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Ultrasonic-assisted sustainable extraction and dyeing of organic cotton fabric using natural dyes from *Dillenia indica* leaf

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ABSTRACT

As a means of preventing environmental damage caused by synthetic dyes, eco-friendly textile dyeing with natural dyes is gaining popularity worldwide. This study focused on the extraction of dyes from the leaf of *Dillenia indica* (*D. indica*) tree using an ultrasonic extraction technique and applied on the organic cotton fabrics. The ultrasonic method was used for both extractions of *D. indica* dyes and dyeing of organic cotton fabrics. Here, the amount of *D. indica* powder used were 5% and 6.67% for producing light and dark shade, respectively. The investigation of the color fastness to washing, rubbing, and light for the dyed organic cotton fabrics indicated an excellent rating. The spectrophotometric analysis revealed the L* (lightness or darkness), a* (redness or greenness), b* (yellowness or blueness), C* (chroma), h* (hue), R% (reflectance), and K/S (color strength) values, which accurately represented the shade of the dyed organic cotton fabric. To understand the interaction between *D. indica* dye and organic cotton fabrics, different characterization including, Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) were performed. The characterization outcomes confirmed the successful deposition of *D. indica* dyes on the organic cotton fabrics. The other comparable testing results such as bursting strength, air permeability, and thermogravimetric analysis (TGA) of dyed and undyed organic cotton fabrics were in the acceptable range. One of the important findings of this research was no chemicals were utilized during the extraction and dyeing of organic cotton fabrics. This process can be referred to as completely chemical-free and advantageous for the environment because no chemicals were needed during extraction or dyeing. Therefore, the natural dye extracted from *D. indica* is extremely promising and could be a viable option for the sustainable dyeing of cotton fabrics in the textile dyeing industry.

1. Introduction

Since the mid-nineteenth century, the invention of the synthetic dye 'mauve' has sparked an avalanche of discovery within synthetic chemistry, and with the advent of industrialization, synthetic dyes have replaced natural dyes in numerous uses, including cosmetics, textiles, and food coloring [1]. Despite the fact that these dyes are commonly inexpensive, generate a wide range of colors, and have high fastness qualities, they can also be allergenic, poisonous, and even carcinogenic, which are hazardous to individuals and the environment [2]. Industrial wastewater resulting from synthetic dyes contains a variety of pollutants, including salts, aromatic

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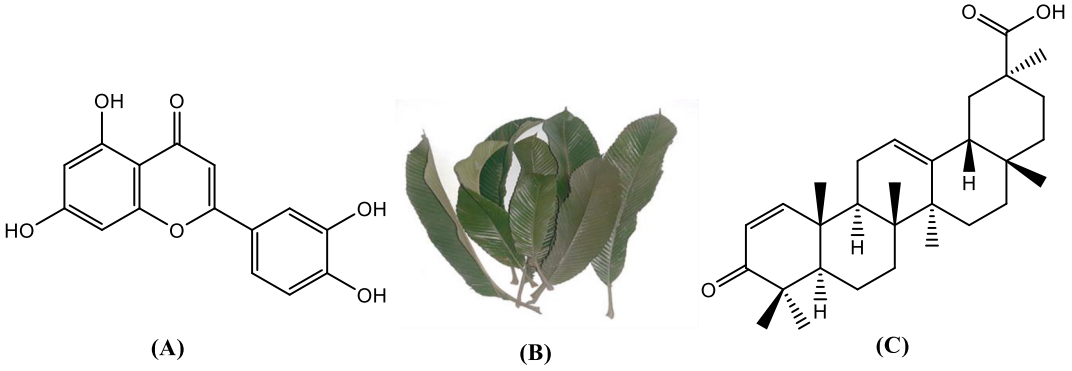
amines, phenolic compounds, hydrocarbons, acids, alkalis, and heavy metals like nickel, cadmium, mercury, and chromium. These constituents render the wastewater complex, toxic, and challenging to treat using natural resources [3]. It is generally accepted that natural dyes are less toxic than synthetic ones, and that the effluent from their use is less hazardous to aquatic life. The growing anxiety for the health of the environment and the viability of future generations is driving a movement toward the use of sustainable dyes [4].

Researchers in the dying industry are continuously exploring new plant sources to discover natural dyes and pigments that can address the environmental concerns associated with most synthetic and natural dyes [5]. In this context, we conducted experiments to extract natural colors from waste onion peel [6], black rice [7], gardenia yellow [8], tulsi leaves [9], *Butea monosperma* plants [10], *Alkanna tinctoria* roots [11], Coral Jasmine flower [12] and neem leaves [13]. However, a significant drawback of natural dyes is their limited affinity for textile materials. To tackle these challenges, various modern procedures have been employed for dye extraction and dyeing of fibers and fabrics. For example, plasma technology used for increase the color stability in natural dyeing which have several drawbacks including cost, energy consumption, complex operating process, limited scalability, material compatibility as well as environmental impact [14,15]. The entire natural dyeing can receive a homogeneous and controlled coloration from gamma ray irradiation; however, these rays emit ionizing radiation, using them calls for stringent safety measures [16]. To protect personnel and guarantee that radiation safety laws are being followed, proper shielding, monitoring, and safety training are important [17]. The speed and color yield benefits of microwave-assisted extraction have been demonstrated, however not all natural dyes may respond to it in the same way [18]. The dye source, the chosen solvent, and the particulars of the operation can all affect the extraction efficiency. To get the desired results, the microwave extraction conditions for each dye substance must be optimized [19]. The ultrasonic extraction method is considered as a "Green Process" because its many benefits [20]. The ultrasonic extraction technique uses high-frequency sound waves to extract chemicals from a variety of materials, including plant components. It is a non-invasive and efficient approach for extracting target molecules that does not require harsh solvents or high temperatures. The item to be extracted is placed in a vessel together with a suitable extraction solvent [21]. The solvent used is determined by the chemicals being extracted. The solvent aids in the dissolution and extraction of desirable chemicals from plant material. Once the material and solvent are in the tank, an ultrasonic generator generates and transmits ultrasonic waves into the mixture. These waves create alternating sequences of high and low pressure, resulting in the construction and quick collapse of minor bubbles recognized as cavitation bubbles. This process causes significant local heating and pressure, which aids in the breakdown of plant cell walls and the release of target substances into the solvent [22]. The extraction procedure is normally carried out at controlled temperatures in order to maximize extraction efficiency while maintaining the integrity of the extracted chemicals. The extraction process can take a long time depending on the item being extracted and the desired extraction efficiency. After the extraction is finished, the mixture is normally filtered to separate the extracted solution from the solid plant residue [23]. The ultrasonic treatment carried out at lower temperatures enhances the temperature sensitivity of the extract's bioactive components, producing a high dye extraction yield. Due to mass transfer kinetics, the ultrasonic treatment also has the benefit of improving functional component separation [24]. Therefore, the ultrasonic treatment has great potential in extracting physiologically active functional chemicals utilized for coloring textile materials, and it also proves to be a clean, uniform, time-efficient, and cost-effective procedure. The current study investigates the use of *Dillenia indica* leaves as a source of natural dye for dying organic cotton garments by utilizing the ultrasonic treatment.

The *D. indica* belongs to Dilleniaceae family includes the genus *Dillenia*, which has over 100 recognized species, also referred to as the Elephant Apple or Chulta. The name "*Dillenia*" comes from British botanist Joannes Jacobus Dillenius, who is credited with helping to identify this genus [25]. The *Dillenia* tree is a large, evergreen tree native to Southeast Asia, including countries like Thailand, Bangladesh, India, and Myanmar. It produces mature sweet-sour fruits that can be consumed fresh, cooked, or processed into jams and jellies. However, it is worth noting that the unripe fruit is acidic and astringent, making it unsuitable for direct consumption [26]. Apart from its culinary uses, various parts of the *Dillenia* tree, such as its fruits, leaves, and barks, have been traditionally employed for medicinal purposes. They are believed to possess therapeutic properties and have been used to treat ailments such as fever, constipation, diarrhea, stomach ache, and other health conditions [27]. Different research suggested that the leaves of *D. indica* have many coloring constitutions including flavonoids, and triterpenoids (Scheme 1) [28]. The physical appearance of the leaves of *D. indica* is shown in Scheme 1B. The structures also suggested that it has a functional group which can make a chemical interaction between textile materials. Consequently, several researchers have utilized the derived substance from *D. indica* in a variety of applications.



Scheme 1. (A) Flavonoid structure, (B) *Dillenia indica* leaf, (C) Triterpenoids structure.

Accordingly, Mohanty et al. conducted a study that focused on the quick synthesis of stable silver nanoparticles using an aqueous medium. They utilized a phenolic-rich ethanolic bark extract from *D. indica*, which exhibited potent free radical scavenging and reducing properties [29]. Sett et al. introduced a distinctive approach for the production of gold nanoparticles using the aqueous fruit extraction of *D. indica* [30]. Swargiary et al. employed an environmentally friendly and cost-effective method using an aqueous fruit extract of *D. indica* to synthesize silver nanoparticles from silver nitrate. These silver nanoparticles were utilized for the decrease of methylene blue dye [31]. Krishnan et al. investigated the use of the leaf extraction of *D. indica* as a source of selenium nanoparticles with potential insecticidal and antimicrobial properties against vector mosquitoes and pathogenic microbes [32]. As of now, no research has been conducted on using naturally extracted *D. indica* dye for coloring organic cotton fabrics. Hence, the main focus of this research was to extract natural dyes from *D. indica* and dye organic cotton fabrics using ultrasonic methods.

Organic cotton plays a crucial role in promoting eco-friendliness in the textile industry. The term "organic" indicates that it is ploughed without the use of commercial agrochemicals such as pesticides, manures, or genetically modified organisms [33]. Prior to obtaining organic certification, traditional pesticides like defoliants, herbicides, and insecticides must be avoided for a minimum of three years. Organic cotton supports biodiversity, safeguards human well-being and the environment, while preserving natural processes [34]. For instance, Karadag et al. explored the dyeing of organic cotton fabrics knitted textiles using yarrows (*Achillea biebersteinii* and *Achillea millefolium* L.) [35]. Güzel et al. conducted research on dyeing organic cotton fabrics with interlock knitted fabric using gallnut (*Quercus infectoria* Olivier) [36]. Buyukakinci et al. demonstrated the coloration of organic cotton fabrics through microwave treatment and conventional dyeing methods using barberry (*Berberis vulgaris* L.), dyer's oak (*Quercus infectoria* Olivier), and dyer's oak + barberry [37]. Mahmud et al. functionalized organic cotton fabrics using eco-friendly synthesized silver nanoparticles [38]. Howsoever, to the best of our information, no research has been carried on dyeing organic cotton fabrics using extracted dyes from *D. indica*.

As a result, natural dyes derived from *D. indica* have been used in this study to attempt to color the organic cotton fabrics. The study investigated the resistance of colored organic cotton fabrics to wash, rubbing, and light. Various characterization techniques, including Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM), were employed to analyze the dealings between *D. indica* dye and organic cotton fabrics. The characterization findings verified that the *D. indica* dyes were successfully deposited on the organic cotton fabrics. The thermogravimetric analysis (TGA) findings of dyed and undyed organic cotton fabrics, as well as other equivalent testing outcomes, were within the acceptable range. This process may be referred to as completely chemical-free and advantageous for the environment because no chemicals were needed during extraction or dyeing. As a result, the natural dye obtained from *D. indica* is really promising and may be a good choice for the environmentally friendly dying of organic cotton fabrics in the textile dyeing industry.

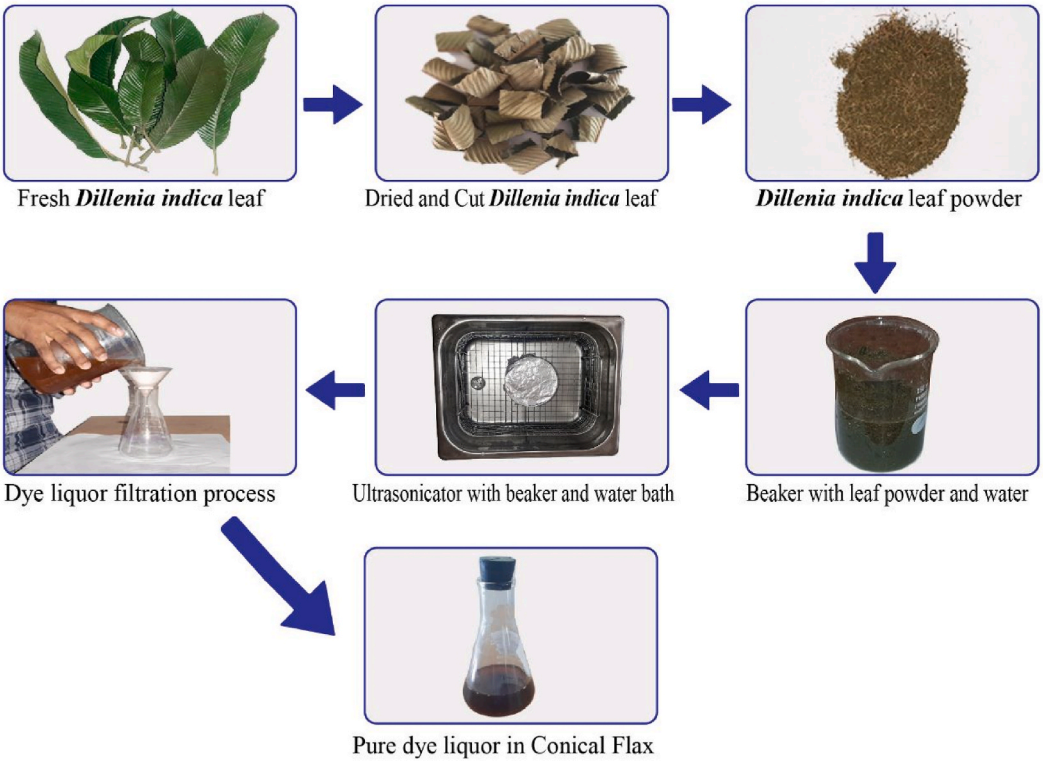


Fig. 1. The step-by-step dye extraction procedure from *Dillenia indica* leaf.

2. Experimental details

2.1. Materials

The leaves of *Dillenia indica* were collected from the garden of Mymensingh, Dhaka, Bangladesh. When compared to other plant parts such as fruits, flowers, or buds, leaves are often more common and readily available. As a result, they are a convenient and easily available source for extracting colors in higher numbers. While the fruits, flowers, or buds of *D. indica* may also contain pigments appropriate for dye extraction, the leaves used in this study were chosen based on availability, color potential, sustainability, and prior knowledge of the plant [39]. During the collection of leaves, the plants were around 18 years old, tall around 19 feet, and had no flowers. The scoured and bleached knitted organic cotton fabric with the specification of 100% organic cotton, carded yarn, yarn count 26/1 Ne, single jersey knit fabric, 160 g per square meter (GSM) was supplied by Pakiza Knit Composite Ltd., Savar, Dhaka, Bangladesh.

2.2. Methodology

2.2.1. Extraction of dye

The collected fresh *D. indica* was washed properly using normal water. Then it was dried for 15 days using the direct sunlight. It was then processed into a fine powder in a blender, vacuum-packed, and placed in a freezer set at the temperature of 4 °C. For the dye extractions, at first 10 g of dried and crushed *D. indica* powder was taken in a beaker. Then, two different amounts of water were added to the dye liquid to create the shade variations. For a dark shade, 10 g of *D. indica* powder and 150 mL of water were combined, resulting in a Material: Liquor (M:L) ratio of 1:15. The computed leaf powder percentage was 6.67%. On the other hand, 10 g of *D. indica* powder was added to 200 mL of water for a light shade, meaning that the M:L ratio was 1:20 and the computed leaf powder percentage was 5%. After that, an ultrasonic machine was used to finish the extraction process. The machine was running for 3–4 h at a temperature of 80 °C. After the extraction, filtering was first carried out using a large strainer, followed by two cycles of filtration using a filter paper. For later usage, the filtered solution was stored in a freezer at 4 °C. The step-by-step dye extraction process from *D. indica* powder is shown in Fig. 1.

2.2.2. Dyeing of organic cotton fabrics

No additional scouring or bleaching was necessary because the used organic cotton fabrics had already been cleaned and bleached. To remove impurities from the fabric surface and ensure level dyeing, only detergent washing was used to clean the dirt and dust from the fabric. The weight of the fabric sample was 3.628 g. The M:L ratio was 1:10 while the standard detergent concentration was 1.5–2 g/L. This procedure was carried out in an infrared sample dyeing machine. This treatment was taken place at a temperature of 60 °C for 10 min. Then, an ultrasonic machine was used for the dyeing process. The organic cotton fabric was dyed using the both amount of dye extraction for producing dark and light shade. The total amount of dyes liquor was 36.28 mL, because the weight of the organic cotton fabric was 3.628 g. Here, the dyeing temperature was 80 °C and dyeing period was 3–4 h. After dyeing, normal cold water was used to remove the unfixed dyes from the fabric surfaces. The dyed organic cotton fabrics were then dried using a drying machine with a temperature of 140 °C. In this dyeing method, no chemicals, including salt, soda, or mordant, was employed. The dyeing curve for the organic cotton fabrics dyeing using *D. indica* is illustrated in Fig. 2.

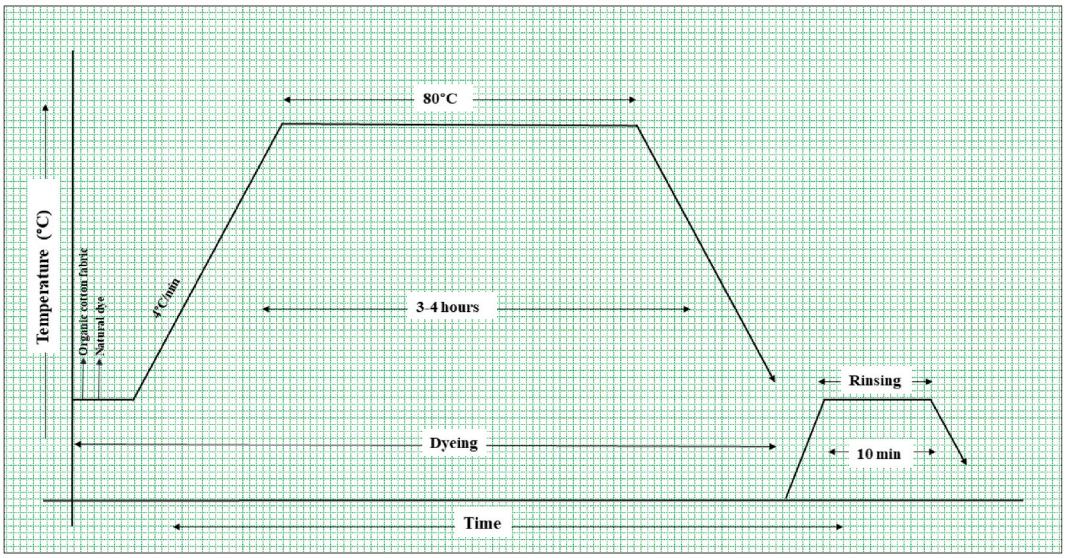


Fig. 2. A schematic diagram showing the dyeing curve of organic cotton fabrics using natural dye extracted from *Dillenia indica* leaf.

2.3. Measurement and characterizations

2.3.1. Spectrophotometric evaluation

The Data-color Spectroflash SF 650X (Texas, USA) was used to calculate the spectrophotometric value while maintaining the following settings of Illuminant D65 (refers to daylight as reference for color matching), medium area view (capturing an image that encompasses a moderate or intermediate area), specular included (presence of specular reflections in an image), and CIE 1964 (International Commission on Illumination) supplemental standard observer (10° observer). CIE 1964 is a set of color matching functions that describe the average human observer's color perception under standard viewing conditions. Each sample was folded twice to create a four-ply opaque view, and the spectrophotometric value was automatically measured. First sample, which was naturally colored, was regarded as the standard, whereas second sample was a batch sample. Using **Equation (1)** and the Kubelka-Munk theory, the K/S value was gained [40].

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

here, K/S = color strength, K = absorption coefficients, S = scattering coefficients, and R = reflectance%.

2.3.2. Color fastness analysis

The color fastness to wash of the dyed organic cotton fabric swatch was measured under ideal conditions using the ISO 105-CO3 A2S (2013) method. The fabrics underwent washing in a standard soap solution at 60 °C for 30 min, with a liquor to fabric ratio of 50:1. The dry and wet rubbing fastness of the dyed organic cotton fabrics were determined using the ISO 105 X12 (2002) method. Additionally, the light fastness was assessed using the ISO 105 B02 (2014) method [41].

2.3.3. Scanning electron microscopy (SEM) measurement

The surface morphology of both the dyed and undyed organic cotton fabric was analyzed using scanning electron microscopy (SEM) images. The SEM images were captured at 5.0 kV of accelerating voltage using the JSM-6610LV, manufactured by JEOL (Japan Electron Optics Laboratory) in Akishima, Tokyo, Japan. SEM utilizes activated light emission energy electrons to generate various signals from the surface of a solid object [42].

2.3.4. Fourier transform infrared spectroscopy (FTIR) measurement

Fourier transform infrared spectroscopy (FTIR) was conducted on samples of organic cotton fabrics, both raw and dyed, to ascertain the presence of free functional groups. FTIR spectra were measured using a Bruker Alpha and Tensor 27 FTIR spectrophotometer, with data analysis performed using OPUS software from Bruker Corporation (40 Manning Road, Billerica, MA 01821, USA). The operational parameters included a resolution of 4 cm^{-1}, 32 scans per sample, and a wavelength range of 400–4000 cm^{-1} [43].

2.3.5. Bursting strength analysis

As per the ASTM D3786/D3786M-09 standard, the bursting strength of both dyed and undyed organic cotton fabric was measured using MESDAN-LAB BURSTMATIC (Model: 338E, Brand name: MESDAN-LAB, Origin: Italy). The test outcomes were expressed in units of Ncm^{-2}. The test space used for measuring the bursting strength of the fabrics was 7.8 cm^2, and a fluid pressure of 7 bar was applied during the testing process [44].

2.3.6. Air permeability analysis

The air permeability of both dyed and undyed organic cotton fabrics was assessed using the TEXTEST air permeability tester (Model: FX-3300, Brand name: TEXTEST, Origin: Switzerland) in accordance with ASTM D737 methods. The test outcomes were reported in terms of $cm^3/cm^2/s$. During the testing process, a 20 cm^2 test area was used, and the samples' air permeability was measured at 200 pa of air pressure [45].

2.3.7. Thermogravimetric (TGA) analysis

Thermogravimetric analyses (TGA) of both dyed and undyed organic cotton fabrics were performed using a TG analyzer in a nitrogen atmosphere at a flow rate of 40 mL/min (Model-Q50, TA Instruments Inc., New Castle, DE, USA). The heat was applied at a rate of 10 °C per min, and the analysis was conducted up to a temperature of 600 °C. Each TG sample, approximately 20 mg in weight, was placed in a platinum crucible for the analysis. TG and derivative TG curves were generated to assess the weight loss and temperature relationship, allowing for the evaluation of the thermal deterioration of the organic cotton fabric samples [33].

2.4. Statistical analysis

The statistical analysis was carried out to measre the significance and variability of the obtained results. The analysis used a total of 10 replications to ensure a thorough evaluation of the experimental results. Here, the mean values are the average measurements acquired from the replications, and they indicate the normal performance of the examined samples. The standard deviations, on the other hand, quantify the variability or dispersion of the data points around the mean. The statistical analysis findings, including means and standard deviations, are shown in the corresponding tables. It is important to note that the reported values represented the mean ± standard deviation.

2.5. Instrumentations

The ultrasonic machine used in the research had a frequency higher than the audible frequency range of human hearing, exceeding 20 kHz. It was supplied by a German company named ISOLAB Laborgeräte GmbH. The manufacturer address is Am Dillhof 2, 63863 Eschau, Germany. The Whatman filter paper with 100-meshe with a size of 125 mm were purchased from the Mitali scientific store, Dhaka, Bangladesh which manufacturer was Cytiva, United states.

3. Results and discussion

3.1. Shade and color of the dyed sample

The organic cotton fabrics sample were dyed using two different concentrations of leaf powder made from *D. indica* produced both light and dark shades. Here, 5% of the leaf powder was made the light shade, and 6.67% of the leaf powder was generated a dark shade. Three samples of organic cotton fabrics are displayed in Fig. 3, where the first was a raw organic cotton fabrics sample (Fig. 3A), and the second and third were dyed light and dark organic cotton fabrics samples in Fig. 3B and C, respectively. According to the CMC analysis, the color of the dyed sample can be inferred to be a deeper reddish yellow. As a result, the color created for the light sample was honey, whereas the color produced for the dark sample was gold.

3.2. Spectrophotometric analysis

The spectrophotometric values were measured using data-color for the dyed light organic cotton fabrics and dark organic cotton fabrics under the light sources of D65, F11, and A illuminant with a 10° observer. In Table 1, the values of CMC choice including the DL*, Da*, Db*, DC*, DH*, CMC DE, and metamerism index for the dyed light and dark organic cotton fabrics were represented. According to Table 1, both dyed light and dark organic cotton fabrics were passed the CMC decision under the different illuminant. The color differences (CMC DE) of dyed light and dark organic cotton fabrics also confirmed the pass value according to the standard measurement (CMC $\Delta E \leq 1$) [46]. In addition, the metamerism index indicated there was no metamerism for both samples which were identical. The CMC results also shown the dyed samples were more daker, redder and yellower [47].

The fabric's affinity to the dye can significantly influence the color strength (K/S) of dyed textile materials. To evaluate the high color yield, the dyed organic cotton fabric swatch underwent an examination of color strength using **Equation (1)**. The color strength and reflectance values of the dyed organic cotton fabrics are presented in Table 2. According to the results, both samples exhibited good color strength. The dyed organic cotton fabrics displayed vibrant colors, indicating successful color generation using the natural dye [48]. As a result, the extracted dyes of *D. indica* have the ability to produce brighter color on organic cotton fabrics dyeing.

In comparison of the raw and dyed organic cotton fabrics color characteristics, the L*, a*, b*, c* and h* values were estimated for both samples and represented in Table 3. Here, raw organic cotton fabric was considered as the standard sample whereas dyed light and dark organic cotton fabrics were considered as the trial sample. The dyed light and dark organic cotton fabrics had a* values of 6.02 and 6.80, respectively, indicating that the dark organic cotton fabric was more red. Dark sample was more yellow than light sample, as shown by the b* values of 15.15 for the light organic cotton fabrics and 18.19 for the dark organic cotton fabrics, respectively. The chroma, c* values of 16.30 and 19.42 for the light and dark organic cotton fabrics, respectively, shown that the purity or saturation of the dark sample was higher than that of the light sample. Additionally, the hue h* for the light and dark organic cotton fabrics were 68.32 and 69.51, respectively, indicating that the shade or tone was nearly same for both dyed organic cotton fabrics. Lightness L* values for the two samples were 72.14 and 69.10, respectively, indicating that the dyed dark organic cotton fabric was darker than the light organic cotton fabrics. This justifies our prediction that the colored sample was darker reddish yellow. The same phenomena were discovered when the wool fabric was dyed using the extraction of *Rhizoma coptidis* [49].

3.3. Analysis of color fastness

Color fastness to washing, perspiration, light, and rubbing was assessed on the dyed organic cotton fabrics, and the findings are summarized in Table 4. The dye diffusion rate and the dye's condition within the fiber played a crucial role in determining the natural

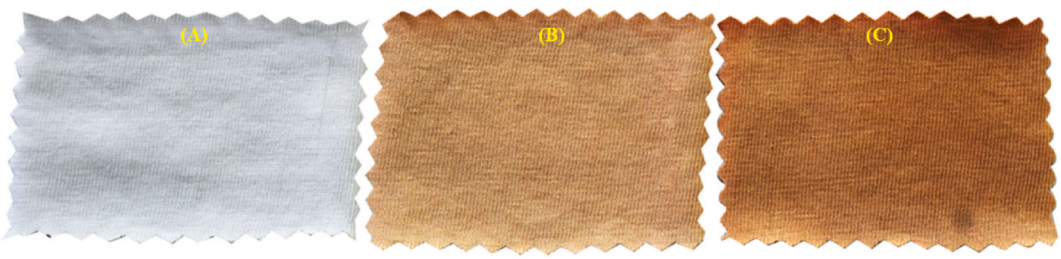


Fig. 3. (A) Raw (B) Dyed light shade, (C) Dyed dark shade of organic cotton fabric.

Table 1
CMC decision and metamerism index for dyed organic cotton fabrics.

Sample type	Illuminant/Observer	CMC Decision	CMC DE	DL*	Da*	Db*	DC*	DH*	Metamerism Index
Dyed (light) organic cotton fabric	D65/10°	Pass	0.35 ± 0.04	−0.46	0.07	0.29	0.25	0.15	
	F11/10°	Pass	0.42 ± 0.03	−0.45	0.06	0.35	0.30	0.19	0.07 ± 0.01
	A/10°	Pass	0.31 ± 0.01	−0.43	0.14	0.30	0.32	0.10	0.09 ± 0.01
Dyed (dark) organic cotton fabric	D65/10°	Pass	0.44 ± 0.04	−0.60	0.16	0.38	0.39	0.14	
	F11/10°	Pass	0.52 ± 0.04	−0.59	0.16	0.46	0.45	0.18	0.08 ± 0.01
	A/10°	Pass	0.43 ± 0.02	−0.56	0.23	0.44	0.48	0.12	0.12 ± 0.01

Here, CMC Decision = decision from color measurement committee, CMC DE = color differences, DL* = lightness differences, Da* = red/green differences, Db* = blue/yellow differences, DC* = chroma differences, DH* = hue differences, and Metamerism Index = the color difference between samples under different lighting conditions.

Table 2
Reflectance and color strength value of the dyed organic cotton fabrics.

Sample type	Wavelength	400–450 nm	460–510 nm	520–570 nm	580–630 nm	640–690 nm	700 nm
Dyed (light) organic cotton fabric	**Reflectance, R**	40.00	40.01	49.59	61.46	70.45	71.25
	R%	.40	.4001	.4959	.6146	.7045	.7125
	Color strength, K/S	19.01	19.02	23.80	29.73	34.23	34.63
Dyed (dark) organic cotton fabric	**Reflectance, R**	39.09	39.39	47.81	58.28	70.45	71.25
	R%	.3909	.3939	.4781	.5828	.7045	.7125
	Color strength, K/S	18.55	18.70	22.91	28.14	34.23	34.63

Table 3
Color characteristics value of the raw and dyed organic cotton fabrics.

Sample type	L*	a*	b*	c*	h*
Raw organic cotton fabric	83.75	−1.00	−1.99	2.22	243.43
Dyed (light) organic cotton fabric	72.14	6.02	15.15	16.30	68.32
Dyed (dark) organic cotton fabric	69.10	6.80	18.19	19.42	69.51

Here, L* = lighter/darker; a* = redder/greener; b* = yellower/bluish; c* = chromaticity; h* = hue saturation.

dye's wash fastness property [50]. For washing fastness of the both samples, the color change and staining rating were satisfactory, resulting in an excellent fastness property. This was because the ultrasonic dyeing methods ensured the more interactions between the dyes and fabric instead of a traditional natural dyeing method. A similar phenomena was observed when using mehedy for the coloration of cotton fabric to create a covalent or durable link between them [51]. Regarding rubbing fastness, the rating of wet color change was observed to be lower than the dry color change rating, resulting in a good rating for the fabric. This difference in ratings could be attributed to the presence of water-soluble dye groups that make it relaxed for them to separate from the fabric during wet rubbing, causing the variation in rubbing fastness [48]. The situation where wet rub obtained the lowest grade may result in some surface coloring. According to Table 4, the organic cotton fabrics colored with *D. indica* had a light fastness rating of 4–5 when a light sample was used. In contrast, the organic cotton fabrics dyed with *D. indica* had a light fastness rating of 4 for the dark sample. As a result, both the light and dark dyed organic cotton fabrics sample ratings were excellent. This outcome demonstrated that the dark sample performed worse than the light sample. It might demonstrate the natural dyestuff produced from *D. indica* was light sensitivity [52]. Indeed, the availability of ionizable groups in natural coloring agents, for example –OH and CO_2H, may provide an explanation for the observed phenomenon. Due to the reduction in anionic environments, these –OH groups become more liquefiable in aqueous solutions [53]. Consequently, the fastness attributes of the dyed organic cotton fabrics endured within the acceptable limit, despite being colored using dyes derived from natural sources like *D. indica*.

3.4. SEM analysis

The morphological changes at the surface of the dyed organic cotton fabrics using *D. indica* natural dyes were studied using the SEM images. The images provided more evidence that *D. indica* dyes were successfully absorbed by the surface of organic cotton fabrics. In Fig. 4, the morphological alterations of organic cotton fabrics brought on by the ultrasonic method of *D. indica* dyeing were examined. The photograph provided illustrates noticeable differences between the raw and dyed organic cotton fabric samples. In Fig. 4(A_1–A_3), the surface properties of the raw organic cotton samples appear to be much smoother and refined compared to the dyed fabric samples. Fig. 4B and C depict the unevenness of the morphology at the microscale, which is a result of the existence of the dye-fabric conjugate in the dyed organic cotton fabrics. This roughness is likely caused by the interaction between the organic cotton fabrics and the dye molecules during the dyeing process [54]. The morphography analysis further demonstrated that there was no dye accumulation observed around the organic cotton fabrics. This outcome indicated the successful dyeing of organic cotton fabrics using *D. indica* extract. The absence of dye accumulation confirmed that the dye was effectively absorbed and distributed throughout the fabric,

Table 4
Color fastness to rubbing, perspiration, wash, and light of dyed organic cotton fabrics.

Sample type	Rubbing Fastness		Perspiration Fastness				Wash Fastness							Light Fastness
			Alkaline		Acid		Color change	Color staining						
	Dry	Wet	Color change	Color staining	Color change	Color staining		Wool	Acrylic	Polyester	Nylon	Cotton	Acetate	
Dyed (light) organic cotton fabric	4-5	4	4	3-4	4-5	3-4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Dyed (dark) organic cotton fabric	4-5	3	4-5	4	4	3-4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4

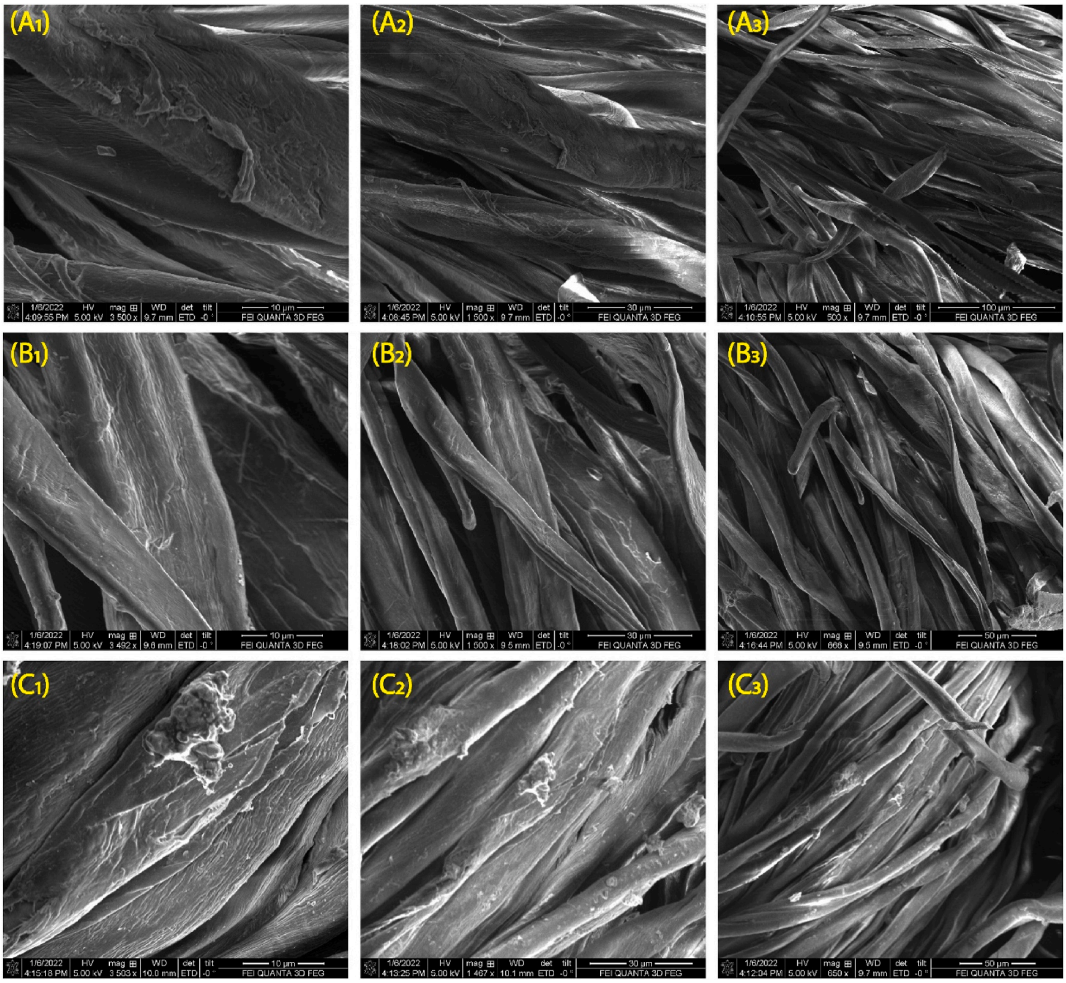


Fig. 4. SEM images of (A_1–A_3) Raw (B_1–B_3) Dyed light shade, and (C_1–C_3) Dyed dark shade of organic cotton fabrics.

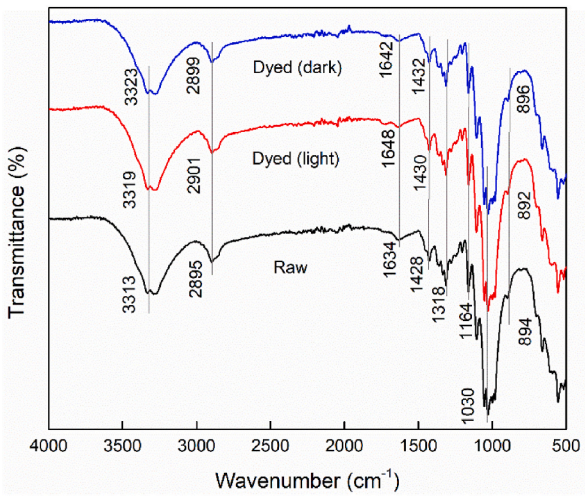


Fig. 5. FTIR Spectra of undyed, dyed (light), and dyed (dark) organic cotton fabrics.

resulting in a successful and uniform dyeing process.

3.5. FTIR analysis

Fourier transform infrared spectroscopy (FT-IR) with attenuated total reflectance (ATR) mode and a resolution of 4 cm^{-1} were utilized to analyze the functional groups on the surface of organic cotton fabrics. Fig. 5 displays the FTIR spectra of both raw and dyed organic cotton fabric samples. In the raw sample, characteristic peaks were observed at 3313 cm^{-1}, indicating the stretching vibrations of the –OH atom in cellulose, and at 2895 cm^{-1}, representing the asymmetric stretching of the –CH aliphatic group. The peak at 1634 cm^{-1} was attributed to the C=C of the aromatic ring, while the peaks at 1428 cm^{-1} and 1318 cm^{-1} indicated the presence of CH_2 groups of cellulose. The C–OH groups of secondary and primary alcohols appeared at 1164 cm^{-1} and 1030 cm^{-1}, respectively, and the C–O–C bond of β-(1,4) glycosidic linkages were identified at 894 cm^{-1} [55]. Upon dyeing the organic cotton fabric with the *D. indica* extract, slight changes in the spectra were observed for both light and dark samples. New peaks appeared at 892 cm^{-1} and 896 cm^{-1}, corresponding to the bending vibration of the heterocyclic cyclic aromatic ring of =C–H. However, the peak intensity of the dyed organic cotton fabric was lower, indicating a chemical interaction between the dye and the fabric. These observed peaks in the dyed organic cotton fabrics using *D. indica* were consistent with previous research [56].

3.6. TGA analysis

Over the past few years, cellulose thermal breakdown has been the subjected to extensive research. It was noted that cellulose's composition and source had a significant impact on how it pyrolyzes. Thermogravimetric (TG) analysis is one of the most often used analytical methods for the investigation of cellulose pyrolysis. According to Fig. 6A, the thermal degradation of the organic cotton fabric can be categorized into three main stages: (i) Moisture and chemical bond water evaporation: This stage occurs at temperatures between 50 °C and 150 °C, during which moisture and chemical bond water are released from the fabric. (ii) Cellulose degradation: This stage takes place between 150 °C and 400 °C and involves the breakdown of cellulose present in the fabric. (iii) Pyrolysis of cellulose: Occurring at temperatures between 300 °C and 400 °C, this stage involves depolymerization, molecular chain breaks, and dehydration of glyco-groups within the cellulose. The lowermost weight loss was observed during the first thermal occurrence at 50–150 °C, indicating the decrease of hydroxyl groups from both the raw and dyed organic cotton fabrics. This decrease in weight corresponds to the evaporation of chemical bond water and moisture during this stage of thermal degradation. The weight loss at 250–400 °C for raw, dyed light and dyed dark organic cotton fabrics were 77.74%, 81.94%, and 80.64%, respectively that indicate that weight loss of dyed fabric was higher than raw organic cotton fabrics at this temperature range. The char residues% for the three samples were 9.73%, 12.95%, and 14.12%, respectively, shown that the dyed sample had a higher char residue% than the raw organic cotton fabrics.

On the other hand, in Fig. 6B, the cellulose breakdown was occurred at a temperature of 370 °C, which resulted in the supreme weight loss at the DTG curve of 81.94%. The maximum peak at the DTG curve for the raw and dyed samples was 363 °C, 370 °C, and 367 °C, respectively, which denotes a trend toward higher temperatures and suggested that the dyed fabric had a higher thermal stability. The summary of the results is presented in Table 5, indicating that the dyed organic cotton fabrics exhibited good thermal stability and even demonstrated greater stability compared to the undyed fabrics. These findings are consistent with a previous report, which confirmed the accuracy and acceptability of the outcomes [57].

3.7. Analysis of bursting strength

Fabric bursting strength refers to the force or pressure required to break the fabric when it is subjected to a vertical force or

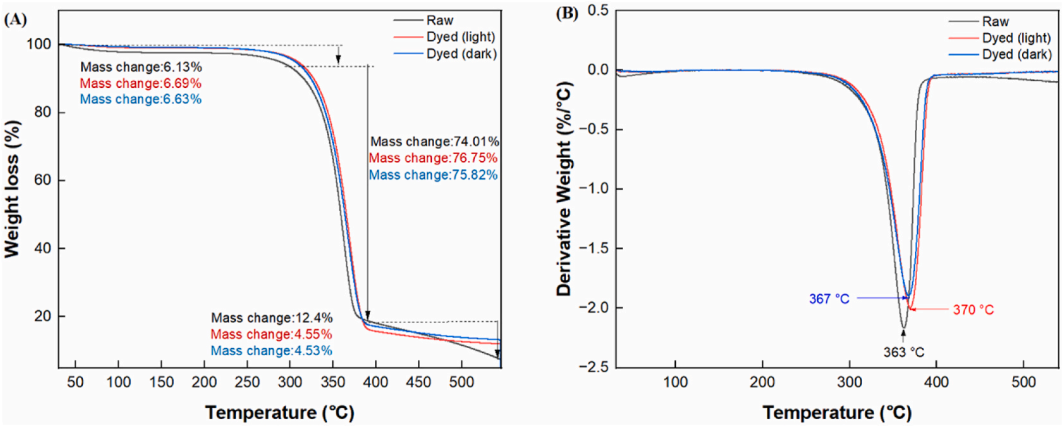


Fig. 6. (A) Thermogravimetric analyses curves and (B) Differential thermogravimetry curves of raw and dyed organic cotton fabrics.

Table 5
Weight loss % at 250–400 °C, char residues% and maximum peak at differential thermogravimetry of raw and dyed organic cotton fabrics.

Sample type	Weight loss % at 250–400 °C	Char residues %	Maximum peak at differential thermogravimetry
Raw organic cotton fabric	77.74	9.73	363 °C
Dyed (light) organic cotton fabric	81.94	12.95	370 °C
Dyed (dark) organic cotton fabric	80.64	14.12	367 °C

pressure. This test measures the ability of the fabric to withstand the pressure without tearing or bursting. The bursting strength is an essential parameter in evaluating the strength and durability of fabrics, particularly in applications where the fabric is subjected to mechanical stresses or pressure [58]. The bursting strength of the raw and dyed organic cotton fabrics is shown in Table 6. Here, the bursting strength of the raw, dyed light and dyed dark organic cotton fabrics were 173.1 ± 2.10 KPa, 157.2 ± 1.56 KPa, and 152.5 ± 1.83 KPa, respectively. The results indicated that the dyed organic cotton fabric had a lower bursting strength compared to the raw organic cotton fabric. Additionally, it was observed that the dark organic cotton fabric samples had weaker bursts than the light organic cotton fabric samples. These findings suggest that the dyeing process and the darkness of the color may have influenced the mechanical strength and bursting resistance of the organic cotton fabrics. The reduction in bursting strength in dyed fabrics and the differences between dark and light samples could be attributed to the chemical interactions between the dye and fabric, altering the fabric's structural integrity and mechanical properties. That was brought about by a successful ultrasonic dyeing procedure. With an ultrasonic dyeing technology, the fabric was colored at a high temperature while also vibrating. However, the bursting strength was satisfactory and appropriate for the dyed fabric sample's application purposes [59].

3.8. Analysis of air permeability

The air permeability of a fabric is defined as the capacity of air in milliliters that permits through a 100 mm^2 area of the fabric in 1 s, under a pressure difference of 10 mm of water [60]. Table 6 presents the air permeability amount of both the raw and dyed organic cotton fabrics. Here, the air permeability of the raw, dyed light and dyed dark organic cotton fabrics were 86 ± 1.04 $cm^3/cm^2/s$, 87 ± 0.72 $cm^3/cm^2/s$, and 88 ± 1.32 $cm^3/cm^2/s$, respectively. Here, the dyed organic cotton fabrics sample had slightly higher air permeability than the raw organic cotton fabrics. Additionally, the dyed dark organic cotton fabrics had higher air permeability than the dyed light organic cotton fabric samples. This was because in dyeing, the fabric sample's pores became more visible than those of an undyed or raw fabric sample. Therefore, the air permeability results of dyed organic cotton fabrics were in the acceptable range according to the previous studies [61].

4. Conclusion

The novelty of this research was in the eco-friendly ultrasonic assisted extraction and organic cotton fabric dyeing using *Dillenia indica* leaf. Here, the ultrasonic extraction method was used which was shown better extraction and coloring properties. The characteristics of the dyed organic cotton fabrics show that it performed better during the dyeing process. The dyed organic cotton fabrics exhibit a reddish yellow tone or hue, according to the spectrophotometric results. The SEM images of the sample of colored organic cotton fabrics show a rough and dry surface that suggests the attendance of dye particles. The FTIR data also shown the presence of the distinctive peaks of the raw and dyed organic cotton fabrics, demonstrating the interactions between the organic cotton fabrics and *D. indica* extract colors. For a dyed organic cotton fabric, the bursting strengths of 152.5 ± 1.83 KPa and 157.2 ± 1.56 KPa were sufficient, and the air permeability of 87 ± 0.72 $cm^3/cm^2/s$, and 88 ± 1.32 $cm^3/cm^2/s$ were in the acceptable range. As a result, concerning the environmental issues, using naturally derived dyes from *D. indica* to color organic cotton fabrics using ultrasonic technology may be a sustainable substitute to synthetic dyes in the textile dying industry.

Author contribution statement

Burhan Uddin Banna: Performed the experiments; Wrote the paper. **Rony Mia:** Wrote the paper; Analyzed and interpreted the data. **Md. Mahabub Hasan:** Contributed reagents, materials, analysis tools or data. **Bulbul Ahmed:** Analyzed and interpreted the data. **Mohammad Abul Hasan Shibly:** Conceived and designed the experiments; Contributed reagents, materials, analysis tools or data.

Data availability statement

Data included in article/supplementary material/referenced in article.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Table 6
Bursting strength and air permeability of raw and dyed organic cotton fabrics.

Sample type	Bursting strength values KPa	Air permeability values $cm^3/cm^2/s$
Raw organic cotton fabric	173.1 ± 2.10	86 ± 1.04
Dyed (light) organic cotton fabric	157.2 ± 1.56	87 ± 0.72
Dyed (dark) organic cotton fabric	152.5 ± 1.83	88 ± 1.32

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