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EXTRACTION OF CHLOROPHYLL FROM PANDAN WANGI LEAVES (Pandanus amaryllifolius Roxb.) AS NATURAL TEXTILE DYES

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Abstract: This research focuses on the utilization of natural materials such as textile dyes, which are environmentally friendly and safe for the health of living things. The natural ingredients used are pandan wangi leaves (*Pandanus amaryllifolius Roxb*.), especially chlorophyll compounds, which are green pigments. The types of fabrics tested consisted of cotton fabrics, linen fabrics, and drill fabrics. The purpose of this study was to extract chlorophyll from pandan wangi leaves via the maceration extraction method with ethanol and maceration for 36 hours. The particle size of the pandan wangi leaves was made up to 60 mesh to obtain the optimum extraction yield. Fabric coloring begins by washing the fabric with Turkish Red Oil (TRO) solution and then mordanting it with various concentrations of 1% and 0.6% alum mordant. Fabrics that have been dyed are then tested for color fastness to sunlight and rubbing. The results of maceration of the ethanol extract produced a yield value of 40.22%. The chlorophyll a and b contents were calculated from the absorbance values obtained via ultraviolet–visible (UV–Vis) spectroscopy, namely, 3.7223 μg/mL and 1.1362 μg/mL, respectively. Fourier transform infrared (FTIR) characterization revealed the presence of methyl, ketone, amine, and ester functional groups. The values of the test results for color fastness to sunlight and rubbing were 4 (good) and 4–5 (very good), respectively.

Keywords: Chlorophyll, Color, Fabric cotton, Maceration, Pandan Wangi leaves

Abstrak: Penelitian ini berfokus pada pemanfaatan bahan alami seperti pewarna tekstil yang ramah lingkungan dan aman bagi kesehatan makhluk hidup. Bahan alami yang digunakan adalah daun pandan wangi (*Pandanus amaryllifolius Roxb*.), khususnya senyawa klorofil sebagai pigmen hijau. Jenis kain yang diuji terdiri dari kain katun, kain linen, dan kain bor. Tujuan penelitian ini adalah mengekstraksi klorofil dari daun pandan wangi menggunakan metode ekstraksi maserasi dengan etanol p.a. dan kontrol waktu maserasi selama 36 jam. Ukuran partikel daun pandan wangi dibuat hingga 60 mesh untuk mendapatkan rendemen

ekstrak yang optimal. Pewarnaan kain diawali Degnan pencucian kain menggunakan larutan Turkish Red Oil (TRO) kemudian dilakukan mordanting dengan variasi konsentrasi mordan tawas 1% dan 0,6%. Kain yang telah diwarnai kemudian diuji tahan luntur warnanya terhadap sinar matahari dan gesekan. Hasil maserasi ekstrak etanol menghasilkan nilai rendemen sebesar 40,22%. Kandungan klorofil a dan b dihitung dari nilai serapan yang diperoleh dengan menggunakan metode Ultraviolet–Visible (UV–Vis) yaitu masing-masing sebesar 3,7223 µg/mL dan 1,1362 µg/mL. Karakterisasi Fourier Transform InfraRed (FTIR) mendeteksi keberadaan gugus fungsi metil, keton, amina, dan ester. Nilai hasil pengujian tahan luntur warna terhadap sinar matahari dan gesekan masing-masing sebesar 4 (baik) dan 4–5 (sangat baik).

Kata kunci: Daun Pandan Wangi (*Pandanus amaryllifolius Roxb)*, Kain katun, Klorofil, Zat warna alami

INTRODUCTION

In the textile industry, the dyes commonly used are synthetic because they are more stable to light and oxidation conditions, provide brighter color intensity, require less color, and have a variety of colors (Shamanta 2020; Rachmawati et al 2020). However, the continuous use of synthetic dyes can pollute the environment, especially wastewater, which is difficult to degrade and can harm the health of living creatures because of the presence of heavy metals such as copper, nickel, chromium, mercury, and cobalt (A. Rosyida and A. Zulfiya 2013; Faisal and A. Chafidz 2019). Thus, natural dyes can be used as alternative dyes in the textile industry by taking several parts of plants that have colored pigments, such as bark, stems, leaves, and twigs (Chintya and B. Utami 2017). One plant that can be used as a natural dye is pandan wangi leaves (Pandanus amaryllifolius Roxb.). Supported by the commodity of pandan wangi leaves, which are widely distributed in Indonesia, the use of pandan wangi leaves as a natural coloring agent can be easily optimized.

The dye obtained from pandan wangi leaves is chlorophyll, which produces a green color. Chlorophyll is sensitive to temperature, oxygen, and chemical degradation processes (Emaini et al 2012). Therefore, taking chlorophyll pigments from pandan wangi leaves must be carried out in dark conditions and does not involve a heating process to avoid changes in the characteristics of chlorophyll. The extraction method that can be used to extract chlorophyll pigments is the extraction method. Factors that can influence the extraction process include time, temperature, type of solvent, ratio of sample to solvent, and particle size (Chairunnisa et al 2019). As in previous

studies that provided different treatments for maceration time and particle size, the best extraction results were obtained with a maceration time of 36 hours and a particle size of 60 mesh, with a yield value of 8.81%, a chlorophyll a content of 23.66%, and a chlorophyll b content of 3.92% (Sayoga et al 2020). Therefore, in this research, samples were sifted to a size of 60 mesh, and a maceration time of 36 hours was used to provide stable and optimal results.

The natural dyes used in textile fabrics cannot bond for a very long time; as a result, the color absorbed decreases. Therefore, a substance is needed that can connect the fabric with the natural dye so that it remains bonded, which is called a mordant. The mordant used in this research was alum or aluminum sulfate because it is considered a safe mordant for use in the application of natural dyes to textiles.

The application of chlorophyll dye from pandan wangi leaves to textile fabrics has not been extensively studied. This encouraged the author to dye chlorophyll dye on 3 different types of fabric, namely, cotton fabric, linen fabric, and drill fabric. The choice of fabric is based on the number of Indonesian people who wear clothes made from these fabrics..

METHOD

Preparation of Sample Powder

Pandan Wangi leaves obtained from the yard of the author's house and neighbors located in Pondok Cabe Ilir, Pamulang, South Tangerang, Banten, were cleaned of dirt using water and then cut into 30 cm lengths. Blanching was carried out using water at a temperature of 75–80°C for ± 1 minute. The samples of pandan wangi leaves were cut into small pieces of up to 1 cm in size and then dried in an oven at 60°C for 14 hours. The dried samples were ground using a blender until they became powder and then sieved through a 60-mesh sieve.

Sample Maceration

Maceration was carried out by soaking the sample powder in ethanol (1:7) for 36 hours at room temperature without being exposed to light by lining the maceration container with aluminum foil. During the maceration process, every 12 h, the resulting filtrate was removed, and the ethanol solvent was replaced. The filtrate was mixed and then filtered through Whatman No. 1 filter paper and then evaporated via a Heidolph Hei-VAP rotary evaporator at 55°C and 40 rpm. The resulting thick extract is put into a sample bottle, the mass is recorded, and the

percentage yield of the extract is calculated.

Fabric Treatment

The fabric was washed first by cutting the cotton fabric and linen fabric and then drilled to a size of 1x1 meter. The TRO solution used as a fabric washing solution is made by dissolving 10 grams of TRO powder in 2 liters of water to wash 1 piece of fabric. The three types of fabric were soaked in TRO solution for 12 hours. The fabric was rinsed with water and then dried in the sun to dryness at room temperature.

The mordant used in the premordanting process is alum. Alum was made into two solutions: 1% and 0.6% alum solutions. The alum powder was mixed with water and then heated on a stove until it boiled. When boiling, the fabric was placed in the mordant solution and left for 45 minutes under heating. After that, the fabric was removed, rinsed with water, and dried at room temperature. The natural coloring agent from the pandan wangi leaf extract was made by mixing 10 mL of extract solution with 1 L of water. The fabric that has been given the mordant is soaked in the dye solution for 1 hour (Ohama and N. Tumpat 2014). After that, the fabric was dried at room temperature. The coloring process was carried out in triplicate.

Fabric Testing

Fabric color fastness testing consists of two parameters, a color fastness test for sunlight and a color fastness test for rubbing (wet and dry), which is carried out at the Laboratorium Balai Besar Kerajinan dan Batik (BBKB), Yogyakarta Special Region. This test follows the provisions of SNI ISO 105-B 01-2010 and SNI ISO 105-X12:2016. The color of the cotton fabric, linen fabric, and drill fabric was measured via a JASCO V-750 UV–Vis spectrophotometer to determine the brightness value (L*), redness value (a*), and yellowish value (b*).

Thin Layer Chromatography (TLC)

The mobile phase used was hexane:acetone (7:3), and the stationary phase used was a silica gel plate. The sample extract solution was spotted onto a spot on a 5x1 cm silica gel plate using a capillary tube until the spot color became dark. The silica gel plate was inserted into the chamber and then left until the mobile phase rose to the endpoint. The color of each spot that appeared on the silica gel plate was observed, and the Rf value was calculated.

Sample characterization

Characterization was carried out via FTIR with the KBr pellet method to identify the functional groups that appeared. FTIR instrumentation using the attenuated total reflectance (ATR) method was used to identify functional groups in the fabric that had been treated with chlorophyll dye. Characterization via UV–Vis Genesys 10 s was carried out to determine the absorbance value and calculate the chlorophyll content value obtained from the Lichtenthaler equation (1987).

Chlorophyll a = $13.36\ A_{664} - 5.19\ A_{649}$ (1)

Chlorophyll b = $27.43\ A_{649} - 8.12\ A_{664}$ (2)

RESULTS AND DISCUSSION

The ethanol extract obtained from pandan wangi leaves was a dark green color, as shown in Figure 1. The ethanol extract from pandan wangi leaves had a yield percentage value of 40.22%. This result is in line with the statement of Sayoga et al. (2020) that a smaller sample size can produce an increasing yield value. In addition, the choice of ethanol, i.e., the solvent, which is polar and has high selectivity, as well as the addition of the blanching method to the sample preparation process, can increase the degree of interaction between the sample and the solvent. The image below (Figure 1) is the result of extracting chlorophyll from pandan wangi leaves using ethanol as a solvent.

To determine what chlorophyll compounds are contained in Pandan Wangi, thin layer chromatography analysis was carried out using mobile phase hexane:acetone (9:1). Eluent selection is based on the chlorophyll compound, which has semipolar (chlorophyll b) and nonpolar (chlorophyll a) properties. Thus, a solvent that is similar to the compound being analyzed, nonpolar hexane and acetone, which are semipolar to attract the color pigments contained therein, is needed. The Rf values and resulting stain spots are listed in Figure 2 and Table 1.

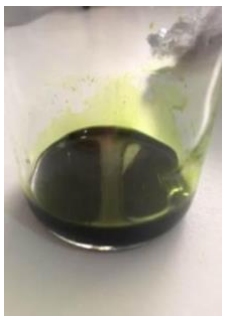


Figure 1. Ethanol extract solution from chlorophyll

Figure 2. TLC analysis of the ethanol extract of Pandan Wangi leaves

Table 1. Rf values, stain color, and type of pigment contained in the ethanol extract of pandan wangi leaves.

Spot	Rf	Color	Pigment Type
1	0.50	Light yellow	Xanthophyll
2	0.51	Yellowish green	Chlorophyll b
3	0.55	Bluish green	Chlorophyll a
4	0.95	Dark yellow	β-carotene

The Rf values obtained in the literature indicate that the most nonpolar component will move faster. As a result, this component has the highest Rf value, namely, a β-carotene pigment of 0.95. This is followed by increasingly polar components, namely, chlorophyll a, chlorophyll b, and xanthophyll. The resulting stain consists of two green spots (chlorophyll a and chlorophyll b) and several yellow spots that are partially covered by green spots (xanthophyll and β-carotene) (Sjursnes et al 2015).

The absorbance of the chlorophyll extract was determined via a UV–Vis instrument at wavelengths ranging from 380–700 nm. This is because chlorophyll absorbs red and blue light in the range of 400–700 nm (Bayang et al 2020). These two rays are in the white or polychromatic light spectrum, which has a wavelength between 380 and 760 nm (Arifah et al 2019) (Figure 3).

As shown in Figure 3, the pandan wangi leaf extract presented absorbance peaks at wavelengths of 413 nm and 666 nm. Therefore, the pigment identified based on the UV–Vis test results may be a chlorophyll a pigment. Chlorophyll concentrations are determined via calculations using the Lichtenthaler equation, which yields a chlorophyll a value of 3.7223 µg/mL and a chlorophyll b value of 1.1362 µg/mL. To determine the functional groups contained in the chlorophyll compounds contained in Pandan Wangi, FTIR analysis was carried out (Figure 4).

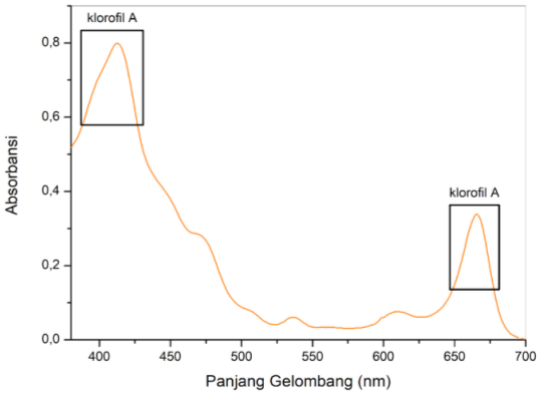


Figure 3. Characterization of chlorophyll extract using UV-Vis

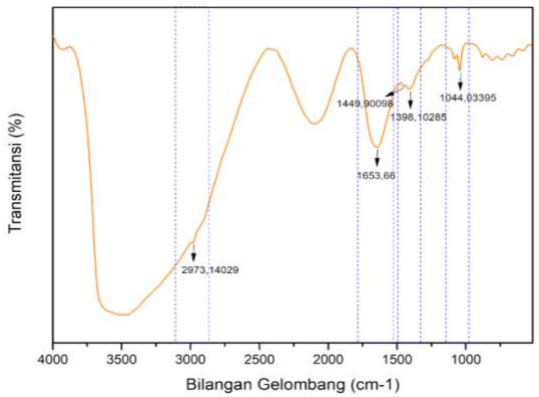


Figure 4. Characterization of Chlorophyll from the Extract of Ethanol via FTIR KBr

Based on the FTIR characterization data above, the peak at 2973 cm-1 indicates the presence of stretched C–H bonds in the methyl group, CH3. The C=O stretching bond vibrations in ketones appear at 1653 cm-1. The peak at 1449 cm-1 corresponds to the bending C-H (sp2) bond vibration of the methylene (CH-) group. The absorption peak at 1398 cm-1 corresponds to the vibration of the C–N bond attached to the tetrapyrrole ring (Aryanti et al 2016). At 1044 cm-1, stretching C–O bond vibrations appear. As explained by Hutajulu et al. 2008. In their research, chlorophyll, which has a methyl group (CH3), presented absorption peaks at 2940 cm-1, 1455 cm-1, and 1380 cm-1. Moreover, chlorophyll b, which has an aldehyde group (-CHO), appeared at 2750 cm-1, 2680 cm-1, and 1650 cm-1. Therefore, in this study, the type of chlorophyll pigment detected by the FTIR instrument was chlorophyll, a pigment that has a structure similar to that shown in Figure 5.

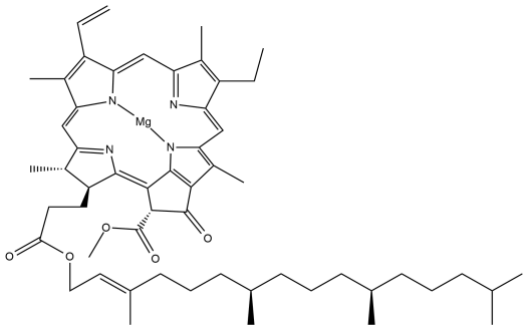
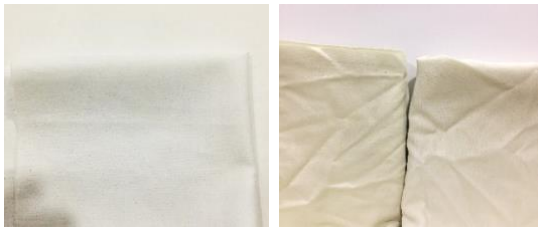


Figure 5. Chlorophyll compound structure a

Based on the dyeing results for the three types of fabric, the use of alum as a mordant gives a bright yellow to greenish-yellow color (Figure 6-8). Compared with the fabric treated with 0.6% alum, the fabric treated with 1% alum produced a deeper color. In linen fabric, the resulting color is bright yellow with the addition of 0.6% alum and dark yellow with the addition of 1% alum. The color of the cotton fabric and drilled fabric was greenish yellow with the addition of 0.6% alum, and with the addition of 1% alum, the color was deeper greenish yellow. The color given by the chlorophyll solution is not the same for each different type of fabric. This finding shows that the color produced from the dyeing process using mordant and natural dyes depends on the basic color of the type of fabric itself.

(a) (b)

Figure 6. Linen fabric (a) before dyeing and (b) after dyeing (left: alum 1%, right: alum 0.6%)



(a) (b)

Figure 7. Cotton fabric (a) before dyeing and (b) after dyeing (left: alum 1%, right: alum 0.6%)

(a) (b)

Figure 8. Drill fabric (a) before dyeing and (b) after dyeing (left: alum 1%, right: alum 0.6%)

Table 2. Color measurement values for cotton fabrics, linen fabrics, and drill fabrics with various alum mordant concentrations

Sample Code	Different Color Values		
	L*	a*	b*
KK(A)[a]	62.98	-5.82	18
KK(B)[b]	64.57	-5.32	17.32
KL(A)[c]	57.15	-5.67	20.51
KL(B)[d]	64.67	-5.7	18.72
KD(A)[e]	56.28	-4.64	16.54
KD(B)[f]	67.42	-4.65	14.97

[a]Cotton fabric with 1% alum; [b]Cotton fabric with 0.6% alum; [c]Linen fabric with 1% alum; [d]Linen fabric with 0.6% alum; [e]Drill fabric with 1% alum; [f]Drill fabric with 0.6% alum

Based on the data in Table 2, the L* value is positive, which indicates that the measured fabric color has a higher brightness level than the original color. The a* value in this study is negative, which indicates that the color of the fabric measured is green. The resulting b* value is positive, which indicates that the color of the fabric being measured is yellow. Alum acts as a chelating compound that binds dye molecules together to form large complex compounds and connects the dye with the fabric (Haar et al 2013). The aluminum ion (Al3+) in alum forms a coordinated complex with the dye during the fabric dyeing process. Cotton fabrics can bond with natural dyes via hydrogen bonds formed through their hydroxyl groups and covalent bonds formed between semiquinone groups in the dye and cellulose (Ragheb et al 2017). The interactions that occur among the cellulose compounds in the fabric, alum mordant, and chlorophyll dye are shown in Figure 9.

Fabrics that have gone through the dyeing process are tested for color fastness to sunlight and rubbing. Based on the results of testing for fastness to sunlight, the average value given was 4, which indicates a good value for the three types of fabric in the mordanting treatment when alum concentrations of 1% and 0.6% were used. In testing the color fastness to rubbing, the average value of color staining both wet and dry was obtained at 4–5, which indicates a very good value for the three types of fabric in the mordanting treatment using alum concentrations of 1% and 0.6% alum. The color fastness test results for the cotton fabric, linen fabric, and drilled fabric are shown in Table 3.

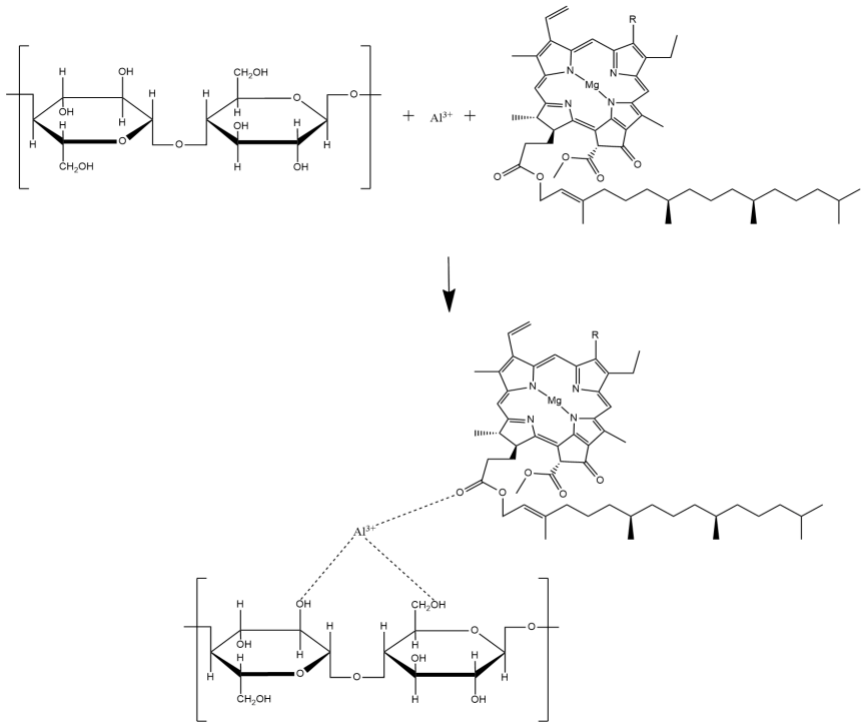
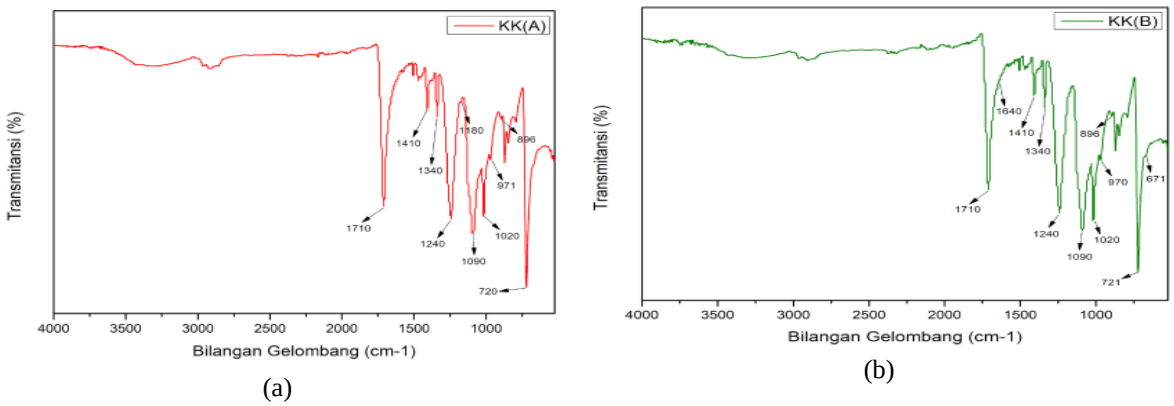


Figure 9. Interactions Between Cellulose Fibers in Fabric, Alum Mordant, and Chlorophyll Dye

Table 3. Fabric color fastness test results

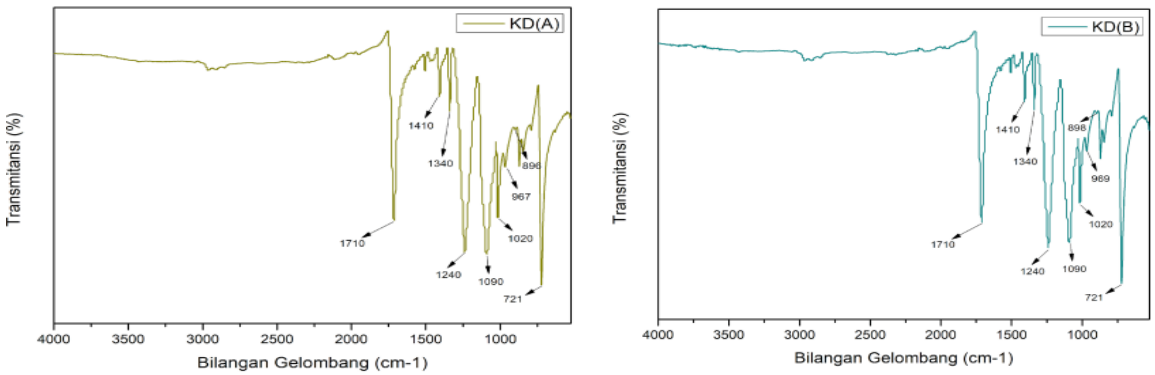
Sample Code	Color Fastness to Light: Daylight	Color Fastness to Rubbing	
		Color Decolorization Value (Wet)	Color Stains Value (Dry)
KK(A)	4	4-5	4-5
KK(B)	4	4-5	4-5
KL(A)	4	4-5	4-5
KL(B)	4	4-5	4-5
KD(A)	4	4-5	4-5
KD(B)	4	4-5	4-5



(a) (b)

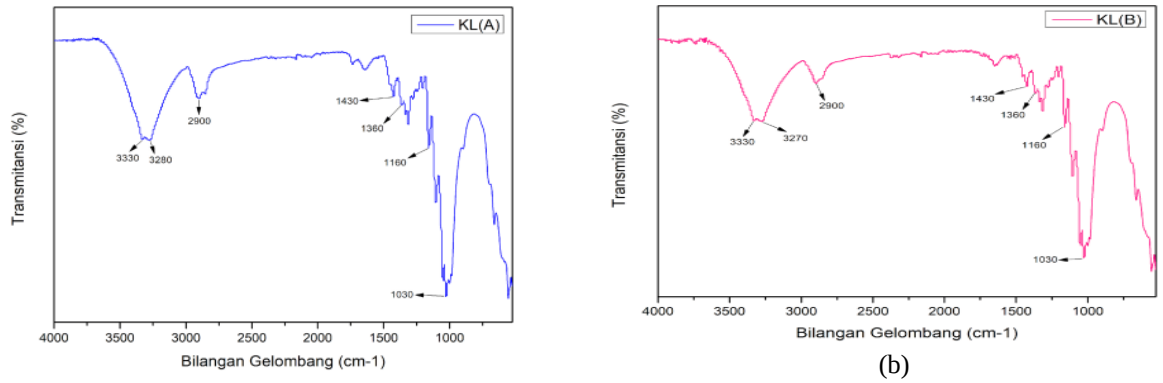
Figure 10. Characterization of cotton fabric with the addition of (a) 1% alum and (b) 0.6% alum after the dyeing process via FTIR ATR

As shown in Figure 10, after being treated with chlorophyll dye, the cotton fabric presented 12 absorption peaks. The C=O stretching bond vibration appears at 1710 cm-1. Cotton fabric with the addition of 0.6% alum presented an absorption peak at 1640 cm-1, which indicates the presence of bending O–H bond vibrations from the absorbed water content [17]. In the wavenumber areas of 1410 cm-1, 1340 cm-1, 721 cm-1, and 720 cm-1, CH2 scissoring, C-H bending, and CH2 rocking bond vibrations occur. Carbonyl groups such as C-O stretches are visible in the wavenumber region of 1090--970 cm-1. At 1240 cm-1, stretching C=O bond vibrations or deformation of O-H or N-H bonds can be observed. Cotton fabric with the addition of 1% alum has asymmetric stretching C–O–C bond vibrations that are less visible, as shown at 1180 cm--1. β-Linkage in cellulose is observed at 896 cm-1[17]. Then, bending (out-of-plane) O-H bond vibrations appear at 671 cm-1.



(a) (b)

Figure 11. Characterization of drill fabric with the addition of (a) 1% alum and (b) 0.6% alum after the dyeing process via FTIR ATR



(a) (b)

Figure 12. Characterization of linen fabric with the addition of (a) 1% alum and (b) 0.6% alum after the dyeing process via FTIR ATR

The characterization results of the drill fabric in Figure 11 show that after being treated with chlorophyll dye, the drill fabric presented 11 absorption peaks. The C=O stretching bond vibration appears at 1710 cm-1. In the wavenumber regions of 1410 cm-1 and 1340 cm-1, CH2 scissoring and C–H bending bond vibrations occur. Stretching C–O bond vibrations are observed at 1090 cm-1, 1020 cm-1, 969 cm-1, and 967 cm-1. The peak at 1240 cm-1 corresponds to the stretching of C=O bond vibrations or deformation of O-H or N-H bonds. Based on drill fabric characterization data, the β-linkage contained in cellulose is visible at 898 cm-1. The wavenumbers at 723 cm-1 and 721 cm-1 indicate the presence of rocking CH_2 bond vibrations.

Based on the characterization data in Figure 12, after being treated with chlorophyll dye, linen fabric produced 6 absorption peaks. At 3330 cm-1, 3280 cm-1, and 3270 cm-1, O–H bond vibrations from intramolecular hydrogen bonds from linen and alum and intermolecular hydrogen bonds appear. The peak at 2900 cm-1 corresponds to C–H bond vibrations from the alkyl group. Moreover, CH_2 bond vibrations appear at 1430 cm-1, and C–H bond vibrations from the methyl group appear at 1360 cm-1. The peak at 1160 cm-1 corresponds to an asymmetric C–O–C bond, and the peak at 1030 cm-1 corresponds to the vibration of the C–O bond in the ether group. [El Sayed, N. A. et al, 2023].

CONCLUSION

The extraction of chlorophyll from pandan wangi leaves via the maceration method was successful, with a yield percentage value of 40.22%. The concentration of chlorophyll extracted from the leaves of pandan wangi plants via the Lichtenthaler equation was 3.7223 µg/mL for chlorophyll a and 1.1362 µg/mL for chlorophyll b. The functional groups from the chlorophyll extracted from the leaves of pandan wangi plants identified via the FT-IR instrument consisted of methyl (C-H), ketone (C=O), amine (C-N), and ester (C-O) functional groups. The quality of the dyes in the three types of fabric was determined by SNI ISO 105-B 01-2010 and SNI ISO 105-X12:2016, with values of color fastness to sunlight of 4 (good) and color fastness to rubbing (dry and wet) of 4–5 (very good).

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