chemistry, Delta State University, Abraka, Nigeria

[2]Department of Chemistry, College of Education, Warri, Nigeria

[3]Department of Chemistry, Nnamdi Azikwe University, Awka, Nigeria

[*]Corresponding Author

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ABSTRACT

The study reviews the potentiality of eco-friendly dyes from the stem bark of Pentaclethra macrophylla as a natural dye source. The objective was to extract, characterise and apply the natural dyes on nylon 6,6 fabric. The extraction process involved Soxhlet extraction techniques, followed by filtration and concentration of the dye extracts using rotary evaporation. The dye components extracted and isolated from the plant material were characterized by thin-layer chromatography (TLC), column chromatography, FTIR, ^{1}HNMR, and ^{13}CNMR techniques. The crude extract was applied to the dyeing of the fabric using pre-mordanting and post-mordanting techniques and mordanted with ferric chloride and copper sulphate. Of the two solvents used, ethanol was the best solvent for the extraction, with a maximum absorbance value of 1.7 at a temperature of 60 °C. The FTIR study suggested the absorption band frequencies 3283 cm^{-1} are assigned to OH, 2981 cm^{-1} to C-H, 1638 to C=C, and 1086-1043 cm^{-1} to the C-O functional group in the dye structure. The proton and carbon-13 NMR in the dye extract suggested the presence of 1.20 (RCH_3), 3.12 (CH_3), 46.7 (CH_3), 46.9 (CH_3), 47.1 (CH_3), 47.8 (CH_3), 47.9 (CH_3), 108.6 ($R_2C=CR_2$), and 144.5 (Ar carbon). The light fastness and wash fastness properties of the dyed fabrics were evaluated; the results indicate moderate to good fastness grades in the range (4-5) for light fastness and good grade (4) for wash fastness. The study concluded that natural dyes from Pentaclethra macrophylla stem bark provide a promising opportunity for sustainable textile production.

Keywords: Extraction, Characterization, Application, Pentaclethra macrophylla, Nylon 6,6.

INTRODUCTION

Nature in its appearance is full of fascinating colours that appeal to human eyes, and without these colours life would have been uninspiring. Dyeing with natural dyes is an ancient art which predates written records (Ado et al., 2014). Environmentally conscious consumers have become more concerned about the health and environmental impact of synthetic dyes in the textile industry (Faizal et al., 2011). Owing to these concerns, there has been an increasing tendency towards the use of sustainable and environmentally friendly natural dyes (Shahid-Ul-Islam and Sun, 2017). Natural dyes are obtained from renewable resources as compared to synthetic dyes, which are derived from non-renewable petroleum resources. They are biodegradable, and the residual vegetal matter left after extraction of dye can be easily composted and used as fertiliser (Chattopadhyay et al., 2013). There is effort in searching for eco-friendly dye sources that are environmentally conservative (Maleki and Barani, 2019); P. macrophylla is one such natural dye source. The plant is relatively large with average height and belongs to the family Fabaceae (Onyekwelu and Stimm, 2015), and it is the sole member of the genus

(Pentaclethra) occurring naturally in the humid lowlands of West Africa. The common name in English is 'oil bean tree' (Afia, 2020). It contains some phytochemicals such as flavonoids, saponins, alkaloids, tannins and glycosides (Enemchukwu et al., 2021). The bark is greyish to dark reddish brown, and the compound leaves possess a stout angular petiole (Asoegwu et al., 2006). The research objective was to extract natural dye from Pentaclethra macrophylla plant material using the Soxhlet technique process, characterise the isolated compound using FTIR, proton and carbon-13 NMR analyses and apply the dye extract on the nylon 6,6 fabric by adopting pre-mordanting and post-mordanting dyeing techniques. The challenges associated with natural dyes in dyeing are low exhaustion colours and poor fastness properties. Metallic mordants such as ferric chloride and copper sulphate are usually applied to improve the colour strength and fastness properties and to obtain multiple shades (UI-Islam et al., 2018; Adeel et al., 2018; Barani, 2018). Thus, the exploitation of Pentaclethra macrophylla will reinforce and enhance knowledge and databases, as research efforts in this area have been negligible.

MATERIALS AND METHODS

Chemicals and Reagents

Laboratory grade ferric chloride ($FeCl_3$) and copper sulphate ($CuSO_4$) were used as mordants. The chemical used for extraction and purification purposes; ethanol (C_2H_5OH), acetone (CH_3COCH_3), chloroform, methanol, and silica Gel 60-120 mesh of analytical grade were purchased from Meck.

Reference detergent A soap (3 g/L) was used to wash the fabric. Soap (5 g) in 1000 ml of distilled water and Soda ash (2.5 g) was prepared in 1000 ml distilled water for wash fastness test.

Plant Material

The plant stem barks were harvested from their matured plant at a farm behind Osubi Airport, Osubi, in Okpe Local Government Area of Delta State, Nigeria by ($5^{0}35^{1}46.8^{11}$ N Latitude and $5^{0}49^{1}13.4^{11}$E Longitude). The plant was identified as Pentaclethra macrophylla from the Department Botany, University of Calabar, Calabar and was given a voucher number (CAL/HBR:1847) which was deposited at the Departmental Herbarium. All the procedures and collection of plant material was done in accordance with local and national guidelines and regulations. The plant materials were rinsed with distilled water to remove dirt, sun dried, and then crushed to powdered form by using grinding machine to facilitate better dye extraction.

Extraction Procedure

In total extraction 10.0 g of the powdered samples was weighed and poured into a 250 ml flat-bottom flask in the inner part of the Soxhlet extractor, and 200 ml of ethanol was added and heated under reflux with a condenser using a heating mantle at various temperatures of 20 ^{0}C, 30 ^{0}C, 40 ^{0}C, 50 ^{0}C, 60 ^{0}C and 70 ^{0}C to generate absorbance. The process automatically repeated itself and the solvent in the dye extract was concentrated to obtain the dye extracts. Similarly, the experiment was repeated for acetone to compare the extraction power of the solvents (Jinasena et al., 2016). A plot of absorbance against temperature was carried out in each case of the extraction as shown in Figure 2-3

Purification Procedure

The ethanolic extract was analyzed to identify specific compounds. The extract was subjected to thin layer chromatography for separation using aluminium foil plates (UV 254) coated with silica gel layer of 0.25 mm thickness to know the actual solvents ratio that will be used for the column, a method described by (Ahmed et al., 2021). The solvents combination, dichloromethane and methanol in the ratio 2.5:1 v/v was used for the TLC, which revealed seven (7) spots on the TLC plate. The isolated extract was then purified using slurry method for the column chromatography. Elution process was carried out using a mixed solvents dichloromethane and methanol in the ratio of 70:30 v/v of suitable solvent. Fractions obtained from the columns were labeled A to F and their retention factor values were 0.86, 0.79, 0.76, 0.51, 0.79 and 0.89 respectively. TLC experiments showed that some of these samples still contained several spots that were again separated to obtain one spot on the TLC plate. Finally, the isolated compound was characterized by FTIR, ^{1}H and ^{13}CNMR.

Fourier Transform-Infrared (FTIR) Analysis

The solvent extractions of the organic compound were prepared and analyze using Agilent spectrophotometer with Model Number (Agilent Cary 630). Dye extract of the isolated compound was prepared, placed in the optical path of the instrument and scanned over the range of 4000-600 cm^{-1} frequency region at 1 cm^{-1} interval. The absorption bands were obtained and recorded as frequency (cm^{-1}) against percent transmittance (% T).The spectrum was interpreted to deduce the functional groups present in the compounds.

Nuclear Magnetic Resonance (NMR) Analyses

The purify dye extract was obtained and a concentration solution of the purified dye extract was prepared by dissolving with a suitable solvent $CDCl_3$ that is compatible with the instrument (Agilent 400 MHz NMR spectrometer). The NMR experiment was ran using a suitable pulse sequence ^1H NMR and ^{13}C NMR.

UV-Visible Analysis

A CARY 360 UV/visible Spectrophotometer was used for all spectrophotometric measurements. All measurements were carried out using quartz cells 10 mm at room temperature (25±2 ℃) using standard procedures (Jack et al., 2020) to detect the changes in their absorption (200-800 nm) were noted.

Preparation of the fabric

The nylon 6,6 material was heated in a soap solution 2 g/l at 60 ^0C for 30 minutes, and was washed thoroughly with water and air-dried at room temperature.

Preparation of stock solution

The mordants ferric chloride and copper sulphate were prepared at concentration of 3 g into 100 ml of distilled water, it was then transferred to volumetric flask. The strip of the nylon fabric material was added and heated to boiling. The content in the baker was removed, pressed and sun dried.

The amount to be taken from volumetric flask was calculated using the formula

$V = W \times P/C$.

Where V= volume which is unknown,

W= weight of sample (fabric)

P= percentage depth shade

C= concentration of stock solution.

Method of mordanting

The dyeing experiment was carried out with nylon fabric by adopting post-mordanting and pre-mordanting techniques i.e. the samples were treated with two metallic salts solution (ferric chloride and copper sulfate) before and after dyeing. The aim of mordanting was to enhance the adsorption of the dyes and ensure a strong bond between the dyes and the fabric.

Dyeing Procedure

The dyeing procedures were performed in accordance with the general dyeing method (Geetha and Judia Harriet Sumathy, 2013; Baaka et al., 2015). Weigh of 3.0 g of nylon 6,6 was cut and 100:3 liquor ratio was used containing 3% depth of shade and at a temperature of 70 ^0C, which was maintained throughout the period. Then 3 ml of dye samples were taken at 0, 5, 10, 15, 20, 25, 30, 35, 40 and 45 minutes, and it was used to generate absorbance value. After the duration of two hours, the nylon fabrics were removed, washed thoroughly with de-ionized water and air-dried.

Percentage of Exhaustion of dye extract

The absorbance of the dyes was determined before and after dyeing at the maximum wavelength (430 λmax) using CARY 630 UV/visible Spectrophotometer. The percentage exhaustion was calculated using the expression below:

$$\text{Percentage } Exhaustion = \frac{\text{absorbance before dyeing} - \text{absorbance after dyeing}}{\text{absorbance before dyeing}} \times 100\%$$

Fastness Test

Measure of colour fastness to washing

The wash fastness test was carried out using heating mantle machine and International Organization for Standardization (ISO) Standard procedure. Soap solution was prepared, 5 g of soap in 1000 ml of distilled water and 2.5 g of sodium carbonate was prepared also in 250 ml distilled water. Each of the dyed samples was cut into (5×5cm) and placed between two adjacent pieces of un-dyed fabric to form a composite. The essence of the undyed fabric was to enable the assessment of the degree of staining in the percentage depth shade of samples measured. The washing solution containing soap 15 ml, sodium carbonate 10 ml, distilled water 25 ml was mixed together to give a liquor ratio of 50 ml. The nylon fabric pieces were immersed in the washing solution and heated for 30 minutes at 40 ^{0}C to 60 ^{0}C. Finally, the composite was then removed, rinsed and dried. The assessment of colour change and staining was done with the standard “grey scale”.

Measurement of colour fastness to daylight

The samples were cut 5×5 cm and placed on light board. This test measures the resistance to fading of dyed textile when exposed to daylight. The samples were exposed to daylight for 24-72 hours, after that period of time; the samples were removed and compare the change with original (unexposed) samples. The changes are assessed by using a “blue wool scale”.

RESULTS AND DISCUSSION

Solvents Extraction of Natural Dyes

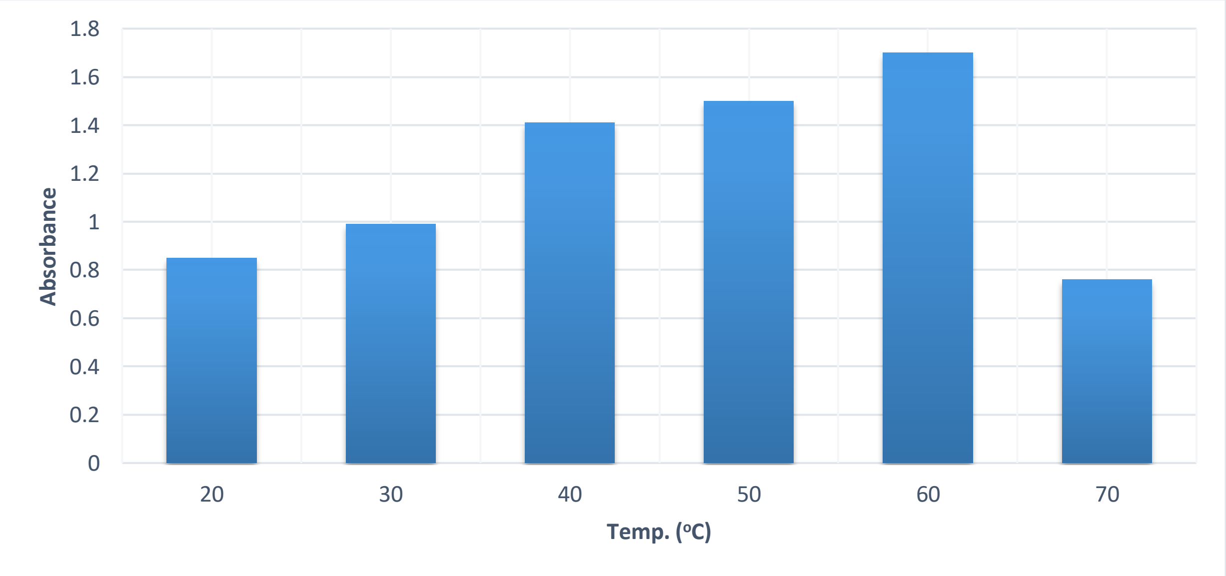

Figure 1: Effect of Temperature on Absorbance of Dye Extracts from P. macrophylla stem bark using Ethanol as Solvent

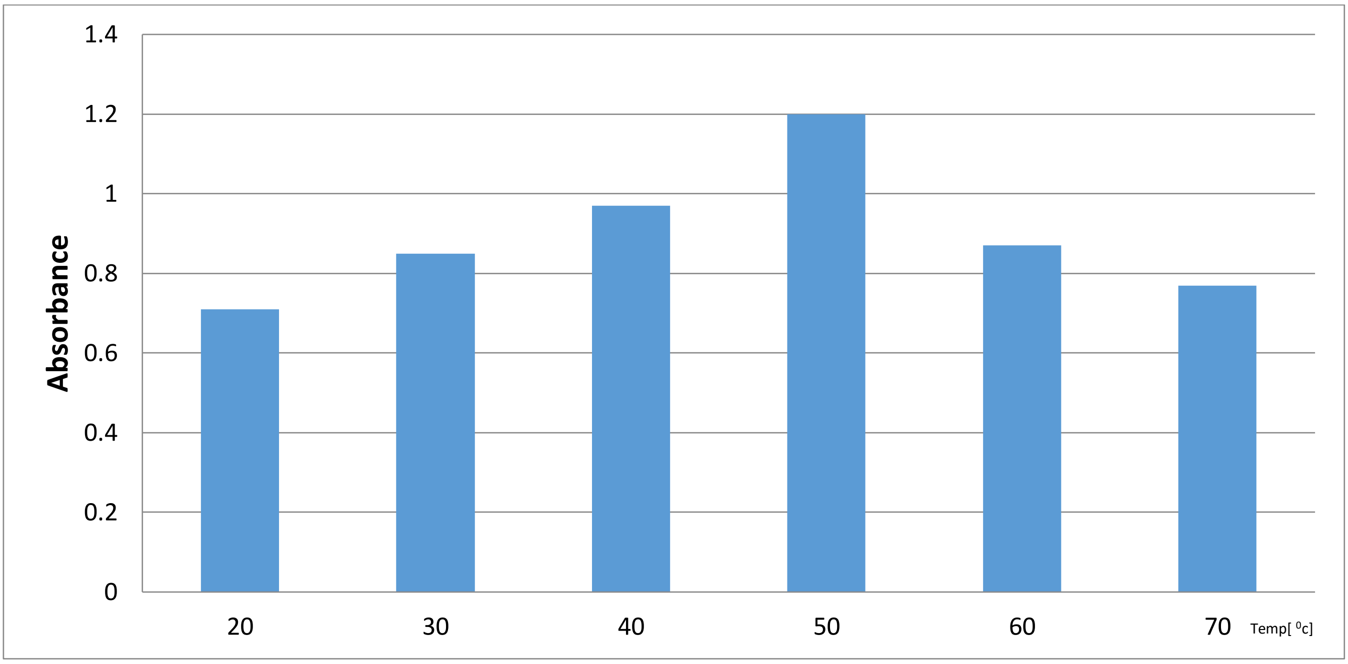

Figure 2: Effect of Temperature on Absorbance of Dye Extracts from P. macrophylla stembark using Acetone as Solvent

Percentage of Exhaustion of Dye Extract

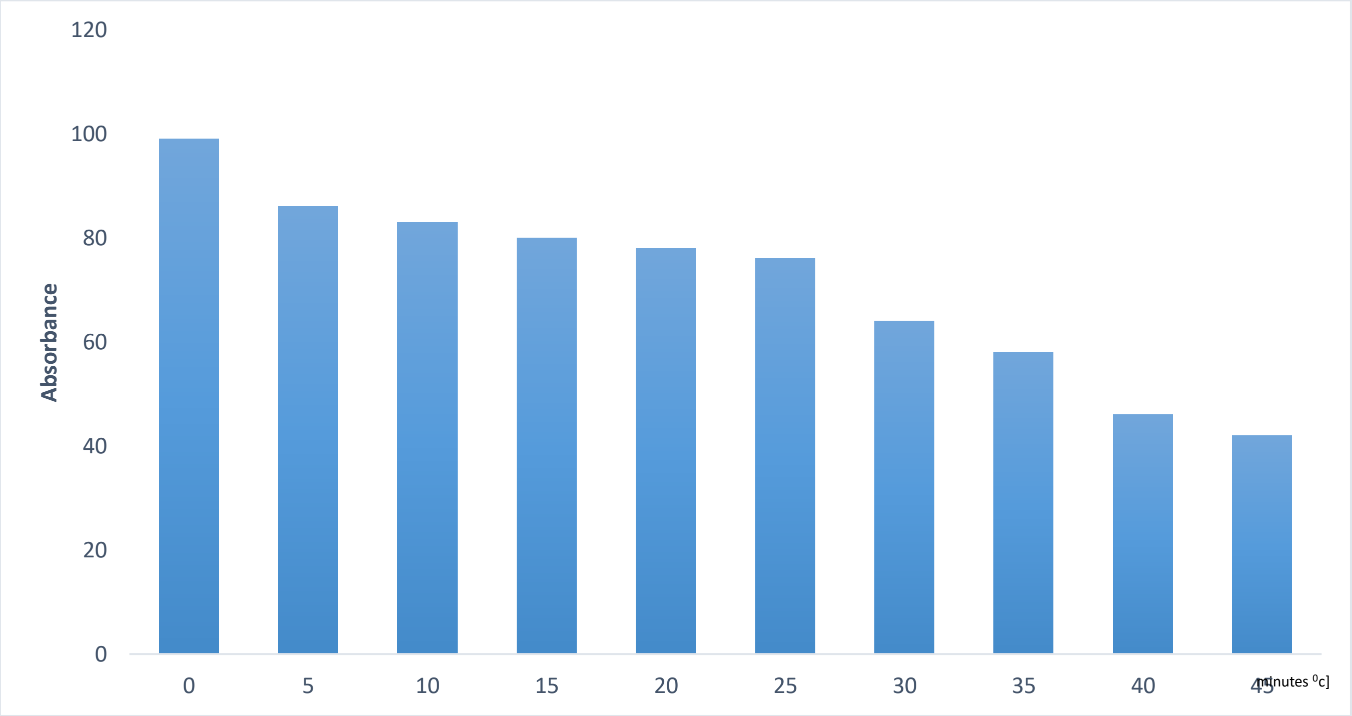

Figure 3: Percentage Exhaustion of Dye Extracts from P.macrophylla stembark at Constant Temperature 70 ^{0}C and at λmax 430

Spectroscopic Data of Dye Extracts

Table 1. Spectra Data of the Isolated Extract

Infrared (cm-^{1})	3283 (O-H of phenol), 2981 (C-H stretch of methine), 2907 (C-H stretch of methyls), 1638 (C=C), 1086-1043 (C-O stretch alcohol)
^{1}H NMR	1.20(RCH$_3$), 3.12(CH$_3$), 3.42-3.70 (ROCH$_3$), and 4.72 R (OH protons)
^{13}C NMR	46.9 (CH$_2$), 47.1(CH$_2$), 47.8(CH$_2$), 47.9 (CH$_2$), 108.6 (R$_2$C=CR$_2$), 144.5 (Ar carbon)

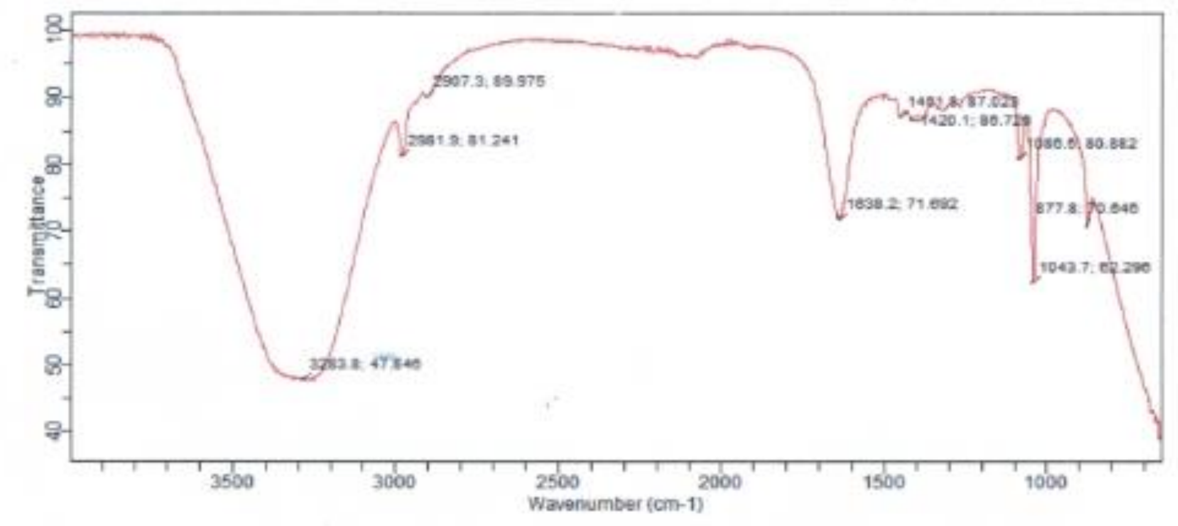

Figure 4: FTIR spectrum of dye extract from P. macrophylla stem bark

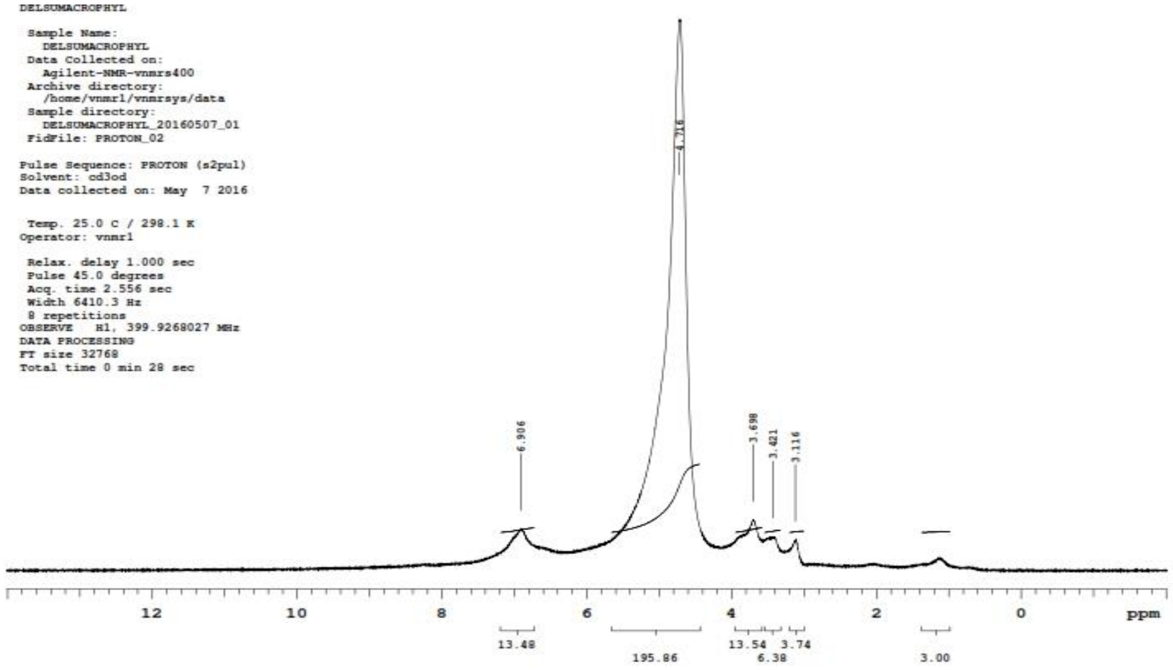

Figure 5: Proton nuclear magnetic resonance spectrum of dye extract from P. macrophylla stem bark

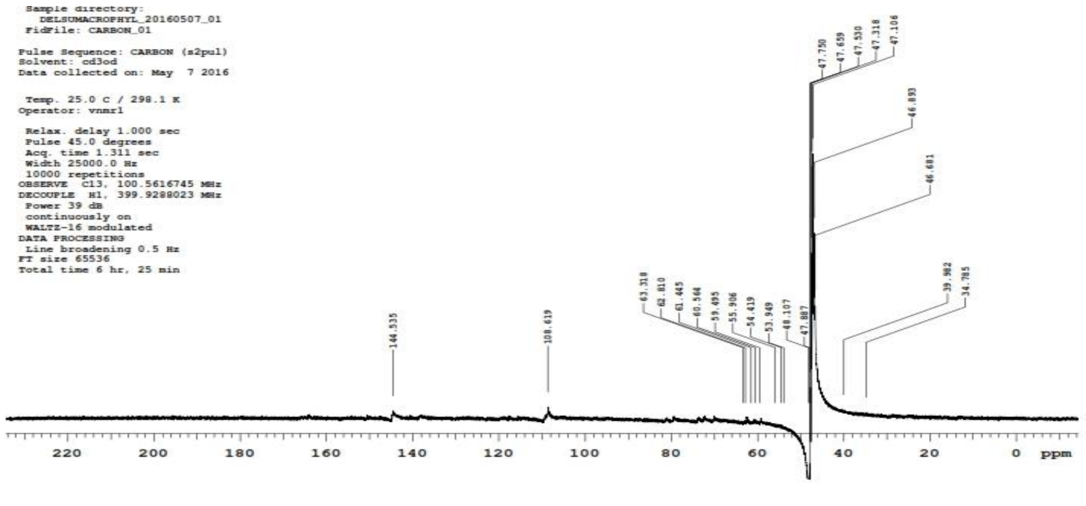

Figure 6: Carbon-13 nuclear magnetic resonance spectrum of dye extract from P. macrophylla stem bark

Table 2: Wash Fastness Properties of Dyed Fabric

Fabric	Plant sample	Premordanted Colour change	Postmordanted Colour change
Nylon	P. macrophylla stem bark	4	4

Key: 1- very poor; 2-poor; 3- fair; 4-good; 5- excellent

Table 3: Light Fastness Properties of Dyed Fabric

Fabric	Plant sample	Premordanted Colour change	Postmordanted Colour change
Nylon	P.macrophylla stem bark	4	5

Key: 1-very poor, 2-poor, 3-fair, 4-moderate, 5- good, 6-very good, 7-excellent, 8-outstanding

DISCUSSION

Solvents Extraction of Crude Dyes

The experiment (Figure 1-2) was carried out to compare the extraction power of the two solvents at various temperatures (Jinasena et al., 2016). The result shows the relationship between absorbance and temperature changes. Among the two solvents used, acetone at a temperature of 50°C, which is in agreement with Abd Razak et al. (2011), yields pigment with a maximum absorption value of 1.2, whereas ethanol at a temperature of 60°C provides the highest pigment yield for the extraction of plant material with an absorption value of 1.7 within the wavelengths of 200-800 nm. Ethanol as an organic solvent has been extensively used to extract natural organic dyes from various plant species (Peschel et al., 2006). The results show that ethanol, with the highest dye yield at a temperature of 60°C, was the best solvent for extracting natural dye from the plant material. Also, the results show that some natural organic dyes are stable at temperatures of 60°C (Razak et al., 2011). The increase in dye yield is as a result of an increase in temperature, which may be caused by an increase in pigment molecule diffusivity and pigment solubility; these properties are related to the increase in the internal energy of molecules, which increases the extract yield (Cacace and Mazza, 2003). Temperatures more than 60°C probably caused a decrease in the dye extract due to chemical structure degradation of pigments in the plant. Thus, a temperature of between 50 and 60°C proves to be the optimum temperature for extraction with high yield.

Characterization of Dye Extract

FTIR Spectrum of isolated compound

FT-IR analysis of the sample identify the presence of many organic functional groups present in P. macrophylla extract indicating their respective compounds from Table 1, the absorption band at 3283cm^{-1}is attributed to the phenolic or alcoholic OH group which is in agreement with Maiyo et al. (2019). The strong absorption bands appearing at 2981cm^{-1}- 2907cm^{-1} region can reasonably be ascribed to C-H stretching vibration of methyls or methines which is in agreement with Nielsen (2010). The characteristic band of C=C stretching vibration was located at 1638cm^{-1}. The bands at 1086-1043cm^{-1} are assigned to C-O stretch which confirmed the presence of OH group. It was found that FT-IR results were comparable with those reported by (Amir et al., 2018; Jabar et al., 2020; Mongkholrattanasit et al., 2021) reported band at 1638cm^{-1} and 1086-1043cm^{-1} in natural dyes for textile dyeing.

Proton and Carbon-13 NMR Spectrum of isolated compound

The likely proton environments shown in Table 2 by ^{1}HNMR spectrum plant extract are those of δ 1.20 (RCH_3), 3.12 (CH_3), 3.42-3.70 ($ROCH_3$), and 4.72 R (OH protons) respectively.

The carbon-13 NMR spectrum of the P.macrophylla stem bark dye extract gave absorptions at δ 46.7, 46.9, 47.1, 47.8, and 47.9 which indicates the presence of sp^2 Carbons (CH_2) probably attached to electronegative atoms. The absorption at δ 108.6 is attached to sp^2 carbons of unsaturated compound ($R_2C=CR_2$).The absorption band at δ 144.5 ppm is attributed to aromatic carbons.

Evaluation Fastness Properties

The study reports fastness to light and wash fastness of nylon 6,6 fabric dyed with P. macrophylla dyes.

Percentage of Exhaustion of Dye Extract

The dye extract absorbance was determined using the CARY 630 UV/visible Spectrophotometer, a method used by Onofuevure et al. (2025), before and after dyeing at the maximum wavelength (430) of each dye and is presented in Figure 3. The percentage of exhaustion in the dye bath was carried out. The liquor solution (3 ml) was taken at 0, 5, 10, 15, 20, 25, 30, 35, 40 and 45 minutes at a constant temperature of 70°C, and it was used to generate absorbance value. The amount of dye uptake and fixation on the nylon fabric was evaluated; the results show that an increment in time increased the transfer of dye molecules from the dye bath onto the fabric, as established by Oforghor et al. (2020). Thus, this demonstrates that the high degree of exhaustion in the dyed bath was due to the increase in temperature and time.

Colour fastness to Wash

Wash fastness of dye was influenced by the rate of diffusion of dye and the state of dye inside the fabric (Kanchana et al., 2013). Wash fastness properties of the dye extract on the fabric was carried out using mordants such as ferric chloride and copper sulfate. It was seen from Table 2 that natural dye extract was applied on nylon fabric before and after applying mordants. The wash fastness grades of the nylon fabric recorded Good (4) grade before and after dyeing which agree with the results obtained by Clark et al. (2023) was observed on the nylon fabric with very slight staining on the fabric adjacent. Thus the result indicates that the plant extract exhibits a better wash fastness performance on nylon 6,6 fabric.

Colour Fastness to Light

When the dye extract was applied on the fabric after the application of mordants such as ferric chloride and copper sulfate in table 3. The fastness grade (4) was observed on nylon fabric which was in alignment with the results recorded by Clark et al. (2023) which indicate appreciate fading. When the dye extract was applied to the fabric using Postmordanting technique, a fastness grade of (5) which agree with Shariful Islam et al., 2020), indicate moderate fading on the fabric adjacent side.

CONCLUSIONS

The present study investigates natural dyes from the stem bark of Pentaclethra macrophylla as a potential dye for dyeing nylon 6.6 fabric, with ethanol as the efficient solvent for extraction at a temperature of 60°C. The characterization results suggested the band at 3283 cm^{-1} corresponds to OH, 2981 cm^{-1} to C-H, and 1086-1043 cm^{-1} to C-O stretching vibrations present in the phytoconstituents of the extract (as shown in Table 1). The protons and carbon-13 NMR in the dye extracts are RCH_3, R_3CH_3, $ROCH_3$, and $R_2C{=}CR$. An increment in time increased the transfer of dye molecules from the dye bath onto the fabric. The crude extract exhibits a good and durable fastness performance on nylon 6,6 fabric, owing to phytochemicals such as flavonoids, saponins, alkaloids, tannins and glycosides present in the plant, according to Enemchukwu et al. (2021), that allow them to bind to the fabrics. The study concluded that dye extracts from the plant stem bark show acceptable fastness properties and hence can therefore be utilized on nylon 6,6 substrate.

Conflict of Interest

The authors declare that they have no conflict of interest regarding the publication of this manuscript.

REFERENCES

1. Abd Razak, N., Tumin, S. M. and Tajuddin, R. (2011). Effect of temperature on the colour of natural dyes extracted using pressurized hot water extraction method. America Journal of Applied Science, 8, 45-49.

2. Adeel, S., Hussaan, F., Rehman, N., Habib, M., Salman, S., Naz, N. and Amin, N. A. (2018). Microwave-assisted sustainable dyeing of wool fabric using cochineal based carminic acid as natural colourant. Journal of Natural Fibres, 5, 1-9.
3. Ado A, Yahaya H, Kwalli A, Abdulkadir R.S (2014). Dyeing of Textiles with Eco Friendly Natural Dyes: A Review. International Journal of Environmental Monitoring and Protection, 1(5), 76-81.
4. Afia K. (2020). A Review of Pentaclethra macrophylla (African oil bean) seed. International Digital Organization Science Resources, 2579-0781.
5. Amir, S. M., Zumahi, A., Hossain, K., Abser, M.N and Matin, R. (2018). Extraction of Different Natural Dyes from Flower Plants Perovskite Materials View Project Solar CellView Project." Materials for Renewable and Sustainable Energy, 9, (23)
6. Aseogwu, S., Ohanyere.,Kanu, O. and Iwueke C. (2006). Physical Properties of African Oil Bean Seed (Penthachethra Macrophylla). Agricultural Engineering International Journal, 8, 1-15.
7. Baaka, N., Ben, M. T., Hadder, W., Hammami, S. and Mhenni, M. F. (2015). Extraction of natural dye from waste wine industry: Optimization survey based on central composite design model. Fibers Polymers, 16(1), 38-45.
8. Barani, H. (2018). Modification of bentonite with different surfactants and substitute as a mordant in wool natural dyeing. Chiang Mai Journal of Science, 45, 492-504.
9. Cacace, J.E. and Mazza, G. (2003). Mass transfer process during extraction of phenolic compounds from milled berries. Journal of Food Engineering, 59, 379–389.
10. Chattopadhyay S. N., Pan N. C., Roy A. K., Saxenas, and Khan B. A. (2013). Development of natural Dyed Jute Fabric with Improved Colour Yield and UV Protection Characteristics. Journal of Textile History, 16(1), 23-43.
11. Clark, P. D., Otutu, J.O., Asiagwu, K. A., Ndukwe, G. I. and Idibie, C. A. (2023). Investigating the Feasibility of utilizing Pennisetum Purpureum Leaves Waste as a sustainable dye: Extraction, Characterization and Application on Textile. Scientia Africana Journal, 22(3), 231-246.
12. Enemchukwu, B.N., Ezeigwe, O.C, Orinya, O.F., Nwali, U.I, Chigbo, C.M. and Iloanya, E.L. Phytochemical evaluation and antimicrobial potential of methanol extracts of mistletoe (Loranthus micranthus) leaves brown on cola tree (Cola nitida) and oil bean tree (Pentaclethra macrophylla). Journal of Medicinal Plants Studies, 9(2), 28-32.
13. Faizal, Elly, and Burhaini. (2011), Indonesia told to produce more green product"(http://www.thejakartapost.com/news/2011/10/29/indonesia-told-produce moregreen products.html). The Jakarta Post Retrieved.
14. Geetha, B. and Judia Harriet Sumathy, V. (2013). Extraction of Natural Dyes from Plants. International Journal of Chemistry and Pharmaceutical Sciences, 1(8), 502-509
15. Jabar, J. M. Ogunmokun, A.I and Taleat. T.A. (2020). Colour and Fastness Properties of Mordanted Bridelia Ferruginea B. Dyed Cellulosic Fabric." Fashion and Textiles 7 (1), 1-13.
16. Jack, I. R., Clark, P. D. and Ndukwe, G. I. (2020). Evaluation of phytochemical, antimicrobial andantioxidant capacities of Pennisetum purpureum (Schumach) extracts. Chemical Science International Journal, 29(4), 1-14.
17. Jinasena, M.A.M., Amarasinghe, A.D.U.S., Amarasinghe, B.M.W.P.K. and Prashantha, M.A.B. (2016). Journal of National Science Foundation, 44, 11–21. Journal of Chemical Science, 9(2):1-7
18. Kanchana, R., Fernandes, A., Bhat, B., Budkule, S., Dessai, S. and Mohan, R. (2013). Dyeing of textiles with natural dyes- an eco-friendly approach. International Journal of Chemical Technology Research, 5 (5), 2103-2109.
19. Maiyo Kimutai Bernard, Munyendo Lincoln Were, Kiprop Kipchumba Ambrose, and MibeRichard, (2019). Extraction and Analysis of Spectral Properties and ChroMophoric Characterization of Natural Dye Extract from Barks of Terminalia brownii Fresen. (Combretaceae). American Journal of Applied Chemistry,7 (4), 116-122.
20. Maleki, H. and Barani, H. (2019). Extraction and antibacterial activity of Pulicaria gnaphalodes as a natural colourants: Characterization and application on wool fibers. Progress in Colour Colorants and Coatings, 12 (3), 45-154.
21. Mongkholrattanasit, R., Nakpathom, M. and Vuthiganond, N. (2021). Eco-Dyeing with Biocolourant from Spent Coffee Ground on Low Molecular Weight Chitosan Cross linked Cotton." Sustainable Chemistry and Pharmacy, 20:100389.

22. Nielsen, S. S, (2010). Food Analysis, chapter 14 and 23, Springer, 4th edition.
23. Oforghor, A., Osebuohien, A.U. and Nasiru L.U (2023). Extraction and application of dyes derived from Gmelina arborea leaves and their dyeing properties on cotton fabric. Nigeria Research Journal of Chemical Sciences, 8, 2682-6054
24. Onofuevure, V., Adamu, H.M., Adamu, D.A., Salubi, L. O. (2025). Optimization of solvents extraction and application of dye from Saccharum officinarum leaves on textile. International Chemical Science, 9(2), 1-7
25. Onyekwelu, J.C Stimm. B. (2015). Pentaclethra macrophylla Bentham, 1841. Enzyklopädie der Holzgewächse: Handbuch and Atlas der Dendrologie. 2015. 30;1–13
26. Peschel, W., Sánchez-Rabaneda, F., Diekmann, W., Plescher, A., Gartzía, I., Jiménez, D., Lamuela Raventós, R., S. Buxaderas, S. and Codina, C. (2006). An industrial approach in the search of natural antioxidants from vegetable and fruit wastes. Journal Food Chemistry, 97, 137–150.
27. Shahid-Ul-Islam, and Sun, G. (2017), Thermodynamics, Kinetics, and Multifunctional Finishing of Textile Materials with Colorants Extracted from Natural Renewable Sources. ACS Sustainable Chemical Engineering. 5, 7451-7466. doi:10.1021/acssuschemeng.7b01486
28. Shariful, I., Mominul A. and Shilpi, A. (2020), Investigation of the Colourfastness Properties of Natural Dyes on Cotton Fabrics. Department of Fabric Engineering, Bangladesh University of Textiles, Tegjaon, Dhaka, 1208.
29. Ul-Islam, S., Rather, M., Shabbir, M. N., Bukhari, M. A. and Khan, F. M. (2018). First application of mix metallic salt mordant combinations to develop newer shades on wool with Bixaorellana natural dye using reflectance spectroscopy. Journal of Natural Fibers, 15, 363-372.