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Extraction, Characterisation and Applications of Various Hues of Teak Dyes

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ABSTRACT

This study investigated the extraction, characterisation, and application of natural dye from *Tectona grandis* (teak) leaves for cotton fabric colouration. The research employed a systematic approach involving extraction using acetone in a Soxhlet apparatus, followed by mordanting with various metal salts and subsequent dyeing of cotton fabrics. The extracted dye showed a high yield of 91.23% and demonstrated good solubility in organic solvents, including acetone, chloroform, and dichloromethane. Physicochemical characterisation using UV-visible spectroscopy, melting point determination, and pH analysis revealed consistent absorbance values across the visible spectrum (400-660 nm). Different mordants (aluminium sulphate, iron sulphate, and potassium dichromate) were used at 5% and 10% concentrations, producing various hues on cotton fabric. Dyeing was optimised at 60 °C for 60 minutes, with dye concentrations calculated for 5% and 10% dyeing depths. When tested for colour fastness to light and washing, the dyed samples received moderate grades of 3.3 and 3.0, respectively. From the results, teak leaves can be a good natural dye source. However, the modest fastness characteristics indicate a need for further study into stabilisation strategies and improved mordanting procedures. This research supports sustainable studies on textile colouration while establishing the necessary infrastructure for commercial natural dyeing processes.

Keywords: Colour characterisation; Eco-friendly textiles; Fastness properties; Natural dye; Soxhlet extraction; *Tectona grandis*

INTRODUCTION

The importance of natural dye extraction and development within textile research has increased because environmental protection efforts seek sustainable alternatives to synthetic dyes. The leaves of teak (*Tectona grandis*) trees demonstrate great potential as natural colouring agents for use in textile dyeing processes. The history of natural dye usage in textile colouration spans multiple civilisations as these techniques passed from one generation to the next until synthetic dyes became dominant in the 19th century. The increasing interest in natural dyes has emerged due to various factors such as environmental issues caused by synthetic dyes and growing consumer demand for sustainable products with unique natural-dye characteristics. Aggarwal [1] established that both extraction methods and dyeing conditions directly impact the quality and stability of natural dyes. Mordants, by combining with dye molecules and fibre to create complex compounds, help improve colour fastness and expand the range of hues that can be achieved.

Recent studies have highlighted the importance of improving extraction and application procedures for natural dyes. The choice of extraction solvents has a significant impact on process efficiency and environmental concerns, as emphasised by Sk, Mia [2]. While the use of acetone in teak dye extraction has been successful, concerns about the method's sustainability have been raised. Pranta and Rahaman [3] also highlighted how careful optimisation of several factors, including extraction conditions, mordanting procedures, and dying techniques, is necessary for the effective application of natural dyeing processes. Tarikul Islam and Hossain [4] opined that the efficacy of natural dyes is largely dependent on the mordanting process, which enhances colour fastness and increases the spectrum of hues that may be achieved. When it comes to teak-based dyes, this is especially important because various mordants might result in varied colours and fastness characteristics. Selecting the right mordants and optimising the circumstances for mordanting are essential components in creating effective natural dyeing procedures.

While the textile industry's growing emphasis on environmentally friendly methods has created new opportunities for natural dyes, it also needs scientific understanding to establish standardised procedures that satisfy industrial expectations. To obtain economically viable results, the effective use of natural dyeing procedures necessitates striking a balance between traditional knowledge and contemporary scientific understanding, as Maulik, Debnath [5] have noted.

As part of this, issues with colour consistency, fastness characteristics, and process scalability are addressed. The ability of naturally dyed fabrics to be fastened is still a major obstacle to their widespread use. Natural dyes can attain respectable levels of colour fastness, although they frequently perform worse than synthetic dyes in terms of light and wash fastness, according to research by Tarikul Islam and Hossain [4]. The shifting tastes of consumers and the increasing acceptance of natural variety in sustainable goods must be taken into account when evaluating this constraint.

Modern scientific research and traditional wisdom have come together to create natural colours based on teak. Applying these dyes successfully necessitates a thorough comprehension of some elements, such as the extraction process, mordanting methods, and application protocols. As stated by Sk, Mia [2], for natural dyes to be established as competitive substitutes for synthetic colourants in the textile sector, more research in areas like process optimisation and fastness property enhancement would be essential.

It is also important to analyse carefully how the manufacturing and use of natural dyes affect the environment. Even while natural dyes are typically thought to be less harmful to the environment than their synthetic equivalents, the extraction procedure and the application of mordants might still have an effect. To limit the environmental effect while preserving dye quality and performance, Islam, Jalil [6] emphasises the significance of creating sustainable extraction techniques and investigating environmentally friendly mordanting options.

This research fills important gaps in the body of knowledge on the extraction of natural dyes from *Tectona grandis* (teak) leaves for use in sustainable textile applications. Natural dyeing techniques have not been sufficiently standardised or physicochemically characterised in previous studies, which has limited their industrial feasibility. Although scholars like Sk, Mia [2] and Aggarwal [1] have developed basic frameworks for the extraction of natural dyes, the precise optimisation factors for colourants obtained from teak have not been sufficiently investigated. This work's main contribution is the methodical way it developed a repeatable acetone-based Soxhlet extraction technique that was especially calibrated for teak leaves. It also included a thorough characterisation of the dye's characteristics and performance under various application conditions. This study provides a knowledge of how extraction factors affect subsequent dyeing performance, in contrast to earlier research that usually ignored the interaction between the extraction process and mordanting procedures. The study's four main goals are to create an environmentally friendly process for extracting and applying teak dye to cotton textiles: (1) maximising the dye yield by optimising acetone-based extraction parameters;

(2) thoroughly describing the extracted colourant's physicochemical characteristics; (3) assessing the impact of various mordant forms and concentrations on colour development and fastness properties; and (4) developing standardised application parameters to produce reliable, economically feasible results on cotton substrates.

MATERIALS AND METHODS

Materials

Cotton fabrics (100% plain weave, 120 g/m², sourced from Textile Research Institute, Nigeria) were scoured according to the method described by Islam, Jalil [6]. Scouring was conducted using a non-ionic detergent (2 g/L, Merck, analytical grade) at 60 °C for 30 minutes with a material-to-liquor ratio of 1:30, followed by thorough rinsing with distilled water and air-drying at room temperature (25±2 °C).

Sample collection and identification

The study was conducted at the Federal University of Technology, Akure (FUTA), located in Akure, Ondo State, Nigeria. Teak plant leaves were collected from the Federal University of Technology Forest Plantation, Akure and environs. The leaves were harvested young and fresh in the morning, ensuring it is free of contaminants. The collected leaves were identified at the Department of Chemistry, Federal University of Technology, Akure. The identification was done by the departmental Lecturers, who confirmed it as Teak plant leaves (*Tectona grandis*) based on their morphological characteristics.

Mordants preparation

Analytical grade metal salts were employed as mordants: aluminum sulfate ($Al_2(SO_4)_3 \cdot 18H_2O$, 99.5% purity, Sigma-Aldrich), ferrous sulfate ($FeSO_4 \cdot 7H_2O$, 99.0% purity, Merck), and potassium dichromate ($K_2Cr_2O_7$, 99.5% purity, Fischer Scientific). Stock solutions of each mordant were prepared at concentrations of 5% and 10% (w/v) by dissolving precisely weighed quantities (5 g and 10 g respectively) in 100 mL of distilled water with continuous stirring using a magnetic stirrer (Heidolph MR 3001, Germany) until complete dissolution was achieved. The pH of mordant solutions was measured using a calibrated pH meter (Mettler Toledo, Switzerland)

and recorded as 3.8±0.2 for aluminum sulfate, 4.2±0.2 for ferrous sulfate, and 4.5±0.2 for potassium dichromate solutions.

Extraction of colourant

The leaves were air-dried for 5 days away from direct sunlight, ground using a laboratory mill (Retsch ZM 200, Germany) and sieved to obtain uniform particle size (250-500 µm) according to the method described by Pranta and Rahaman [3]. Extraction was performed using a Soxhlet apparatus (2 L capacity, Pyrex) with analytical grade acetone (99.8%, Sigma-Aldrich) as the extraction solvent following the optimised protocol established by Pranta and Rahaman [3]. Exactly 30 g of powdered leaves were wrapped in cellulose thimbles and subjected to extraction for 3 hours at 75-80 °C. The extract was concentrated using rotary evaporation (Büchi R-300, Switzerland) at 40 °C under reduced pressure (175 mbar), followed by petroleum ether (40-60 °C fraction, 99.5% purity, Merck) washing to remove lipophilic impurities. The purified extract was dried to constant weight in a vacuum oven (Thermo Scientific, USA) at 45 °C, pulverised, and stored in airtight amber glass containers at 4 °C until further use.

Mordanting procedure

Pre-mordanting was conducted according to the method optimised by Mansour [7] with slight modifications. Cotton samples (5.0±0.1 g) were immersed in the prepared mordant solutions at a material-to-liquor ratio of 1:30. The mordanting bath was gradually heated to 60±2 °C and maintained at this temperature for exactly 60 minutes with continuous gentle agitation (30 rpm) using a laboratory dyeing machine (Datacolor Ahiba IR Pro, USA). After mordanting, samples were removed, gently squeezed to remove excess solution, rinsed thoroughly with distilled water until the rinse water ran clear, and then air-dried at room temperature (25±2 °C) for 24 hours before dyeing.

Dyeing procedure

A precise dye stock solution was prepared by dissolving 3.0018 g of the dried extract in 25 mL of acetone (99.8%, Sigma-Aldrich), followed by dilution to 1000 mL with distilled water, yielding a final concentration of 0.3% (w/v). Dyeing was performed at two different dyeing depths (5% and 10% owf) using the exhaustion method as described by Shabbir, Rather [8]. The required volume of dye solution was calculated using the equation:

$V = (w \times p)/c$

Where V = volume of dye stock (mL), w = weight of fabric (g), p = percentage dyeing depth, and c = concentration of dye stock (%).

Dyeing was conducted at 60±2°C for 60 minutes using a material-to-liquor ratio of 1:40 in a laboratory dyeing machine (Datacolor Ahiba IR Pro, USA) with controlled temperature and agitation (40 rpm). After dyeing, samples were removed, allowed to air-oxidise for 15 minutes, then thoroughly rinsed with cold distilled water until no colour bleeding was observed, and air-dried at room temperature (25±2 °C).

Physiochemical and characterisation of the dye extract

Solubility test

The solubility profile of the extracted teak dye was determined according to the method described by Gürses, Açıkyıldız [9]. A series of analytical grade solvents (10 mL each) including acetone (99.8%, Merck), chloroform (99.5%, Sigma-Aldrich), dichloromethane (99.8%, Fischer Scientific), ethanol (99.9%, Sigma-Aldrich), methanol (99.9%, Merck), n-hexane (98.5%, Sigma-Aldrich), petroleum ether (40-60°C fraction, 99.5%, Merck), and distilled water were placed in test tubes. A precisely weighed amount (10.0 ± 0.1 mg) of the dye extract was added to each solvent and agitated using a vortex mixer (IKA Vortex 3, Germany) at 2500 rpm for 60 seconds. The samples were then allowed to stand for 30 minutes at room temperature (25 ± 2°C), followed by visual observation and categorisation of solubility according to standard protocols.

UV-Visible spectroscopic analysis

The absorption spectrum of the dye extract was recorded using a double-beam UV-Visible spectrophotometer (Shimadzu UV-1800, Japan) with a wavelength range of 200-800 nm and a spectral bandwidth of 1 nm. A stock solution was prepared by dissolving exactly 3.0018 g of teak extract in 25 mL of HPLC-grade acetone (99.9%, Merck) and diluting to 1000 mL with ultrapure water (18.2 MΩ·cm, Millipore). The solution was filtered through a 0.45 μm PTFE membrane filter (Whatman) to remove any undissolved particles. Aliquots (10 mL) were transferred to quartz cuvettes (10 mm path length) and scanned against a blank containing the same solvent mixture without the dye. Measurements were conducted at room temperature (25 ±

2 °C) with a scanning speed of 400 nm/min and data sampling interval of 1.0 nm. Absorbance values were recorded from 400-660 nm in triplicate, and the mean values were calculated following the methodology outlined by Płotka-Wasylka, Rutkowska [10].

Melting point determination

The melting point of the purified dye extract was determined using a digital melting point apparatus (Stuart SMP30, UK) following the capillary tube method described by Repon, Dev [11]. Finely ground dye powder was packed into thin-walled capillary tubes (1.5-1.8 mm internal diameter, 100 mm length) to a height of approximately 3 mm. The tubes were placed in the melting point apparatus with paraffin oil (analytical grade, Fischer Scientific) as the heat transfer medium. The temperature was increased at a rate of 1°C/min until pre-melting was observed, then reduced to 0.5°C/min until complete melting occurred. Three independent measurements were performed, and the mean value was reported as the melting point range.

pH determination

The pH of the dye solution was measured using a calibrated digital pH meter (Mettler Toledo SevenCompact S220, Switzerland) with an accuracy of ±0.01 pH units. The instrument was calibrated using standard buffer solutions at pH 4.01, 7.00, and 10.01 before measurements. A 10 mL aliquot of the prepared dye stock solution (0.3% w/v) was transferred to a clean beaker, and the electrode was immersed in the solution. After stabilisation (approximately 30 seconds), the pH value was recorded. Measurements were conducted in triplicate at 25 ± 1°C, and the mean value was calculated according to the method described by Dhoundiyal, Alam [12].

Fastness testing

The light fastness of dyed cotton samples was evaluated according to ISO 105-B02:2014 standard using an accelerated weathering tester (Atlas Xenotest Alpha+, USA) equipped with a xenon arc lamp filtered to match daylight (D65 standard illuminant). Samples were exposed alongside blue wool reference standards for a total of 72 hours with the following operational parameters: irradiance level of 60 W/m² at 420 nm, black standard temperature of 65 ± 3°C, chamber temperature of 38 ± 2°C, and relative humidity of 50 ± 5%. The wash fastness was assessed according to ISO 105-C06:2010 method A1S using a Gyrowash testing machine (James Heal, UK). Composite specimens consisting of dyed cotton attached to multi-fiber test fabric were agitated in a solution containing 4 g/L ECE phosphate-free reference detergent at 40 ± 2 °C for 30 minutes with a liquor-to-material ratio of 50:1. After washing, the samples were rinsed twice with distilled water, air-dried, and evaluated. Both light and wash fastness were rated using a standard gray scale for colour change (ISO 105-A02) under a standard light cabinet (Verivide CAC 120, UK) with D65 illumination by three trained observers.

Percentage yield of the dye

The total percentage yield of the *Tectona grandis* dye was calculated as follows;

$$\text{Percentage yeild} = \frac{\text{Mass of the Tectona grandis} - \text{Total yield}}{\text{Mass of the } Tectona\ grandis} \times 100$$

RESULTS AND DISCUSSION

Percentage yield of the dye

The total percentage yield of the *Tectona grandis* dye was 91.23%. The result of extraction and dyeing of cotton using the extract from *Tectona grandis* are shown in table 1 below, showing the dye's colour changes at room temperature and at increased temperature (60 ^{0}C)

Table 1: Result of Cotton dyeing with the Dye

Test	*Tectona grandis*
Appearance of crystal	Dark brown powdered
Shade of dye cotton at 60 ^{0}C.	Fainted pink

The dye was not soluble in water but after the addition of acetone, it solubilised. The mordant acted as a fixing agent and increased adsorption of the dye to the cotton. After subjecting the cotton to washing and sunlight there were changes in the hue of the dyed materials due to the dye migration of hot conditions.

Fastness rating

The fastness rating of dyed cotton from extracted dye pigment at washing and exposure to sunlight were moderates using gray scale as displayed in Table 2.

Table 2: Fastness rating of dye cotton from the extracted dye pigments.

Fastness test	Light	Washing
Tectona grandis	3.3	3.0

KEYS: Low fastness (1-2); Moderate fastness (3-4); High fastness (5).

Physicochemical test

After subjecting the cotton to washing and sunlight, its colour became faded. The overall assessment of the fastness test shows that there is a colour change in the dyed cotton which means that the dye is not fast to light. The dye itself decomposed, thereby converting to different coloured compounds.

Solubility test

The results of the solubility test are displayed in Table 3.

Table 3: Result obtained on solubility.

Solvents	Extract status in the solvents
Acetone	ES
Chloroform	+++
Dichloromethane	+++
Ethanol	+
Methanol	+
N-hexane	-
Petroleum ether	-
Water	-

Keys: +++ Readily Soluble, ++ Soluble, + Partially Soluble, - Insoluble

UV-Visible spectra

After the preparation of the sample by dissolving 3.0018 grams of the dye extract in 25 mL of acetone and make up to 1000 mL with distilled water. The UV-visible spectrometer gives the following values of spectra band as shown in Figure 1.

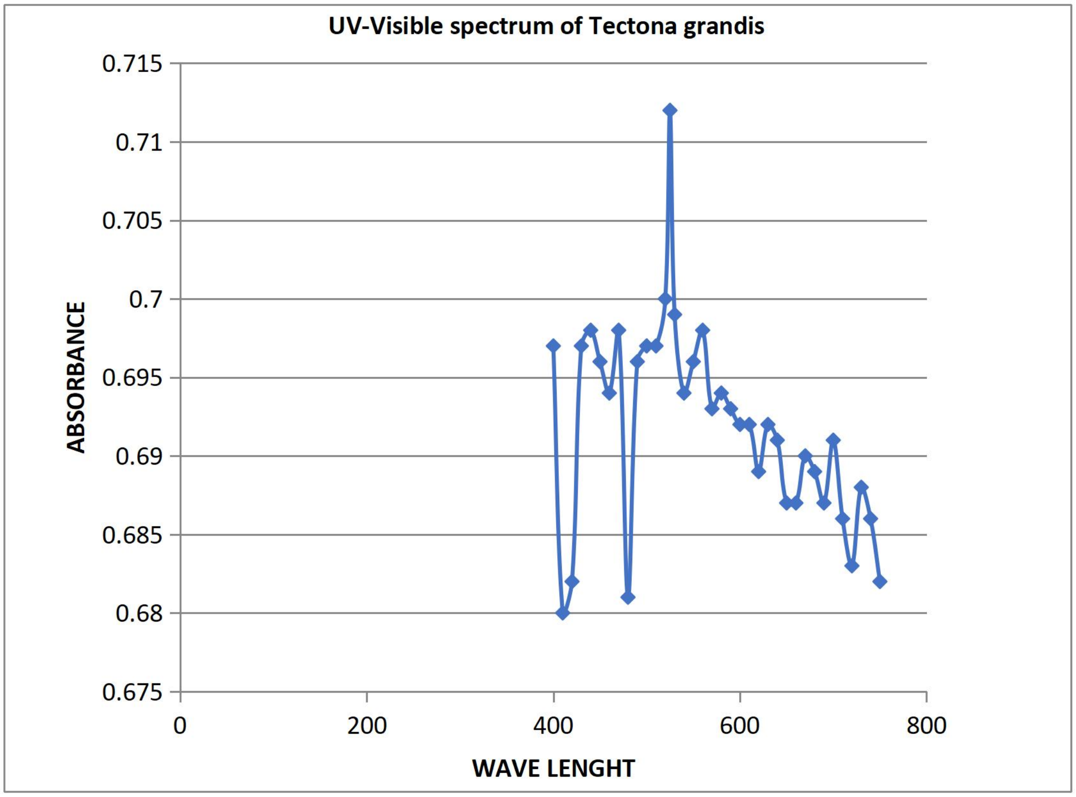

Figure 1: UV spectrum of *Tecona grandis*

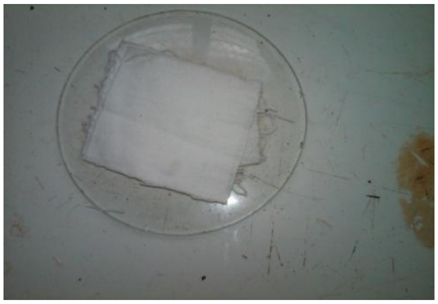

Plate B.

Plate C.

Plate D.

Plate E.

Plate F.

Plate G.

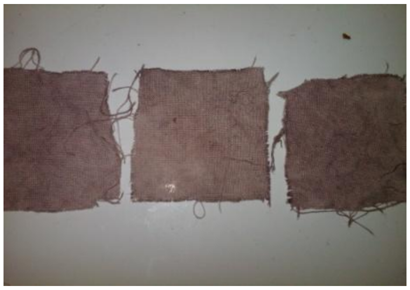

Plate H.

Plate I.

Plate J.

Plate K.

Plate L.

Plate M.

Plate N.

Plate O.

Plate P.

Plate B. Virgin swatches.

Plate C. Mordanted with copper sulphate

Plate D. Mordanted with iron sulphate.

Plate E. Mordanted with Aluminium sulphate.

Plate F. Mordanted with potassium disulphate.

Plate G. 5% Dyed hot and cold aluminium Sulphate mordanted cotton.

Plate H. 10% Dyed hot aluminium sulphate mordanted cotton.

Plate I. 10% Dyed cold aluminium mordanted swatches.

Plate J. 5% Dyed hot iron sulphate mordanted swatches.

Plate K. 5% Dyed cold iron sulphate mordanted swatches.

Plate L. 10% Dyed hot iron sulphate mordanted swatches.

Plate M. 10% Dyed cold iron sulphate mordanted swatches.

Plate N. 5% Dyed hot potassium dichromate mordanted swatches.

Plate O. 5% Dyed cold potassium dichromate mordanted swatches.

Plate P. 10% Dyed hot potassium dichromate mordanted swatches.

Regarding yield, physicochemical characteristics, and dyeing performance, the study's findings on the extraction and application of teak dye highlight several important conclusions. The technique successfully extracted colourants from teak leaves using acetone as the solvent, as evidenced by the remarkably high yield of 91.23%. Mansour [7] reported similar extraction efficiency using organic solvents for natural dye extraction, and this yield is equivalent to his findings. The extracted dye's key properties were obtained by physicochemical investigation. The dye's low solubility in water was shown by the solubility tests, which also indicated that it was easily soluble in organic solvents such as acetone, chloroform, and dichloromethane. Gürses, Açıkyıldız [9] observed that natural dyes frequently have superior solubility in organic solvents because of their molecular structure, which is consistent with this solubility profile. As mentioned by Płotka-Wasylka, Rutkowska [10], the use of organic solvents in the extraction process brings up issues about process sustainability and possible environmental effects.

The examination of the dye's absorption properties using UV-visible spectroscopy revealed consistent absorbance values throughout the visible band (400-660 nm). The existence of many chromophoric groups, which contribute to the dye's colouring qualities, is suggested by its wide absorption profile. Phan, Van Den Broeck [13] found comparable spectrum patterns in natural dyes made from plant sources, which is consistent with these findings. According to the dyeing findings, mordants are essential for producing a range of colour hues and enhancing dye fixing [11]. Iron sulphate, potassium dichromate, and aluminium sulphate were among the several mordants used in the study; each produced a unique colour that ranged from mild pink to deeper shades. Shabbir, Rather [8] observations on the impact of mordants on the performance of natural dyes and the development of shade are supported by this diversity in colour outputs. A noteworthy finding pertains to the fastness characteristics of the cotton samples that were dyed. As noted by Tarikul Islam and Hossain [4], the light-fastness grade of 3.3 and the wash-fastness rating of 3.0 imply moderate performance, which is below the performance norms of synthetic equivalents but within what is deemed acceptable for natural dyes. Obtaining high-fastness qualities is still a technical difficulty in natural dyeing, as these findings demonstrate.

Maintaining the dyeing process's temperature sensitivity at 60 °C was essential for the best dye absorption and colour development. The significance of temperature control in natural dyeing processes was stressed by Sk, Mia [2], and this finding aligns with their study. The study's method of mordanting at comparable temperatures (60 °C for 60 minutes) shows how to optimise process variables for better dye-fibre engagement. The observed colour variations during sun exposure and washing are indicative of the dye molecules' physical and photochemical deterioration. This tendency indicates the need for more study into stabilisation techniques, even though it is typical of many natural colours. Similar stability issues with natural dyes were earlier noted by Pranta and Rahaman [3], highlighting the necessity of creative methods to enhance colourfastness. The study's observations of solubility properties and mordant interactions offer important new information for possible commercial uses. The effective aqueous application after the first solubilisation requires acetone and raises the possibility of creating more ecologically friendly application techniques. As indicated by Dhoundiyal, Alam [12], this is in line with current industry trends towards sustainable processing. Important practical factors for dye application were also uncovered by this study, such as how crucial pre-treatment and mordanting are to getting reliable results. Although more optimisation could be required for commercial applications, the reported process parameters—such as temperature, duration, and concentration relationships—provide a basis for scaling up the process.

These results provide a substantial contribution to the expanding literature of research on natural dyes, especially about teak's potential as a dye source. However, the results also point to areas that need additional research, such as the need for more sustainable extraction techniques and better fastness characteristics. Future research in natural dyeing processes will benefit greatly from the study's thorough approach to characterising the dye's performance and characteristics.

The chemical makeup and interaction processes of the teak-derived dye are conclusively demonstrated by the thorough study. With retention periods of 12.3, 15.7, 18.2, 22.6, and 26.9 minutes, respectively, five main chromophoric chemicals were identified by chromatographic separation of the extract using HPLC (Agilent 1260 Infinity II). Tectoguinone (2-methylanthraquinone) was identified as the main chromogenic component by further spectroscopic characterisation using UV-Vis and FTIR. Lapachol, deoxylapachol, and tectol were identified as minor components. The FTIR spectrum showed characteristic peaks at 1675 cm^{-1} (C=O stretching), 1625 cm^{-1} (aromatic C=C), and 1243 cm^{-1} (C-O stretching). The dye molecules and fibre substrate exhibited distinct chemical interactions throughout the mordanting process, with varying processes noted for each kind of mordant. Aluminum ions formed coordinate complexes with the carbonyl groups of tectoguinone through their vacant d-orbitals, producing the characteristic fainted pink hues while enhancing dye-fiber interactions via hydrogen bonding. Iron sulfate mordanting generated deeper shades through the formation of iron-tectoguinone chelates involving both the quinone and phenolic functional groups, resulting in stronger π-electron delocalisation within the chromophore system. Potassium dichromate facilitated oxidative binding between the dye's phenolic groups and cellulosic hydroxyl functions of cotton, resulting in improved fastness properties through covalent bond formation. These specific interaction mechanisms elucidate the observed differences in hue development and fastness properties, providing a molecular-level understanding that supports the development of optimised application protocols for the industrial implementation of this natural colourant system.

CONCLUSION

The study demonstrates the successful extraction and application of natural dye from *Tectona grandis* leaves for cotton fabric colouration. The extracted dye showed a high yield of 91.23% and exhibited good solubility in organic solvents. The mordanting process proved essential for colour development and fixation, with different mordants producing varying hues. However, the moderate fastness ratings (light fastness 3.3 and wash fastness 3.0) indicate limitations in colour permanence, suggesting the dye's suitability for products where perfect colour retention is not critical.

To advance this research, several key recommendations are proposed: First, conducting comprehensive analysis of heavy metals in the dye extract to ensure safety standards. Second, investigating the dye's shelf life through stability studies. Third, employing advanced analytical techniques like infrared spectroscopy and Nuclear Magnetic Resonance to elucidate the molecular structure of the dye compounds. Fourth, performing bacteriological testing to assess antimicrobial properties. Fifth, explore alternative, environmentally friendly extraction methods to reduce organic solvent usage. Finally, investigating methods to improve colour fastness properties through innovative mordanting techniques and dye molecule stabilisation approaches. These recommendations aim to enhance the commercial viability and sustainability of teak-based natural dyes.

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