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Study on dyeing ability of cotton fabric 100% with naphthaquinone extracted from nipa coconut shells

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Abstract

Natural dyes are known widely thanks to friendly properties to envi**ronment** as health protecting ability for human. In this study, naphthaquinone of nipa coconut shells extracted by the reflux method that created effectively. The influences of ratio of dye extract, temperatures and time of dyeing and types of mordants based on the color strength (K/S) and color difference (ΔE) were studied. Our study applies the methods of extracting colorants in ethanol **solvent**, dyeing on an infrared dyeing machine**, Mesdan** by the exhaust **dyeing** method, measuring color on an X-Rite Color i5 spectrometer machine, analyzing **effecting parameters by** CIELab **popular** color space, and measuring Fourier-transform infrared (FTIR) spectroscopy to evaluate the presence of naphthaquinone in dye extract **and bonding on fabric**, **testing more** scanning electron microscopy (SEM) to observe the surface of **dyed** samples **clearly**. As results, the Nipa fruticans shell extract can dye 100% cotton fabric with a color difference ΔE of 41.07 and a K/S color strength of 12.96 when dyed with 10% $CuSO_4.7H_2O$ because of their complex formation with the colorant with cellulose. Color fastness to washing on dyed cotton **samples** according to the ISO 105 – C06 A1S and ISO 105-X12 standard were tested to clarify further impacts of mordants to dye ability.

Keywords: cotton, dyeing, fabric, coconut, shell, extract, color strength, color difference

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1. Introduction

Quinones are widely known for their vibrant coloration and significant biological activities, with naphthoquinones being the second most commonly occurring quinone class in nature. These compounds possess two carbonyl groups attached to a benzene ring, often positioned in either the ortho or para configurations. Their structure includes α,β-unsaturated carbonyl systems, which allow for extensive electron delocalization. This delocalization contributes to their strong absorption in the visible light region, producing intense colors. Among the isomers, 1,4-naphthoquinone is the most abundant and chemically stable. Naphthoquinones are found in a wide range of organisms, including higher plants, bacteria, and certain animals and fungi. In plants, they may occur alongside anthraquinones, particularly in families such as Bignoniaceae and Verbenaceae, and are typically present either in free form or as glycosides [1–4].

Several naturally occurring naphthoquinones—such as lawsone, juglone, alkannin, and lapachol—have been identified as key contributors to plant pigmentation. Their vivid coloration, combined with potential antimicrobial and antioxidant properties, has attracted interest from researchers in the textile industry. Studies have explored the use of these natural colorants on textile substrates like cotton, wool, and silk through innovative approaches, including plasma treatment, bio-based mordants, and machine learning-assisted dye prediction [5–8]. The use of plant-derived dyes is increasingly favored for their eco-safety and potential health benefits, aligning with current trends in sustainable and functional textiles.

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Cotton, as a widely used natural fiber, is composed of linear cellulose chains with an average diameter of 0.8 nm. It consists of approximately 5000 repeating cellobiose units and exhibits both crystalline (65–70%) and amorphous (30–35%) domains, affecting its dye uptake characteristics [9,10].

In the context of exploring novel dye sources, nipa palm (Nypa fruticans), the plant propagates through water currents and is widespread along the coasts and estuaries of regions surrounding the Indian and Pacific Oceans, from Bangladesh to the Pacific Islands, commonly known as the water coconut, represents a promising yet underutilized biomass. This plant thrives in brackish, tidal environments across Southeast Asia. In Vietnam, nipa palms are found predominantly in the Mekong Delta and South Central Coast, including provinces such as Ben Tre, Tra Vinh, and Ca Mau [11–15]. Their durable, lignocellulosic shells are rich in phenolic compounds, offering potential as a sustainable source of natural dyes.

Previous investigations have confirmed that factors such as pH, temperature, and dyeing time significantly influence dye uptake and color development on cotton fabrics [16–18]. These effects are often attributed to changes in fiber surface chemistry and the interaction between hydroxyl groups and mordants during the dyeing process [20–22].

Standard analytical tools, including Fourier-transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM), have been employed to investigate the bonding mechanisms and surface morphology changes resulting from various dyeing parameters. Such methods have contributed to improving the color fastness and overall performance of natural dye applications on cotton [23,24].

This study contributes to the advancement of sustainable textile dyeing in several meaningful ways. It introduces nipa palm shell extract as an innovative and underutilized natural dye source for cotton fabrics, with potential environmental and economic benefits. By systematically examining the effects of dyeing parameters such as pH, temperature, dyeing time, and mordanting conditions, this work provides valuable insights into the dye-fiber interaction mechanisms. The application of FTIR and SEM techniques further clarifies the chemical bonding and morphological changes occurring on cotton surfaces after treatment, thereby enhancing understanding of dye fixation and color fastness. Ultimately, the research supports the valorisation of agricultural by products particularly nipa palm shells into functional materials, aligning with the principles of green chemistry and promoting circularity in the textile industry.

2. Materials and methods

After harvesting, the water coconut will be split into small parts, remove the rice, collect the shell and young fruits, wash to remove impurities, mud, and sand. They are then dried at 80°C and ground into a fine powder using a blender. Finally, it will be screened to remove the coconut fibre, which obtains water coconut powder. Dissolve water coconut powder in Ethanol solvent 99° with extraction rates according to the experimental survey plan. Seal the jar with foil to limit evaporation of the solution so that the solution stays at room temperature for 48 hours. Then the solution is filtered through filter paper, and the extract from the coconut shell is obtained. Cotton parameters are shown as Table 1.

Table1. Table of fabric raw material parameters

Sample's Information	Content
	100% Cotton (C.0.0)
Weaving style	Woven fabric, dot pattern
Yarn Index	30 Ne
Fabric density	Pd = 260/10cm; Pn = 160/10cm

The effectiveness of dyeing fabrics with water coconut shell extract will be affected by the following factors: (1) extraction rate, (2) dye pH, (3) dyeing temperature, (4) dyeing time, (5) mordants. Therefore, in this project, the following surveys will be carried out:

Survey 1: The effect of the dyeing extraction rate affects the color strength, changing the dyeing extraction ratio is 1:15, 1:20, 1:25 respectively.

Survey 2: The effect of the pH of the dyeing solution affects the color intensity, the change in the pH of the dyeing solution is pH ≈ 3, pH ≈ 5, pH ≈ 7, pH ≈ 9, pH ≈ 11, respectively.

Survey 3: The effect of the dyeing temperature affects the color intensity, the dyeing temperature changes are 60°C, 80°C, 90°C, respectively.

Survey 4: The effect of dyeing time affects the color intensity, changing the dyeing time is 45 minutes, 60 minutes, 75 minutes, respectively.

Survey 5: The effect of color mordants affects color intensity, changing mordant types are Tannin, $CuSO_4.5H_2O$, $KAl(SO_4)_2.12H_2O$.

The instruments and equipment in the research including a heating magnetic stirrer (Velp, Italy), electronic scale (Ohaus PX224E, USA), infrared laboratory dyeing machine (Mesdan, Italy) for textile laboratory, dryer (Gavazzi) were used for preparation and treatment, pH differential method (S220, Metter Toledo), scanning electron microscopy (SEM, TM4000Plus, Hitachi), Fourier-transform infrared spectrometer (FTIR, Brucker Tensor 37) and spectrophotometer machine (X-rite Color i5D, USA) were used to clarify contents of colorants as well as dyeability of dyed cotton fabrics. Color fastness of dyed fabrics to washing and rubbing was investigated according to the international standard method (ISO 105 – C06 A1S and ISO 105-X12).

Measurements
Color strength (K/S)
K/S is a function of color depth and is calculated as below:

$$K/S = \frac{(1-R)^2}{2R}$$

where R is the surface reflectance coefficient, K is the light absorption coefficient, S is the light scattering coefficient.
Color difference (ΔE)
In the CIELab color space, color comparison is made through the total color difference between two samples, denoted as ΔE*. This value is divided into lightness difference (ΔL*) and chromaticity differences (Δa* and Δb*).

The total color difference ΔE* is calculated as follows:

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$$

3. Results and discussion

3.1. Evaluation of efficiency of water coconut shell extraction

Water coconut shell powder is dissolved in Ethanol 99° at a ratio of 1:15 which is soaked for 48 hours at room temperature. After filtration, the dyeing solution volume and performance are obtained at Table 2.

Table 2. Efficiency of water coconut shell extraction

Initial Weight (g)	Solvent Volume (ml)	Volume of extracted solution (ml)	Acquisition Rate (%)
75	750	548	73.07
75	750	547	72.93
75	750	551	73.33
75	750	549	73.20
75	750	546	72.80
Average performance			73.06

It can be seen that the extraction efficiency from water coconut shell is at a relative level, the reason is that water coconut shell powder contains a lot of fiber, can be insoluble in solvent and absorbs a certain amount of solvent.

3.2. Evaluation of effect of dye extraction rate on color strength

To assess the effect of dye extraction rate on the color absorption of fabric of 100% Cotton according to survey 1, the fabric samples were measured on the X-rite Color i5 spectrophotometer. The average values at the wavelength of 380 nm were obtained in Table 3.

Table 3. Parameter values ΔL*, Δa*, Δb*, ΔE_{CMC} and K/S of 100% Cotton fabric according to the extraction ratio

Samples	ΔL*	Δa*	Δ b*	ΔE_{CMC}	K/S
C.0.0					9.66
C.1.1 (1:15)	-19.46	4.62	25.31	30.94	12.42
C.1.2 (1:20)	-23.97	8.14	20.54	27.11	12.08
C.1.3 (1:25)	-13.85	7.62	19.52	24.73	3.31

These results revealed that both the color difference (ΔE) and the color strength (K/S) achieved peak values at an extraction ratio of 1:15 for 100% cotton fabric (ΔE = 30.94; K/S = 12.42). When the ratio increased to 1:20 and 1:25, a gradual reduction in these values was observed. Additionally, the L* parameter showed a 21.88% decrease in brightness, while the b* value reached 10.32 in the C.1.1 sample, indicating a tendency toward a golden tone. These results are consistent with the mass transfer theory, which posits that the concentration gradient between the solid material and the solvent is the main driving force in extraction processes. At higher solvent volumes, the concentration gradient initially increases, facilitating more effective pigment migration. However, once equilibrium is reached, further increases in solvent volume do not enhance extraction efficiency. Therefore, the extraction ratio of 1:15 is considered the most suitable for 100% cotton under the conditions investigated.

3.3. Evaluation of effect of pH on color strength

In order to evaluate the effect of pH on the color absorption of Cotton according to survey 2, the fabric samples were also measured on the X-rite Color i5 spectrophotometer. The average value of color difference ΔE and color intensity K/S at the current wavelength of 380nm are taken in Table 4 and Figure 1.

Table 4. Parameter values ΔL*, Δa*, Δb*, ΔE_{CMC} and K/S of 100% Cotton fabric according to pH

Samples	ΔL*	Δa*	Δ b*	ΔE_{CMC}	K/S
C.0.0					9.66
C.2.1 (pH≈3)	-11.58	-0.21	26.20	30.92	11.32

C.2.2 (pH≈5)	-13.08	4.44	28.08	33.81	11.83
C.2.3 (pH≈7)	-19.15	5.15	25.36	31.12	12.59
C.2.4 (pH≈9)	-24.05	9.87	33.25	40.17	12.72
C.2.5 (pH≈11)	-28.40	11.12	24.50	33.23	12.42

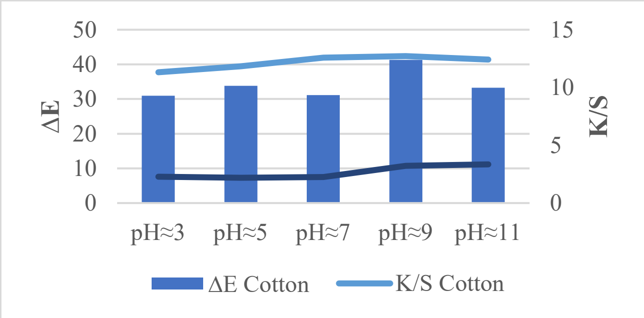


Figure 1. ΔE color difference and K/S color strength by pH

The brightness of the dye sample decreases as the pH of the dye solution increases (pH from 3 to 11). The pH of the dyeing tank has little effect on the a∗ value of the dyeing sample but it has a significant effect on the b∗ value of the dyeing sample. The dye samples become more golden as the pH of the dyeing tank rises to the optimum level, which can also be seen in the spectral measurements of the dyed fabric samples. After dyeing in 3 pH environments, 100% Cotton samples have a gradual decrease in brightness, redness and yellowness increase sharply from acid dyeing medium to alkaline environment. The overall color difference is evident in the ΔE value, which proves that the sample color is dyed 100% Cotton in an alkaline environment for the darkest color. With the color difference values ΔE and K/S color intensity increasing steadily and reaching their highest values at pH ≈ 9 (ΔE = 40.17 and K/S = 12.72) then when the values began to decrease when the pH was increased to near 11, indicating that pH ≈ 9 was the best pH for the 100% Cotton sample.

3.4. Evaluation of effect of dyeing temperature on color strength

The dyeing temperature is an important factor affecting the dyeing quality, as it affects the kinetic energy of the dye molecules in the dyeing tank and the degree of expansion of the cellulose fibers.

Based on the data in Table 5 and Figure 2 we can notice that as the temperature increases, the L* value begins to decrease.Color difference value ΔE and color intensity K/S both fabrics reach the highest value at 60°C with 100% Cotton fabric (ΔE = 39.30 and K/S = 12.568) then began to gradually decrease when the temperature ratio increased to 80°C - 90°C. In addition, based on the L* value, the brightness decreases (12.10% decrease), the golden light is clearly shown with a b* value of 14.91, corresponding to the 100% Cotton sample. So the best dyeing temperature for 100% Cotton fabrics is 60°C.

Table 5. Parameter values ΔL*, Δα∗, Δ b*, ΔE_{CMC} and K/S of 100% Cotton fabric according to temperature

Samples	ΔL*	Δa*	Δ b*	ΔE_{CMC}	K/S
C.0.0					9.67
C.3.1 (60°C)	-11.58	-0.21	26.20	39.30	12.57
C.3.2 (80°C)	-13.08	4.44	28.08	27.54	11.44
C.3.3 (90°C)	-19.15	5.15	25.36	32.05	11.73

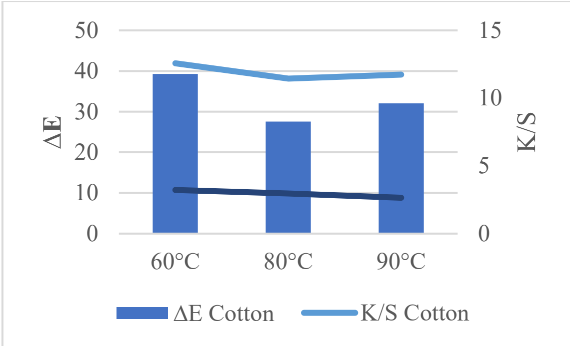


Figure 2. ΔE color difference and K/S color strength by temperature

3.5. Evaluation of effect of dyeing time on color strength

Dyeing time is an important factor that affects dye absorption, color uniformity, and color fastness. In actual production, the dyeing time can be appropriately shortened according to the properties of the dye and the requirements of the dyeing color.Based on the data in **Table 6** and **Figure 3,** We can see that fabrics were similar when from the 45-minute period, the ΔE and K/S values gradually increased and reached their highest values at 60 minutes with 100% Cotton (ΔE = 39.24 and K/S = 12.312) then began to decrease when the time was increased to the 75-minute time. In addition, for 100% Cotton fabrics, L* brightness can be reduced (down 33.84%), showing a golden sheen with a b* value of 14.08. So the most suitable dyeing time for fabric 100% Cotton are 60 minutes.

Table 6. Parameter values ΔL*, Δa*, Δb*, $ΔE_{CMC}$ and K/S of 100% Cotton fabric according to time

Samples	ΔL*	Δa*	Δ b*	$ΔE_{CMC}$	K/S
C.0.0					9.67
C.4.1 (45 minutes)	-29.60	7.26	16.37	24.93	9.16
C.4.2 (60 minutes)	-22.09	6.04	29.07	39.24	12.31
C.4.3 (75 minutes)	-23.58	4.32	14.39	20.45	10.23

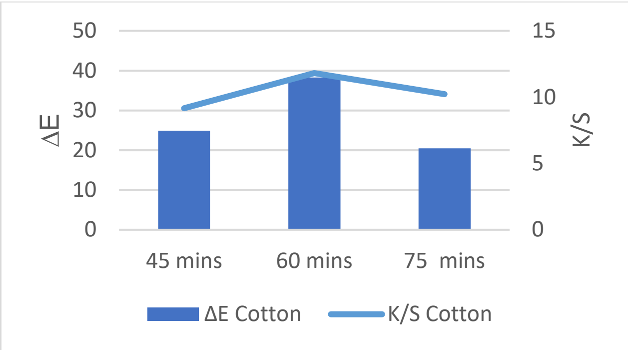


Figure 3. ΔE color difference and K/S color strength by time

3.6. Evaluation of effect of mordants on color strength

The cellulose polymer structure has hydroxyl (–OH) groups as the binding site responsible for the absorption of dye molecules into the fiber. However, cellulose fibers have low strength to the natural colorant Napthoquinone resulting in poor dye affinity. This restriction occurs due to the polarizing properties of cellulose and different polar Napthoquinone dyes that inhibit stronger dye-cellulose bonds, as well as the complexity of their molecules that limit dye diffusion. To improve this limitation, using mordants helps not only to improve the color of the dyed fabric, but also enhances the color fastness. The color improvement is clearly shown in **Table 7**.

100% Cotton samples after being colored for darker shades. Based on the data table Table 3.6 see that the color difference value of ΔE and K/S color intensity reaches the highest (ΔE = 41.07 and K/S = 12.96) when holding the color with $CuSO_4.5H_2O$. In addition, it can be seen that when holding the color with $CuSO_4.5H_2O$, the L* brightness value decreases and shows more red than yellow. Moreover, bonding mechanism between Cellulose – Metal salts– Napthoquinone Colorant as shown in Figure 4.

Table 7. Parameter values ΔL*, Δa*, Δb*, $ΔE_{CMC}$ and K/S of 100% Cotton fabric according to various mordants

Samples	ΔL*	Δa*	Δ b*	$ΔE_{CMC}$	K/S
C.0.0					9.66
C.5.1 (No mordanting)	-24.05	9.87	33.25	40.17	12.63
C.5.2 (Tannins)	-27.86	10.57	27.41	35.91	12.13
C.5.3 ($CuSO_4.5H_2O$)	-31.14	13.62	28.59	41.07	12.96
C.5.4 ($KAl(SO4)_2.12 H_2O$)	-21.69	10.84	22.28	31.74	11.72

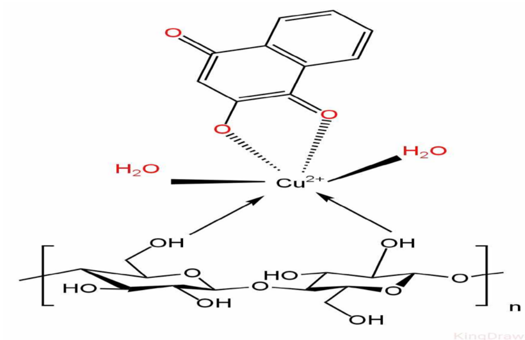


Figure 4. Bonding Mechanism

3.7 Results of measuring fabric color fastness after dyeing

The fabric is dyed with an extract from the peel of a water coconut with medium washing fastness, the washing fade is 3 or 3-4 out of a point of 5, depending on the type of fabric and the mordant type that we use. This is a relatively good result for a naturally derived extract. If no mordanting is used, the color fastness result achieved is level 3. However, if you use more the mordant, the color fastness of the laundry is improved in **Table 8**.

Table 8. Washing color fastness results

Sample	Fading	(Color Wiring on Gray Scale) Acetate Cotton Nylon Polyester Acrylic Wool
C.5.1	3	4-5
C.5.3	3-4	4-5

Fabrics dyed with liquid coconut shell extract have relatively high friction strength for both longitudinal and horizontal strength, dry strength is achieved at 4 or 4-5, wet strength is achieved at 3 or 3-4 on a 5-point scale. As such, wet strength is less durable than dry strength. This is a good result for a naturally derived extract. If no mordant is used, the friction color fastness result is 3 or 4. However, if more mordant are used, the friction color fastness is improved in **Table 9**.

Table 9. Friction Strength Results

Sample	Dry	Wet
C.5.1	4	3
C.5.3	4-5	3-4

3.8 FTIR Infrared Spectroscopy Results

Fabric samples were analyzed by FTIR spectroscopy to identify structural changes due to the effects of dyes and mordants.

FTIR Spectrum of 100% undyed Cotton Sample in **Figure 5a, 5b**.There is a fairly large number of peaks indicating that the material contains a variety of compounds and functional groups. Featured peaks include: Wide peak at 3332.8 cm^{-1} (OH group in cellulose). The peak of 2900.2 cm^{-1} is the C-H elongation oscillation in the saturated hydrocarbon circuit). The absorption bands at 1425.7 cm^{-1}, 1314.5 cm^{-1}, 1204.1 cm^{-1}, 1029.5 cm^{-1} are the elongated and bending oscillations of -CH_2 and -CH, -OH and C=O bonds in cellulose. The FT-IR spectrum is based on the oscillation of the bonds in the molecule to determine which groups are present in the molecule based on the absorption capacity of the groups. The results of the FT-IR spectrum of 100% Cotton fabric before and after dyeing both showed the absorption spectrum of the characteristic groups O-H (3331.1 cm^{-1}), C-H (2899.6 cm^{-1}), etc .These are the groups that characterize the composition of cellulose fabric and the pigments naphthaquinone and tannins present in the extract from coconut shells. Based on the results of the collected peaks, it can be predicted that the functional groups present in the 100% Cotton fabric sample after dyeing with water coconut shell extract, the characteristic peaks obtained of the post-dyed fabric sample have a gradually increasing optical transmittance compared to the pre-dyed fabric. The change in the fabric after dyeing is due to the appearance of peaks at 1621.9 cm− 1 (C=O group) and the appearance of new absorbent bands in the region of 1100-1200 cm− 1, which is the region characteristic of the C=O bonds in the carbonyl group of Napthoquinone. This allows it to be determined that these functional groups in the composition of the water coconut shell extract are involved the process of dyeing cellulose fabrics.

Figure 5c:The FTIR spectra of samples stained with $CuSO_4.5H_2O$ coconut shell extract with a concentration of 10% included peaks of 3336.0 cm^{-1} (OH group), 2904.2 cm^{-1} (C-H group), peaks 1505.5, 1408.5, 1339.5 cm^{-1} (C-H, C-O and C-C bonds) and peaks 1244.3, 1095.3, 1057.6, 1017.7 cm^{-1} (C-O-C and C-OH bonds) corresponding to the position shift and increasing optical transmittance with pre-dyed fabrics and non-stained fabrics that occur as a result of the association between the dye, the pigment and the cotton fabric increases the intensity of the absorption peaks.

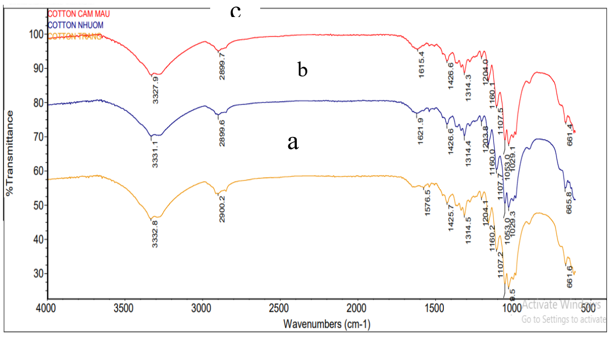
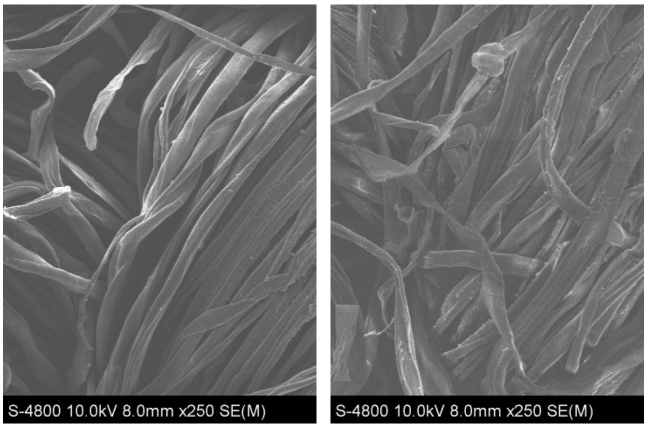


Figure 5. FTIR Spectrum of Samples (a) 100% Undyed Cotton ----; (b)100% Cotton Dyed no mordanting; (c) 100% Cotton Dyeing with mordant----

3.9 Surface assessment by SEM Analysis

The SEM structural analysis sample is a standard 100% Cotton sample and a 100% Cotton dyed sample with the best conditions, mordanted with$CuSO_4.5H_2O$ 10%. The sample was taken with X250, X1000 magnifications, the results are shown in the **Figure 6.**



(a) Undyed (b) Colored dyed sample

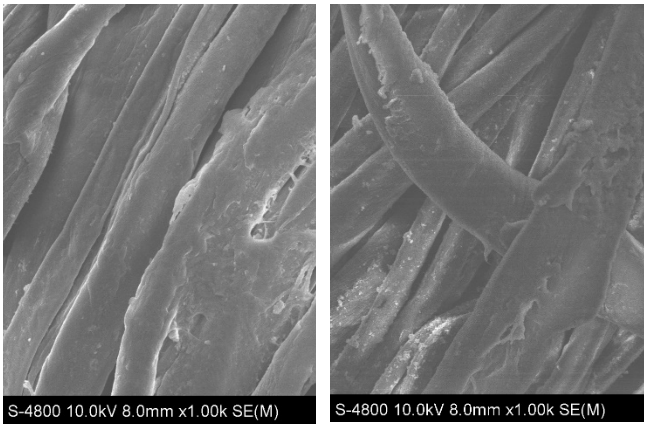


Figure 6. SEM image of the 100% Cotton sample at X250, X1000 magnification

Looking at the results, at the X1000 magnification for 100% Cotton fabric, the image shows that along the surface of the fibers there are debris and tiny particles clinging around. It can be said that the surrounding thin film layers are formed from colorants, these fragments and tiny particles are made up of a combination of material particles of water coconut shell extract. As it turns out, the colored elements have bonded to the fabric, causing the surface structure of the fabric to change after dyeing.

4. Conclusion

Through the study process, conducting various investigations on 100% Cotton fabric from water coconut shell extract carried out on an infrared dyeing machin under various effects e, we has achieved quite good results. For 100% Cotton fabric: the extraction ratio is 1:15, pH ≈ 9, 100% dye concentration, temperature 60°C for 60 minutes, use $CuSO_4.7H_2O$ mordant. Natural dyes have always been a leading object for scientists to make more friendly and safer environments. The color fastness of dyed cotton samples with mordant was still higher than that of dyed samples without mordants. Accordingly, concentration of used mordant should be used the most suitable, it could optimize dyeing condition and affect the least environment around and our health. Moreover, future research can aim to further optimize the microwave-assisted dyeing process compare to conventional method for a wider range of various fabrics from this natural dye, with a focus on examining the effects of various pretreatment techniques on dye absorption and fabric quality. Additionally, investigating the scalability of this method for industrial use is essential, particularly in terms of integrating sustainable practices into existing dyeing operations. A comprehensive assessment of sustainability, including energy consumption and the environmental impact of chemicals, should also be prioritized.

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