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Production, Characterization and Testing of Teak Leaf (*Tectona grandis*) Dye Extract on Cotton Fabric

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Abstract

The natural dye was extracted from the leaves of Tectona grand is using a Soxhlet extractor, and a percentage yield of 16.84% was obtained. The color of the dye extract is maroon. The dye extract was characterized using FTIR, GC-MS, AFM, and UV-visible spectroscopy. The results of AFM showed that the dye extract from teak leaf has a root mean square roughness of 2568.2 nm in the 50μm×50μm scan area, and FTIR characterization suggested the presence of N-H (amine), O-H (alcohol), C-H (alkane), C-H (alkane), C≡C (alkyne), C = C (alkene), C = O (carbonyl groups), alkyl ketone, alkyl amine, and alkyl halide functional groups in the dye extract. The GC-MS separated and identified 36 compounds. Among the compounds identified in the dye extract, cis-vaccenic acid has the highest percentage area (18.68%), and azobenzene has the lowest percentage area (0.37%). The chromophores in the dye extract are C≡N and N=N. The natural dye extract was applied to the cotton fabrics without mordant. The color fastness to washing shows 3-4 and 3 for color change and staining, respectively, while the color fastness to rubbing shows 3 and 2 for dry and wet rubbing respectively. The outcome of color fastness shows that the dye has poor affinity to remain on the fabric. Therefore, the use of different kinds of mordant is recommended.

Keywords: Characterization, Cotton fabric, Dye, Extraction, *Tectona grandis*

INTRODUCTION

The detrimental effects of synthetic dyes on the environment and human health have drawn a lot of attention to the use of natural dyes in the textile industry (Saxena et al., 2014). Synthetic colors come from non-renewable resources; natural dyes are safe, non-toxic, and environmentally favorable substitutes. The leaves, roots, flowers, and bark of plants are among the parts from which natural colors are made (Slama et al., 2021). The release of harmful compounds linked to synthetic dyes is directly reduced when natural dyes are used (Adeel et al., 2019). Because of the harsh conditions required for their synthesis—high pH, high temperature, strong acids, and heavy metal catalysts—synthetic dyes are not environmentally friendly. Synthetic colors can irritate skin and eyes in addition to being poisonous and carcinogenic (Mehta *et al.,* 2019). There are huge applications of natural dye on textiles, so it is imperative to promote technology for extraction (Kulkarni et al., 2011). Joshua *et al.,* (2023) studied the production and testing of *Baphia ntida* stem dye and its application to cotton fabric. It was observed that the red dye extracted from *Baphia nitida* possesses intrinsic affinity for cotton fabric. The observed affinity of the dye extracts for the textile substrates used for the study may be due to the presence of santalins and santarubin compounds. The FTIR characterization of the dye extract also suggested the presence of OH, CH, C≡N, C≡C, C=O, N=O, C=C, C-Cl, and C-Br functional groups in the dye components. The chromophores in the dye extract were N=O and C=O. The GC-MS separated and identified 30 compounds. Among the compounds identified from the dye extract, benz(a)anthracene, 7,8-ddimethyl, had the highest percentage area and 1,3,5-triazine, 2,4,6-ttriamine had the lowest percentage area. There are thousands of plants and animals that contain colorants that can be used as natural dyes. This present study is therefore focused on the extraction, characterization, and application of dye extract from *Tectona grandis (Teak leaf)* on cotton fabric. Teak leaf (*Tectona grandis*), a large deciduous tree of the family Verbenaceae, or its wood, is one of the most valuable timbers. Teak has been widely used in India for more than 2,000 years. The name teak is from the Malayalam word tēkka. Locally, it is used as a chew stick for the treatment of toothache and fertility-related issues in women; it is also used to relieve gastro-intestinal complaints, inflammation, joint pain, and diarrhea (Venkatesh et al., 2023). The aim of this research work is to extract, characterize, and apply the natural dye extract from *Tectona grandis (Teak leaf)* to cotton fabric.

Plate 1: Teak leaf (*Tectona grandis)*

MATERIALS AND METHODS

Sampling and sample preparation

The teak leaf (*Tectona grandis*) was collected from Wukari LGA in Taraba State. The sample was shade dried and ground into powder form using a mortar and pestle, which was ready for extraction **Extraction of natural dye**

The sample was measured using an analytical weighing balance. 200 g of the sample was partitioned into five portions, each containing 30 g of the sample, and 200 mL of absolute ethanol was added into the round bottom flask for each of the 30 g of samples fitted into the thimble for extraction. The heating mantle was on, and extraction was done for 6 hours on each of the samples. The temperature for extraction was 78.37 °C on the sample, which is the boiling point of ethanol. A rotary evaporator was used to remove excess solvent, leaving the dye in a dry state. The percentage yield and color were determined for the purified dye samples (Rather et al., 2020).

FT-IR spectroscopy analysis

The dye extract was measured with an FTIR spectrophotometer using a Shimadzu IR prestige 21 spectrometer in the wavelength range of 400-4000cm.

GC-MS analysis

The dye extracted was characterized on a Shimadzu GC-2010 connected with MS QP-2010. The column that was used was a Restec Rtx-5MS measuring 60 mm × 0.25 mm with an with an ID thickness of 0.25 µm and packed with 95% dimethyl polysiloxane. Helium gas was used as carrier gas at a flow rate of 1 ml/min, and an injection volume of 1 µl was utilized.

AMF (Atomic Force Microscopy) analysis

AFM (Atomic Force Microscopy) TM-AFM model, results conducted on teak leaf extract revealed likely provided detailed information on the surface morphology and topography of the dye leaf. This technique is crucial for characterizing samples at the nanoscale level, offering insights into their size, shape, and distribution.

UV-Visible spectroscopy analysis

The dye extract was introduced in a quartz cell (lcm pathway) and measured with UV-Vis spectrophotometer. Scans from 200 to 800nm were performed in to generate the characteristic absorption spectra of the sample.

Scouring Process

The Scouring process was carried out using 3M of sodium hydroxide. To each of the beakers labelled A, B and C 50ml of the prepared sodium hydroxide was used to carry out the scouring process for 1 hr 100^{0}C (Taleb et al., 2020)

Bleaching process

To each of the beakers labeled A, B, and C 3 M of sodium hypochlorite was measured with a measuring cylinder, and 100 ml of the prepared solution was used to carry out the bleaching process for l hr at 100^{0}C (Sannapapamma et al., 2022).

Dyeing the cotton fabric

The cotton fabric to be dyed was added to the dye solution in the bath and dyeing was carried out at certain concentration, temperature and time using a fixed amount of liquor ratio (1:100). During dyeing, the fabric was frequently stirred in order to obtain the good penetration of dye molecules into the fabric. After dyeing, the dyed sample fabric was thoroughly washed with cold water to remove the unfixed dye particles and squeezed by hand, then the dyed fabric will be dried at room temperature (Karabulut and Atav, 2020).

Wash fastness properties

Procedure: The dyed fabric was first cut (4x4 cm), after which the undyed white fabric was cut (4×4) and machine stitched together to give one piece of fabric. 100 ml of water was measured with a measuring cylinder, and 3 g of detergent (Viva Plus) was weighed using a weighing balance and dissolved in the 100 ml of water contained in the beaker. The stitched fabric was introduced into the detergent solution, stirred vigorously for 10 minutes, and rinsed and dried at room temperature (Geetam et al., 2017)

Rubbing fastness properties

Procedure: The dyed fabric was first cut at 4x4 cm, after which the undyed white fabric was cut (4×4) for both dry and wet rubbing. For dry rubbing, the undyed white fabric was rubbed against the surface of the dyed fabric for 10 minutes. For wet rubbing, the undyed white fabric was first immersed in water (wet) and rubbed against the dyed fabric for 10 minutes (Samanta et al., 2020)

RESULTS AND DISCUSSION

Extraction of dye

The Soxhlet extraction method was used in the extraction of the dye from Tectona grandis. Tectona grandis was pulverized and fed into the soxhlet extractor. Ethanol was used as a solvent, and the extraction was carried out for 8 hours. The temperature for the extraction was 78.37 °C, which is the boiling point of ethanol. The result of the extraction of dye from Tectona grandis, as described in Table 1, showed that 200 g of the sample was used for the extraction. After the extraction, the weight of the sample was reduced to 166.33 g, and the actual yield of dye extracted was 33.67 g. The percentage yield of the dye extract was 16.84%, and the color of the dye extract was brown (Hassan 2023).

Table 1: Extraction of dye from *Tectona grandis*

Tectona grandis powder (g)	Weight after extraction and purification (g)	Actual yield (g)	Percentage yield (%)	Colour of the dye extract
200	166.33	33.67	16.84	Maroon

Plate 2: Dye produced from *Tectona grandis*

FTIR Analysis of Dye Extract from teak leaf (Tectonagrandis)

The FTIR spectrum of teak leaf *(Tectonagrandis),* as highlighted in figure 1, showed a band at 3676.71 cm1, indicating the presence of N-H of the amine group; an away band at 3241.72 cm1 is due to the presence of O-H stretching vibrations (alcohol). The strong bands at 2930.31 cm1 and 2864.09 cm1 are assigned to the C-H stretching of alkane groups. In the spectrum of the dye extract, vibrations occurred at band 2352.15 cm1, which is assigned to the C=C of alkane; a weak band at 2047.29 cm1 is an indication of the presence of C≡C stretching vibrations. The strong absorption band at 1743 $^{\text{cm1 suggests}}$ the presence of C=O groups, which are carbonyl groups, and the absorption band at 1243.13 cm1 is assigned to alkyl ketone compounds. The bands at 1160.29 cm1 and 1083.03 cm1 are assigned to alkyl amine, while the absorption band at 727.27 cm1 is an indication of an alkyl halide (C-Cl) (Cai et al., 2017).

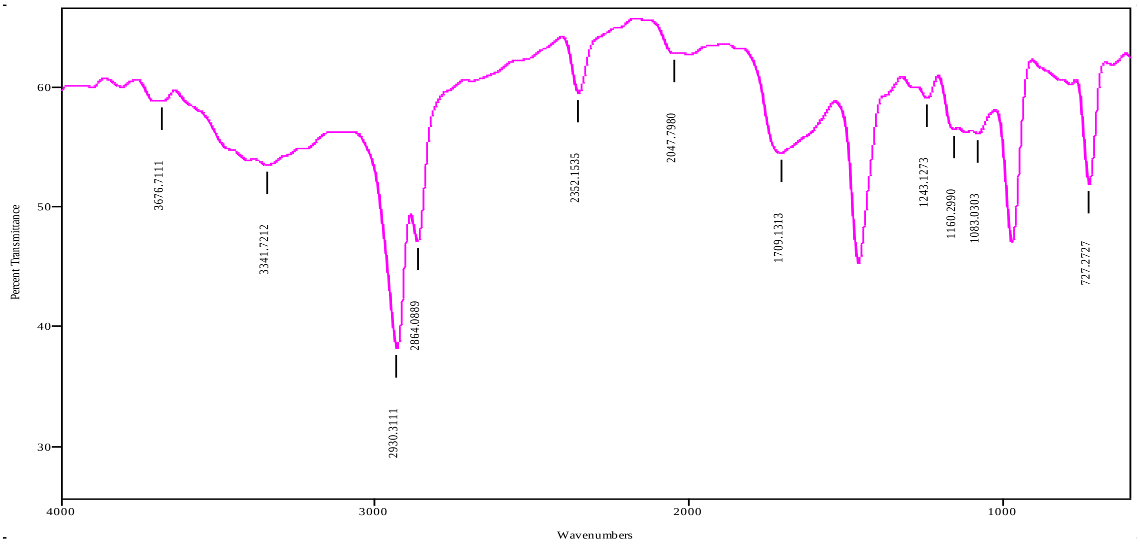

Fig 1: FTIR spectrum of dye extract from teak leaf *(Tectonagrandis)*

GCMS Result of the dye extract from teak leaf *(Tectonagrandis)*

The dye extracts obtained were subjected to GC-MS analysis for the determination of various volatile and semi-volatile compounds in the teak leaf *(Tectonagrandis)*. The analysis was carried out using Shimadzu GC-2010 and QP-2010. The columns that were used were Restec Rtx-5MS measuring 60 mm × 0.25 mm with an with an ID thickness of 0.25 μm and composed of 95% dimethyl polysiloxane. Helium gas was used as a carrier gas at a flow rate of 1 ml/min, and an injection volume of 1 μl will be utilized. The GC-MS examination of the dye extract from teak leaf *(Tectonagrandis)* led to the identification of 36 compounds. The retention time (RT), percentage of peak area, molecular formula, molecular weight, and characterized compounds are listed in Table 2. Some of the identified compounds were p-Pentylacetophenone (0.43%); 2,6-Dimethylbenzaldehyde (1.40%); Phytol (2.53%); 5-Hydroxymethylfurfural (3.50%); Lupeol (3.83%); Oleic Acid (3.08%); Retinol (9.031%); and cis-Vaccenic Acid (18.68%) (Sankaralingam et al., 2023). Among the identified compounds of dye extract from teak leaf *(Tectona grandis)*, cis-vaccenic acid has the highest percentage of 18.68%, while azobenzene has the lowest percentage of 0.37%.

Table 2 GCMS Result of the dye extract from teak leaf *(Tectonagrandis)*

Peak Molecular	Retention Time	% peak Area	Compound identified	Nature of compound	Molecular formula	Molecular weight (g/mol)
1	6.303	0.43	p-Pentylacetophenone Benzene, 1,3-bis(1,1-dimethylethyl	Organic compound Organic compound	$C_{11}H_{14}O_2$ $C_{14}H_{22}$	173.33 190.32
2	7.092	1.40	Inden-5-ol, 2,3-dihydro- 2,6-Dimethylbenzaldehyde	Organic compound Organic compound	$C_9H_{10}O$ $C_9H_{10}O$	134.75 134.75
3	8.768	2.58	Phytol 2-Hexadecen-1-ol, 3,7,11,15-tetramethyl-, [R-[R*,R*-(E)]]-	Organic compound Organic compound	$C_{20}H_{40}O$ $C_{20}H_{40}O$	296.58 296.58
4	9.930	3.50	Phenol, 3,5-bis(1,1-dimethylethyl) 5-Hydroxymethylfurfural	Organic compound Heterocyclic compound	$C_{14}H_{22}O$ $C_6H_6O_3$	206.32 90.00
5	12.088	1.16	1-Naphthalenecarboxylic acid,5-2(3furanyl)ethyl]decahydro-1,4a-dimethyl-6-methylene-, methyl ester 5-Hydroxymethylfurfural	Heterocyclic compound Heterocyclic compound Organic	$C_{21}H_{30}O_3$ $C_6H_6O_3$	330.46 90.00

			Cyclohexane,2-butyl-1,1,3-trimethyl	compound	$C_{13}H_{26}$	182.00
6	13.724	2.45	Ethanedione, Diphenyl- Benzophenone Phenyl 4-pyridyl ketone	Organic compound Organic compound Organic compound	$C_{14}H_{10}O$ $C_{13}H_{10}O$ $C_{12}H_9NO$	210.23 182.00 183.21
7	14.245	0.37	Azobenzene Benzenecarbothioic acid, S-methyl ester 1,2,4,5-Tetroxane, 3,3,6,6-tetraphenyl	Organic compound Organic compound Heterocyclic compound	$C_{12}H_{10}N_2$ C_8H_8O $C_{26}H_{20}O_4$	182.22 136.15 396.00
8	14.256	4.04	Hexadecanoic acid, methyl ester	Organic compound	$C_{26}H_{34}O_2$	270.47
9	16.144	7.03	cis-13-Octadecenoic acid methyl ester 11-Octadecenoic acid, methyl ester, 9-Octadecenoic acid, methyl ester	Organic compound Organic compound Organic compound	$C_{19}H_{36}O_2$ $C_{19}H_{36}O_2$ $C_{19}H_{36}O_2$	296.48 296.48 296.48
10	16.390	2.63	Methyl stearate Heptadecanoic acid, 16-methyl-, methyl ester	Organic compound Fatty acid	$C_{19}H_{38}O_2$ $C_{18}H_{34}O_2$	298.51 282.46
11	17.59	10.24	cis-Vaccenic acid trans-13-Octadecenoic acid cis-13-Octadecenoic acid	Fatty acid Fatty acid Fatty acid	$C_{18}H_{36}O_2$ $C_{18}H_{36}O_2$ $C_{14}H_9O_2$	282.46 282.46 282.46
12	17.918	3.83	Lupeol Lupeol acetate	Organic compound Organic compound	$C_{30}H_{50}O$ $C_{30}H_{50}O_2$	426.73 426.73
13	18.153	4.33	cis-Vaccenic acid Oleic Acid 6-Octadecenoic acid, (Z)-	Fatty acid Fatty acid Fatty acid	$C_{18}H_{34}O_2$ $C_{18}H_{34}O_2$ $C_{18}H_{34}O_2$	282.48 282.48 282.48
14	18.559	2.93	cis-Vaccenic acid cis-13-Octadecenoic acid trans-13-Octadecenoic acid	Fatty acid Fatty acid Fatty acid	$C_{18}H_{34}O_2$ $C_{18}H_{34}O_2$ $C_{18}H_{34}O_2$	282.48 282.48 282.48
15	19.023	2.93	Oleic Acid trans-13-Octadecenoic acid	Fatty acid Fatty acid	$C_{18}H_{34}O_2$ $C_{18}H_{34}O_2$	282.48 282.48
16	19.185	2.19	cis-Vaccenic acid cis-13-Octadecenoic acid trans-13-Octadecenoic acid	Fatty acid Fatty acid Fatty acid	$C_{18}H_{34}O_2$ $C_{18}H_{34}O_2$ $C_{18}H_{34}O_2$	282.48 282.48 282.48
17	18.349	1.96	9,10-Anthracenedione, 2-ethyl-	Organic compound	$C_{20}H_{14}O_2$ $C_{18}H_{34}O_2$	319.98 282.48

			trans-13-Octadecenoic acid n-Propyl 11-octadecenoate	Fatty acid	$C_{19}H_{34}O_2$	294.48
18	19.538	2.67	trans-13-Octadecenoic acid cis-13-Octadecenoic acid cis-Vaccenic acid	Fatty acid Fatty acid Fatty acid	$C_{18}H_{34}O_2$ $C_{18}H_{34}O_2$ $C_{18}H_{34}O_2$	282.48 282.48 282.48
19	19.818	3.08	Oleic Acid trans-13-Octadecenoic acid Cyclopropaneoctanal, 2-octyl-	Fatty acid Fatty acid Cyclic compound	$C_{18}H_{34}O_2$ $C_{18}H_{34}O_2$ $C_{16}H_{32}O$	282.48 282.48 240.43
20	20.115	1.98	Octadec-9-enoic acid 9-Octadecenoic acid, (E)- Oleic Acid	Fatty acid Fatty acid Fatty acid	$C_{18}H_{34}O_2$ $C_{18}H_{34}O_2$ $C_{18}H_{34}O_2$	282.48 282.48 282.48
21	20.333	2.88	Nonanoicacid,ethyl ester cis-13-Octadecenoic acid trans-13-Octadecenoic acid	Fatty acid Fatty acid Fatty acid	$C_{18}H_{34}O_2$ $C_{18}H_{34}O_2$ $C_{18}H_{34}O_2$	282.48 282.48 282.48
22	20.728	3.29	Dodecanoic acid trans-13-Octadecenoic acid cis-13-Octadecenoic acid	Fatty acid Fatty acid Fatty acid	$C_{12}H_{24}O_2$ $C_{18}H_{34}O_2$ $C_{18}H_{34}O_2$	200.42 282.48 282.48
23	20.917	2.17	9-Octadecenoic acid, (E)- cis-Vaccenic acid Oleic Acid	Fatty acid Fatty acid Fatty acid	$C_{18}H_{34}O_2$ $C_{18}H_{34}O_2$ $C_{18}H_{34}O_2$	282.48 282.48 282.48
24	21.603	9.03	Retinol cis-Vaccenic acid	Organic compound Fatty acid	$C_{20}H_{30}O$ $C_{18}H_{34}O_2$	270.46 282.48
25	22.021	18.68	cis-Vaccenic acid trans-13-Octadecenoic acid cis-13-Octadecenoic acid	Fatty acid Fatty acid Fatty acid	$C_{18}H_{34}O_2$ $C_{18}H_{34}O_2$ $C_{18}H_{34}O_2$	282.48 282.48 282.48

UV- Visible analysis of dye extract from teak leaf (Tectonagrandis)

The UV-visible dye extract from teak leaf *(Tectonagrandis)* as presented in figure 2 appeared at a maximum wavelength of 524 nm with an absorbance of 1.30, as shown in the spectra in figure 2. These are attributed to $n\rightarrow\pi$. This suggests that the chromophores C≡N and N=N may be present (Cai et al., 2017).

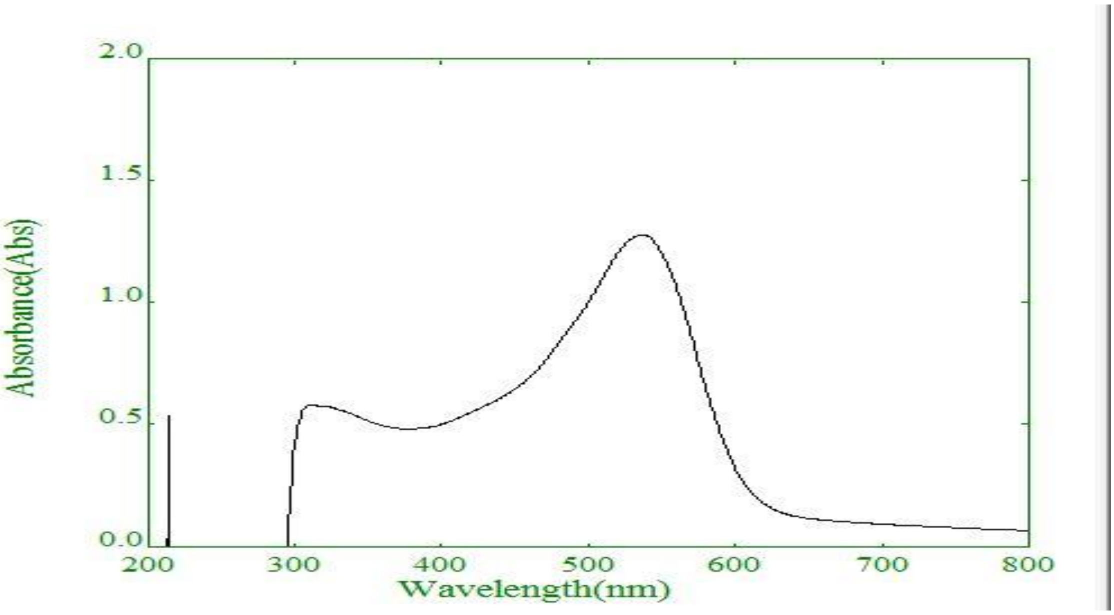

Figure 2: UV spectral of Dye extract from teak leaf *(Tectonagrandis)*

Atomic Force Microscopy Analysis of the Dye extract from teak leaf *(Tectonagrandis)*

The surface morphological properties of the dye were investigated using atomic force microscopy (AFM) of the dye extract, as illustrated in Fig. 3. At the nanoscale scale, the modified dye extract's surface structure was examined in detail. There was a noticeable level of roughness on the dye extract's surface. In the 50μm×50μm scan area, the dye extract's root mean square roughness was approximately 2568.1 nm. The surface roughness of the dye extract is 2568.1 nm, which indicates that it has a distinct texture. More active sites may be present on rough surfaces, which could enhance the dye's reactivity or ability to interact with other materials. Comprehending the surface properties is beneficial since they impact several aspects such as adhesion, light reflection and how they engage with other materials. Because of the dye extract's rough surface, more pomegranate dye can be adsorbed onto the film's surface, increasing the amount of light that is absorbed when it strikes the film. The extremely consistent color distribution in the dye's phase image suggests that the dye is evenly dispersed over the entire layer (Alsteens et al., 2017).

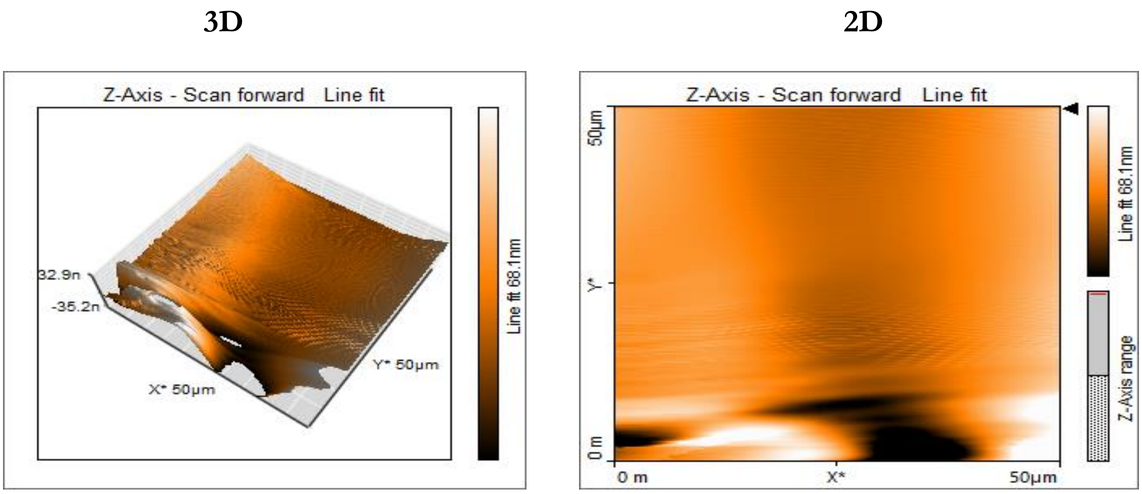
3D **2D**

Figure 3: The AFM analysis of the dye extract from teak leaf *(Tectonagrandis).*

Scouring and bleaching of Cotton fabric

As seen in figure 4, the process of scouring and bleaching cotton fabrics involved using sodium hydroxide (NaOH) to scour three cotton fabrics of varying weights and sodium hypochlorite (NaClO) to bleach them. The elimination of natural wax and non-fibrous impurities during scouring and bleaching causes the fabric to become permanently white and brighter, which desizes the cloth and reduces its weight (Fried et al., 2022). The results demonstrated a progressive decrease in the weight of the fabric after these processes.

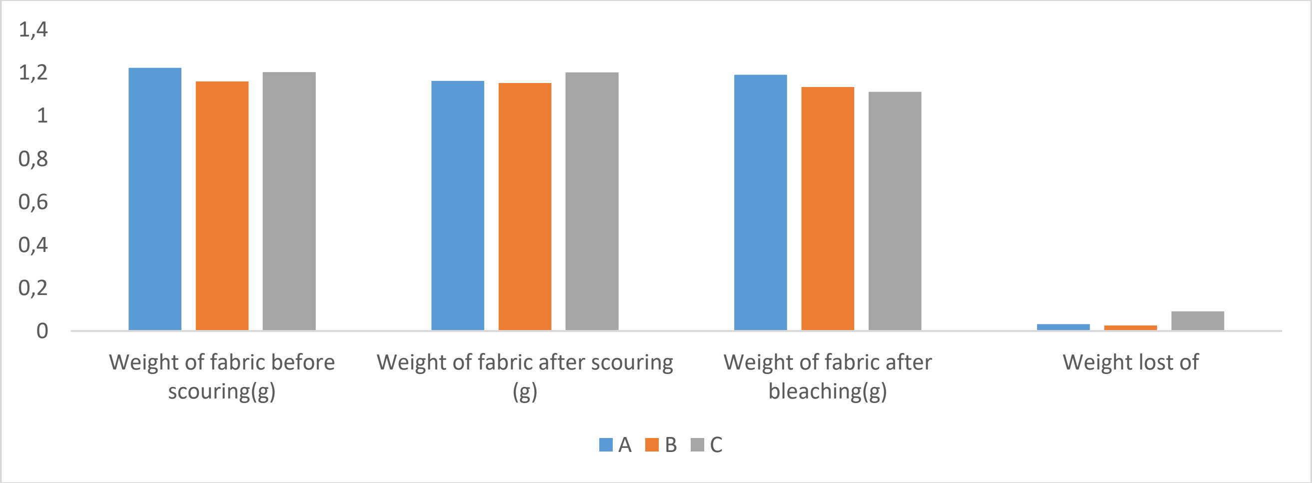

Fig 4: Scouring and bleaching of cotton fabric *Dyeing*

The weight of the three distinct fabrics increased gradually as a result of the dyeing process, as seen in figure 5. This is because the fabric's absorbency power has increased, which also raises the fabric's affinity for dyes (Otutu et al., 2012).

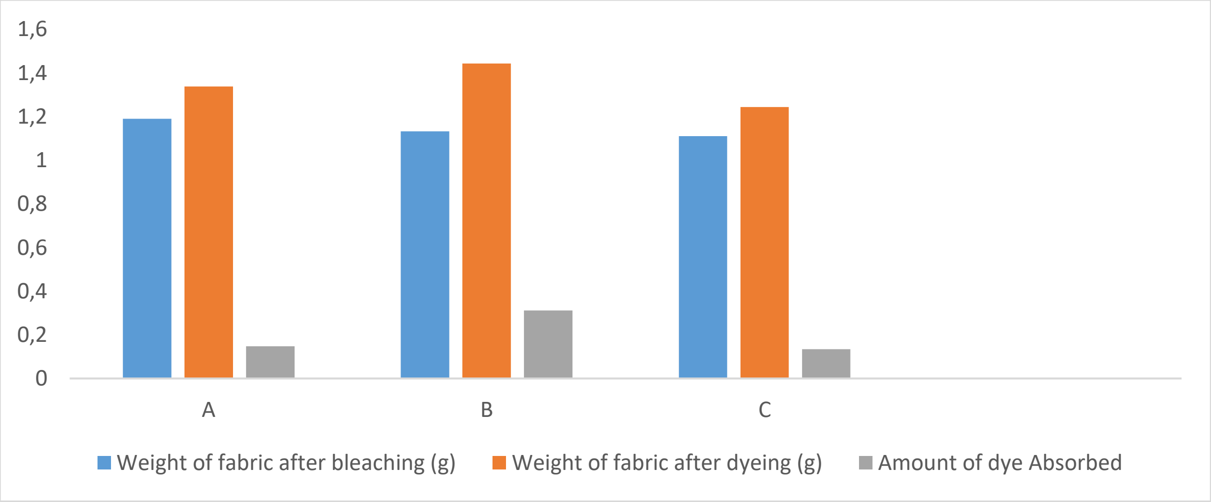

Fig 5: Results of dyeing

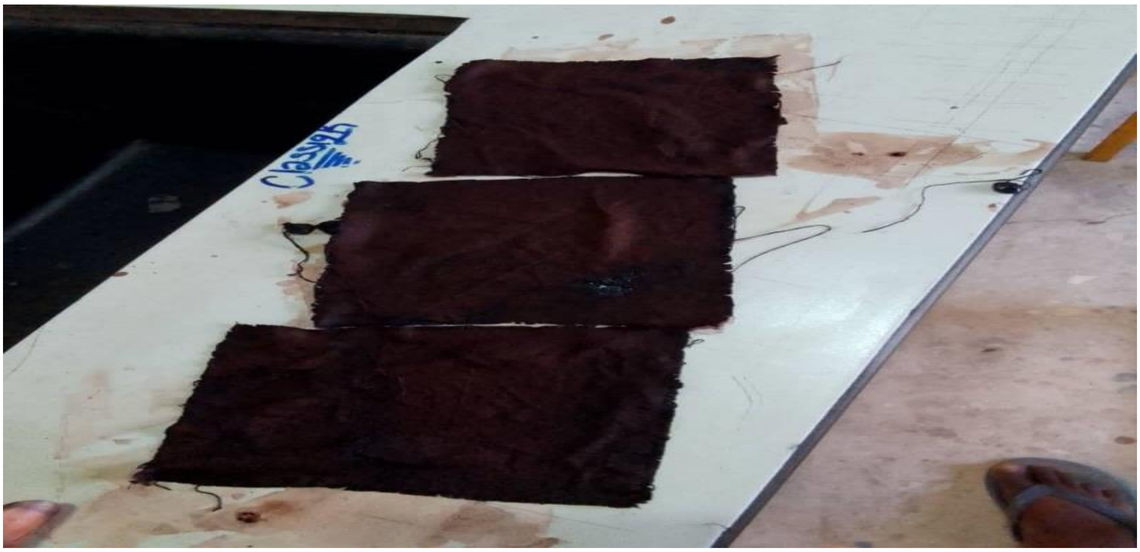

Plate 3: Dyed fabrics

Wash Fastness properties of dyed fabric

According to Table 5's results of the wash fastness properties of the teak leaf dye extract, cotton fabric dyed without the use of mordants has a fastness grade of 3–4, which is considered fair fastness. A staining grade of 4, which denotes poor staining, was observed on the nearby undyed fabric (Cai et al., 2017).

Table 5: Wash fastness properties of dyed fabric

Fabric type	Color change	Color staining
Dyed fabric	3-4	3

Wash fastness:1- very poor; 2- poor 3-fair 4- good 5-excellent, Staining of adjacent white fabric: 1- deep staining; 2- significance staining; 3- moderate staining; 4- very light staining; 5- no staining.

Plate 4: Color change and staining of dyed fabric

Rubbing Fastness properties of dyed fabric

The results of the rubbing fastness properties of the cotton dyed sample with teak leaf dye extract, as shown in Table 6, indicate that rubbing fastness grades of 3 and 2 respectively, for both dry and wet rubbing. this indicate poor fastness to rubbing (Cai et al., 2017).

Table 6:. Rubbing fastness properties of dyed fabric

Fabric type	dry rubbing	wet rubbing
Dyed fabric	3	2

Rubbing fastness; 1- very poor; 2- poor; 3- fair; 4- good; 5- excellent

Plate 5: Dry rubbing **Wet rubbing**

CONCLUSION

Natural colorant was successfully extracted from teak leaf using a soxhlet extractor with ethanol as a solvent. The dye extracts were applied to cotton fabric without mordants. The results obtained in this study suggest that the maroon-colored dye extracted from *teak leaf* possesses intrinsic affinity for cotton fabric. The observed potential affinity of the dye extracts for the textile substrates used for the study may be due to the presence of santalins and santarubin compounds. The FTIR characterization of the dye extract also suggested the presence of *N-H (amine), O-H (alcohol), C-H (alkane), C-H (alkane), C≡C (alkyne), C = C (alkene), C = O (carbonyl groups), alkyl ketone, alkyl amine, alkyl amine, and alkyl halide* functional groups in the dye components. The chromospheres in the dye extract are *C≡N and N=N,* and the AFM suggests that the area roughness was about 2568.1 nm in the 50µm×50µm scan area. The color fastness to washing shows a 3-4 and 3 for color change and staining, respectively. While fastness to rubbing shows 3 and 2 for dry and wet rubbing, respectively.

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