NILEST JOURNAL OF SCIENCE, TECHNOLOGY, AND MANAGEMENT
(NIJOSTAM) VOLUME 3, ISSUE 1, NOVEMBER, 2024, pp. 100-108
eISSN: 3027-2580 www.nijostam.org

EXTRACTION AND CHARACTERIZATION OF NATURAL DYE FROM *AZADIRACHTA INDICA* (NEEM) BARK

[1]Oyelakin O.K., [1]Tagang J.I., [1]Babatunde O.A., [1]Akawu I., [1]Oko J.O., [1]Idoko G.O. and [1]Adamu S.

1. Nigerian Institute of Leather and Science Technology, Zaria, Nigeria

Correspondence: oyelakinbabatundesesan@yahoo.com

ABSTRACT

Natural dyes, derived from renewable biological sources, are increasingly recognized for their environmental benefits and applicability in various industries, including textiles, leather, and food. This study explores the extraction and characterization of dye from Azadirachta indica (neem) bark using ethanol and water as solvents. The yield of the extracted dyes was analyzed via UV-Vis spectroscopy, with maximum absorption wavelengths (λmax) ranging from 450–540 nm for water-extracted dye and 460–530 nm for ethanol-extracted dye. Functional groups were identified using Fourier-transform infrared (FTIR) spectroscopy, revealing O–H, C–H, C=O, and N–H stretching vibrations. The percentage yields were 23.4% for ethanol extraction and 32.4% for water extraction, with the extracted dyes appearing as light brown (water) and dark brown (ethanol). This study underscores the potential of neem bark as a sustainable source of natural dyes.

KEYWORDS

Azadirachta indica, Natural dye, Extraction, UV-Vis spectroscopy, FTIR

ARTICLE HISTORY:

Received: June, 2024
Received: in revised: October, 2024
Accepted: October, 2024
Published online: November, 2024

INTRODUCTION

Dyes are organic colour substances applied to substrates to impart colour by temporarily altering or disrupting the crystalline structure of the material (Corneiro *et al.*, 2007). They are typically applied in aqueous solutions and often require a mordant to enhance their fastness on fibers

(Tamilarasi & Banuchitra, 2021). While dyes are generally soluble in water, pigments are insoluble. However, some dyes can be converted into insoluble lake pigments through the addition of salts (Smelcerovic, 2021). Dyes may also function as colourants formed through chemical reactions, with substrates including textile fibers, paper, leather, hair, plastics, foodstuffs, and pharmaceuticals (Ogunmakin, 1992).

Dyes are characterized as coloured substances because they absorb light within the visible spectrum. This property arises from the presence of specific groups of atoms in the molecule, known as chromophores (Maria *et al.,* 2010). When chromophores are incorporated into organic compounds, they selectively absorb light in the visible region, producing colours. For example, while anthracene is colourless, anthraquinone is a pale-yellow compound (Ogunmakin, 1992; Tamilarasi & Banuchitra, 2021). The wavelength of light absorbed by a dye molecule is influenced by its substituents, particularly their electron-withdrawing or electron-donating effects and their positional relationships within the molecule (Vankar, 2017).

Globally, colour has played a pivotal role in shaping human cultures, influencing aspects of life ranging from clothing to home furnishings (Colchester, 2007). Natural dyes, compared to synthetic ones, are recognized for being eco-friendly and for their lack of carcinogenic or allergenic effects, especially for those involved in dyeing processes (Tamilarasi & Banuchitra, 2021). Recent environmental regulations in developing countries have restricted the excessive use of synthetic dyes (Verssimo *et al.*, 1991), prompting a resurgence of interest in researching natural dye sources and their applications (Acguah & Oduro, 2012).

Azadirachta indica (Neem) is one such plant with immense potential for natural dye production. Widely revered in Nigeria for its traditional medicinal uses, every part of the neem tree has documented therapeutic properties. Commonly referred to as the 'Tree of Wonder' (Khanal, 2021; Khetarpal, 2010) or "Nature's Drug Store" (Paul *et al.*, 2011), neem belongs to the mahogany family (Meliaceae). It is a fast-growing deciduous tree, reaching heights of 14–18 meters, with wide branching and leaf shedding during winter. Highly drought-resistant, neem thrives in tropical and subtropical climates.

The neem tree's active constituents include azadirachtin, salanin, and nimbin, with nimbin being the first chemical limonoid isolated from the tree (Kuravadi *et al.*, 2015). Extracts from its seeds, leaves, and bark contain azadirachtin, which demonstrates strong biological activity against insect pests while exhibiting low toxicity to mammals and the environment (Makeri *et al.*, 2007; Umar *et al.*, 2002). Across Africa, plants have been widely utilized to extract natural products for applications such as drugs, pesticides, and herbicides (Mayunga, 2007). However, efforts to explore and utilize natural dye compounds from African plants, particularly for the leather industry, remain limited.

The resurgence of interest in natural dyes is driven by increasing environmental regulations and a demand for sustainable materials in industries (Acguah & Oduro, 2012). Among natural dye sources, *Azadirachta indica* (neem) stands out for its multifaceted applications, including its use as a biopesticide and medicinal agent (Khanal, 2021; Kuravadi *et al.*, 2015). Neem bark, rich in secondary metabolites, offers a promising source of natural dyes with potential for industrial applications. However, its application in leather dyeing and other industries remains underexplored, particularly in Africa. This study investigates the extraction of dye from neem bark using ethanol and water, characterizing the extracts to determine their suitability for industrial use.

MATERIALS AND METHODS

Materials

Neem bark was sourced from the Nigerian Institute of Leather and Science Technology, Zaria (NILEST), and identified at the Department of Botany, Ahmadu Bello University, Zaria. Ethanol and other analytical-grade reagents were obtained from the Quality Control Laboratory, Department of Leather Technology.

Extraction of Natural Dye

The bark was cleaned, oven-dried, and ground into a fine powder. Using a Soxhlet extractor, 10 g of powdered bark was subjected to solvent extraction with ethanol and water. For ethanol extraction, the system was maintained at 75–76°C for 48 hours. For water extraction, the powdered bark was immersed at room temperature for 2 hours. Extracts were concentrated by solvent evaporation to yield paste-like dyes.

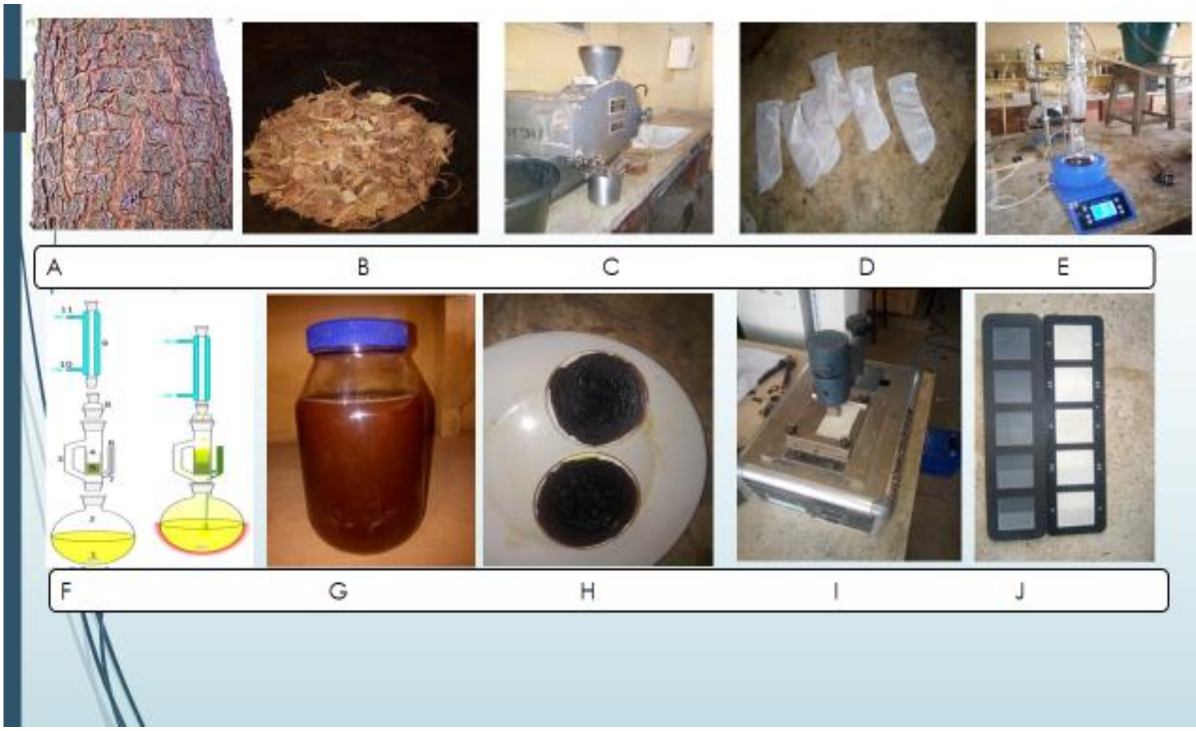

Figure 1: Overview of the extraction of dye from *A. Indica*

Characterization of Extracted Dye

UV-Vis spectroscopy

The UV-Vis spectra of the dye extracts were recorded within the 400–800 nm range using a Cary 300 UV-Vis spectrophotometer to determine the maximum absorption wavelengths (λmax).

Fourier-transform infrared spectroscopy (FTIR)

FTIR analysis was performed using the KBr pellet method to identify functional groups responsible for the dye's properties. Spectra were recorded in the 4000–400 cm^{-1} range.

RESULTS AND DISCUSSION

Extraction Yield

Table 1: Extraction and percentage yield (%)

S/N	Extraction types	Solvent used	Initial wt. (G)	Finial wt.(G)	Temp .(o)	Time (hrs.)	Extraction Apparatus	%yield= $\frac{W1-W2}{W1}$
1	Solvent Extraction	Ethanol	100	76.57	75-76	48	Soxhlet	23.4%
2	Water Extraction	water	100	67.58	RT^{o}	2	Conical flask	32.4%

Where % yield = $\frac{W1-W2}{W_1}$= 100
Key: RT= Room Temperature
W_1 = Initial weight of the sample before extraction
W_2 = Final weight of the sample after extraction

The extraction yields (Table 1) of 32.4% for water and 23.4% for ethanol underscore the influence of solvent polarity on the solubility and recovery of phytochemicals from *Azadirachta indica* bark. Water, being a highly polar solvent, is more effective in dissolving hydrophilic compounds such as tannins, polyphenols, and flavonoids, which are abundant in neem bark. Ethanol, while also polar, exhibits intermediate polarity, making it less effective in extracting certain highly hydrophilic constituents. This differential yield highlights the significance of solvent selection in optimizing the extraction process for specific compounds.
The higher yield with water could also suggest the potential use of aqueous extracts in cost-sensitive and environmentally conscious industrial applications, as water is both inexpensive and non-toxic. However, the slightly lower yield of ethanol extraction may offer distinct compositional advantages, potentially isolating more lipophilic components, which could contribute to unique dyeing properties.

UV-Vis Spectroscopy

The UV-Vis spectra of the extracted dyes, with maximum absorption peaks at 450–540 nm for water-extracted dye and 460–530 nm for ethanol-extracted dye, confirm the presence of chromophores that absorb light within the visible range. These absorption bands correspond to $\pi\rightarrow\pi^*$ transitions, which are typical of conjugated systems found in phenolic compounds and flavonoids.

The broader range of absorption in the water extract may indicate a more diverse array of chromophores, possibly due to the higher solubility of polyphenols and their derivatives in water. This suggests that the water-extracted dye could produce richer colour intensities in applications requiring uniform absorption across a wide wavelength range. Conversely, the ethanol-extracted dye's slightly narrower absorption band might be indicative of a more specific chromophore composition, which could be advantageous in achieving specific tonal variations.

These spectral characteristics not only demonstrate the presence of dye-active molecules but also suggest potential applications where customized colour profiles are required, such as in leather finishing or textile dyeing processes.

FTIR Analysis

The functional groups identified via FTIR spectroscopy provide insights into the molecular structure of the extracted dyes. The presence of O–H and N–H stretching vibrations at 3220.4 cm^{-1} indicates hydroxyl and amine functionalities, which enhance hydrogen bonding and affinity to hydrophilic substrates. These groups are characteristic of tannins and flavonoids, known for their excellent dyeing properties.

The C–H stretching vibrations at 2102.2 cm^{-1} and the C=O stretching at 1982.9 cm^{-1} signify the presence of aliphatic and carbonyl groups, respectively. Carbonyl groups are particularly important as they often act as key chromophores, contributing to the dye’s colour intensity and stability. Furthermore, these groups can form coordinate bonds with transition metal ions, suggesting the potential for mordant dyeing techniques to enhance fastness properties.

The combination of these functional groups implies that the extracted dyes possess a robust framework of reactive sites, making them versatile for use in various industries. The interplay of these groups facilitates strong adherence to substrates, ensuring durability and uniform colour distribution.

Industrial Implications

The light and dark brown hues produced by water- and ethanol-extracted dyes indicate their suitability for diverse industrial applications. In leather production, these hues align with natural aesthetic preferences and can serve as alternatives to synthetic dyes, which are often associated with environmental hazards. The use of neem-derived dyes in textiles and food industries further supports the growing demand for non-toxic and biodegradable colourants.

The eco-friendly nature of neem-derived dyes not only addresses sustainability concerns but also provides industries with opportunities to reduce reliance on petrochemical-based dyes. Their potential for customization, as indicated by the differences in UV-Vis absorption spectra and FTIR profiles, allows for tailored applications, enhancing their commercial value. Moreover, the renewable nature of neem bark ensures a sustainable supply chain, making these dyes economically viable for long-term use.

CONCLUSION

This study demonstrates the successful extraction of natural dye from *Azadirachta indica* bark using ethanol and water. Water extraction was more efficient, yielding 32.4% compared to ethanol's 23.4%. UV-Vis and FTIR analyses confirmed the presence of chromophores and functional groups essential for dyeing applications. These findings establish neem bark as a viable source of natural dyes, contributing to the development of sustainable alternatives in industrial processes.

REFERENCES

Acguah, S. B., & Odura, K. A. (2012). Traditional cloth dyeing enterprise at Ntonso: challenges and opportunities. *West African Journal for Applied Ecology*, *20*(1), 25-36.

Carneiro P. A., Nogueira, R. F. P., & Zanoni, M. V. B. (2007). Homogeneous photodegradation of C.I Reactive Blue 4 using a photo-Fenton process under artificial and and solar irradiation. *Dyes and Pigments*, *74*, 127-132.

Colchester, P. (2007). *Textile today: A global survey of trends and traditions*. Thames and Hudson Ltd., 27-28.

Khanal, S. (2021). Qualitative and quantitative phytochemical screening of Azadirachta indica juss plant parts. *International Journal of Applied Sciences and Biotechnology*, *9*(2), 122-127. https://doi.org/10.3126/ijasbt.v9i2.38050.

Khetarpal, S. (2010). Neem: A wonder tree. Asia-Pacific. *Biotech News, 14*(18), 18-20. DOI: 10.1142/S0219030310000522.

Kuravadi, N. A., Yenag, I. V., Rangiah, K., Mahesh, H. B., Rajamani, A., Shirke, M. D., & Gowda, M. (2015). Comprehensive analyses of genomes, transcriptomes and metabolites of neem tree. *Peer J* 3: e1066. DOI: 10.7717/peerj.1066.

Maria, Z., Eleni, N., Evangelia, V., & Eforia, T. (2010). Colour fastness of natural dye: revival of traditional dyeing techniques. *Colouration Technology, 127*, 18-27.

Mayunga, H. H. N. (2007). Towards the discovery of drugs and pesticide agents from east Africa flora. The 12th Napreca Symposium.July 22-26, Hotel Africana, Uganda. Rogers Museum of Art. Science Publishers

Ogumakin, A. (1992). Short historical review of dyes. Paper presented at a seminar on synthetic dyes for textile fibre. Held at the Lagos Chapter of the Textile Society of Nigeria. *Pacific Journal of Tropical Medicine*, 309-312.

Paul, R., Prasad, M., & Sah, N. K. (2011). Anticancer biology of *Azadirachta indica* L. (neem): a mini review. *Cancer Biology & Therapy, 12*(6), 467-476.

Smelcerovic, M., Dordevic, D., & Novakovic, M. (2006). Textile dyeing by dyestuffs of natural origin. *Hemijska industrija, 60*(5-6), 120-128.

Tamilarasi, A., & Banuchitra, M. (2021). Classification and types of natural dyes: A brief Review. *International Journal of Creative Research Thoughts*, *9*(11), 527-532. www.ijcrt.org.

Umar, A., Abdulraham, H. T., & Kokori, M. (2002). Preliminary studies of the efficiencies of the aqueous extracts of leaf and seed kernel of neem for the control of cowpea bruchid *Callobruchus maculates*. *Research Journal of Science*, *8*(1 and 2), 25-30.

Vankar, P. S. (2007). *Handbook on natural dyes for industrial applications*. National Institute of Industrial Research.

Verssimo, S. A., Oliveira, E. L., Ladchumananadasivan, R., Cruz, L. P. R., & Aquino, M. S. (1991). Extraction, characterization and application of Annatto Dye in the Dyeing of natural fibres. *India Textile Journal*, *2*, 33.