



Full Length Research Article

Production and Characterization of Natural Dyes for Ecoprinting Leather from the Extracts of Three Mangrove Species

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ABSTRACT

This study aimed to determine the potential of *Rhizophora mucronata*, *Maclura cochinchinensis*, and *Ceriops tagal* extracts as a dye for ecoprinting leather. The parameters evaluated were color values (L^* , a^* , and b^*), Fourier-Transform Infrared Spectroscopy (FTIR) analysis, and ecoprinting leather color fastness. The results showed that *R. mucronata* extract produced a brownish-red color with the L^* , a^* , and b^* values of 17.53, 36.27, and 30.19, respectively. *M. cochinchinensis* extract produced a brownish-yellow color with the L^* , a^* , and b^* values of 34.59, 31.87, and 58.61, while *C. tagal* extract produced a reddish-brown color with the L^* , a^* , and b^* values of 32.36, 36.80, and 34.06. The results of FTIR analysis of the three mangrove extracts identified several functional groups such as O-H stretch, C=O stretch, C=C aromatic, C-O-C stretch, and C-N stretch. The color fastness of wet rubbing showed that *R. mucronata* and *C. tagal* extracts have a value of 4, indicating a good category. In contrast, *M. cochinchinensis* extract has a value of 5, with an excellent category. It showed that the extracts of *R. mucronata*, *M. cochinchinensis*, and *C. tagal* could be used for leather coloring with the ecoprinting method.

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1. Introduction

The ecoprinting technique is a coloring technique using natural dyes, which is carried out by transferring colors and shapes to a cloth and leather through direct contact. The ecoprinting technique uses plant parts in the form of leaves and flowers as a source of dye. The use of ecoprinting can eliminate the impression of mass-produced products. In addition, it produces new images for designs on leather and is also environmentally friendly (Nurcahyanti and Septiana 2018). The advantages of natural dyes for ecoprinting leather are environmentally friendly, renewable, non-toxic, sustainable, and soft in color. Furthermore, plant-based dyes are easy to obtain, and their substrates are fragrant and soft (Pervaiz et al. 2016). Some uses of natural dyes for leather coloring that have been carried out include natural coloring using *Bixa orellana* seed extract (Selvi et al. 2012), henna plant extract (Chukwu et al. 2011), *Osyris quadripartite* plant

extract (Teklay and Kechi 2018), *Acacia catechu* extract (Singh et al. 2020), and *Tagetes erecta* extract (Pervaiz et al. 2017).

Meanwhile, the limited number of natural resources is one of the obstacles for global producers in obtaining the raw materials to produce natural dyes (Saxena and Raja 2014). Indonesia is among the countries blessed with abundant flora wealth, making it easy to obtain natural dyes (Putra et al. 2014) from various main parts of plants such as bark, twigs, stems, leaves, roots, seeds, flowers, and sap. The use of natural dyes from wood or bark extracts has several advantages and disadvantages. The advantages include its tanning effect on the leather, increasing the fullness of the leather, and various metal salts. In addition, it gives different or the same color to the meat (flesh) and nerfs (grain). However, excessive amounts give the leather a tough nature (Upadhyay and Choudhary 2014).

Rhizophora mucronata is widely distributed in Indonesia, with an estimated area of approximately 4.25 million ha (Suhendry et al. 2017). The bark of *R. mucronata* contains tannins, flavonoids, and quinones as a source of natural brown dye (Yusro 2011). *Maclura cochinchinensis* is a plant that is widespread in South Asia (Himalaya, Nepal, and India), East (Japan), and Southeast (Malay Peninsula, Papua Island, Bismark Island, New Caledonia to Eastern Australia). The compounds found in the bark and wood of *M. cochinchinensis* are flavonoids, alkaloids, steroids, saponins, and tannins. The main flavonoid in *M. cochinchinensis* wood is morin, which gives silk its yellow color (Swargiary and Ronghang 2013). Meanwhile, *Ceriops tagal* trees grow in many areas in Indonesia, including Central Java, East Java, Sulawesi, and Kalimantan, especially in coastal areas. The chemical compounds of dye in the bark of *C. tagal* are tannins and trimeric. *C. tagal* wood is generally used as firewood, while the bark is used as a dye for batik (Kasmudjiastuti 2017). Indonesia is rich in sources of natural dyes from wood or bark. Therefore, it is necessary to explore and develop its use for application to leather, especially for coloring the ecoprinting technique. Since there are limited reports on natural dyes, this study focused on the use of natural dyes from *R. mucronata*, *M. cochinchinensis*, and *C. tagal* for ecoprinting leather.

2. Materials and Methods

2.1. Production of Dye Extract

The dye from *R. mucronata* was extracted from leaves and stem samples. The leaves and stem samples were cut into 2 cm, and the pieces weighing 500 g were put in a saucepan. Water in a ratio of 1/5 (w/v) was added, and the ingredients were boiled at 75°C until the water volume became half (Bogoriani 2010). Subsequently, the solution was filtered using gauze. A natural dye solution is the filtered extract solution, which is ready for use after cooling. *M. cochinchinensis* extract was produced using water solvent (w/v= 1/5) at 75°C for 1 h. The extraction at room temperature was carried out by immersion for 72 h after the temperature reached and filtered, while the filtrate was stored in a closed container (Atika and Salma 2017). The *C. tagal* extract was made from bark. A total of 5 cm long, 2 cm wide, and 5 mm thick with a weight of 5 kg *C. tagal* bark (*Ceriops tagal*) was reduced in size using a crusher machine with a length of 16.7 mm, a width of 4.7 mm, and a thickness of 1.8 mm. 10 kg of *C. tagal* bark was dried and extracted with countercurrent system. The extraction ran at a temperature of 75°C for 24 h. The ratio of materials and solvent used in the experiment is 1/5 (w/v) (Kasmudjiastuti et al. 2015).

2.2. Production of Ecoprinting Leather

The process of making ecoprinting leather was conducted by soaking sheep leather in water until it turned blue. The leaves were arranged on the surface of the leather and then covered with a cloth colored with natural ingredients. The rolling was followed by steaming at room temperature of 28-75°C. After cooling down, the leather was soaked with alum solution for 2 h, unrolled, and then tested (Pancapalaga et al. 2021).

2.3. Observed Parameters

2.3.1. Color test

Color analysis was conducted using a colorimeter (ColorFlex EZ Spectrophotometer, Hunterlab, USA) to obtain the color value (L^* , a^* , b^*) and reflectance spectrum data. The L^* value represented the lightness-darkness in the range of 0-100 (white to black). The a^* value represented reddish (redness) and green (greenness) when positive and negative, respectively, while the b^* value was yellowness when positive and blueness at negative (Hosseini et al. 2013). The sample was placed on a ColorFlex cuvette, and the color properties were measured.

2.3.2. Phytochemical test

2.3.2.1. Tannin test

R. mucronata, *M. cochinchinensis*, and *C. tagal* extracts of 2 ml were put into 2 test tubes. Tube 1 (control) and Tube 2 were added 2-3 drops of FeCl_3 5% or FeCl_3 10%. The green or blue color indicates the presence of tannin (Ugochukwu et al. 2013).

2.3.2.2. Anthocyanin test

The mangrove extracts were added 3 drops of 2 ml HCl, then heated at 100°C for 2 minutes. Positive results were indicated when a red color was observed (Mathivanan et al. 2014).

2.3.2.3. Anthraquinone test

R. mucronata, *M. cochinchinensis*, and *C. tagal* extract of 2 mg was mixed with 10 ml water, then heated for 5 minutes and filtered. Next, 3 ml extract was put into 2 test tubes. Tube 1 was added NaOH 1 N, with positive results indicated when a red color was observed. Tube 2 is the control sample (Ugochukwu et al. 2013).

2.3.2.4. Carotenoid test

R. mucronata, *M. cochinchinensis*, and *C. tagal* extract 2 mg were added to 2 drops of 2M H_2SO_4 . The presence of blue or greenish-blue color indicates the presence of carotenoid compounds (Bogoriani 2014).

2.3.2.5. Fourier-transform infrared spectroscopy (FTIR) analysis

The FTIR test was carried out using an FTIR spectrophotometer (FT/IR-6800, JASCO International Co. Ltd., Tokyo, Japan) with a TGS detector and attenuated total reflectance (ATR

PRO ONE, JASCO Asia Portal Japan) at 45° and a scanning speed of 2 mm/sec. The FT/IR-6800 can measure from the visible (25,000 cm⁻¹) to the far IR (50 cm⁻¹).

2.3.3. Ecoprinting leather color fastness test

The wet rubbing color fastness test was carried out based on Indonesia National Standard, SNI 06-0996-1989 (BSN 1989). The results of the color fastness to wet and dry rubbing were carried out visually by comparing the color changes that occurred with the standard color change using the staining scale. The results of the wet and dry rubbing color fastness test are shown in **Table 1**.

Table 1. Scoring table of color in the staining scale

The value of color fastness	The description of color fastness
5	Excellent
4-5	Good
4	Good
3-4	Fairly good
3	Fair
2-3	Poor
2	Poor
1-2	Very poor
1	Very poor

2.4. Data Analysis

The data collected included color value, phytochemical test results, FTIR analysis, and ecoprinting leather colorfastness were analyzed using descriptive statistics.

3. Results and Discussion

3.1. Ecoprinting Leather Color Value

The results of the ecoprinting leather color values are shown in **Table 2**. The results showed that staining with the *R. mucronata* extract produced a darker color than with *C. tagal* and *M. cochinchinensis* extracts. The L^* value in *R. mucronata* was the lowest compared to other colorings, showing a value of 17.53. Meanwhile, the brightest coloration was produced by *M. cochinchinensis* extract with an L^* value of 34.59. All staining results with *R. mucronata*, *C. tagal*, and *M. cochinchinensis* extracts produced a positive a^* value. This tendency indicated that the color of the *R. mucronata* and *C. tagal* extracts staining points towards red unless the *M. cochinchinensis* is more yellow with a lower a^* value.

Table 2. Results of color values (L^* , a^* , b^*) of the extracts of mangrove species

No.	Mangrove species	Color value		
		L^*	a^*	b^*
1	<i>R. mucronata</i>	17.53 (1.2)	36.27 (1.2)	30.19 (1.5)
2	<i>C. tagal</i>	32.36 (0.6)	36.80 (1.6)	34.06 (1.4)
3	<i>M. cochinchinensis</i>	34.59 (1.4)	31.87 (1,1)	58.61 (1.2)

The highest positive a^* value of 36.80 was obtained using *C. tagal* extract staining at the pH of the acid fixation solution with alum, showing a reddish-yellow color (light orange). All staining results with *R. mucronata*, *C. tagal*, and *M. cochinchinensis* produced positive b^* values, indicating that the ecoprinting leather staining results from the three mangrove extracts are yellow. It is shown in the highest positive b^* value of 58.61 from staining with *M. cochinchinensis* extract pH of alum fixation acid solution. Through learning of the value of this color in the various extract of natural dyes, color absorption on ecoprinting leather can be increased by adding mordant. This tendency is similar to [Ahmad et al. \(2011\)](#), stating that the additional metal mordant forms coordinate bonds with carboxyl groups (-COOH) cellulose and (-NH₂) in leather protein fibers. One dye molecule can form one bond with one fiber molecule. However, one mordant molecule can form two or more bonds with the dye molecule and can increase the absorption of the dye.

3.2. Phytochemical Test of Pigment Content of Three Mangrove Extracts

Table 3 shows the phytochemical test results, indicating that *R. mucronata* and *M. cochinchinensis* extracts contain a class of tannin dyes with a positive symbol. Meanwhile, the extract of *C. tagal* contains tannins, anthocyanins, and anthraquinones but does not contain carotene due to its negative sign. The content of tannin dyes was discovered in the three extracts of *R. mucronata*, *M. cochinchinensis*, and *C. tagal*. Generally, tannins are divided into two, namely hydrolyzed and condensed, while based on the color produced, they are classified into three types, namely clear, yellow, and red-brown tannins ([Atika and Salma 2017](#)).

Table 3. Phytochemical test results of the extracts of mangrove species

No.	Mangrove species	Tannin	Anthocyanin	Anthraquinone	Carotene
1	<i>R. mucronata</i>	Yes (+)	None (-)	None (-)	None (-)
2	<i>C. tagal</i>	Yes (+)	Yes (+)	Yes (+)	None (-)
3	<i>M. cochinchinensis</i>	Yes (+)	None (-)	None (-)	None (-)

The use of tannins from plant parts as a vegetable tanning material will cause color, which is a secondary effect of the tanning process. Tannins are mixtures of digalloyl derivatives of glucose that are soluble in water as colloids, usually colorless but sometimes yellowish-brown or brown ([Hassan et al. 2014](#)). The compounds are catechins, hydroxy acids, and leuco anthocyanins (colorless), which become colored after reacting with a metal salt and are used as dyes and tanning agents. Flavonoids are polyphenolic compounds with 15 carbon atoms consisting of two aromatic rings, linked by three carbon atoms, with the basic structure of flavones and flavanones that give color to the leather. It includes *Acacia catechu*, *Mangrove* sp., *Phyllanthus emblica*, *Terminalia catappa*, and *Uncaria gambir*. Quinone, such as anthraquinone, is a chemical compound that produces natural dyes, which can give a red color, namely alizarin ([Prabhu and Bhute 2012](#)).

3.3. Fourier-Transform Infrared Spectroscopy (FTIR) Analysis

The results of the FTIR analysis of natural dyes from *R. mucronata*, *M. cochinchinensis*, and *C. tagal* are shown in **Fig. 1** and **Table 4**. The results obtained several wavenumber spectra identical to that of tannins. The UV Vis spectra of the materials showed that the maximum absorbance occurred at wavenumbers 3198 cm⁻¹ for *C. tagal*, 3225 cm⁻¹ for *M. cochinchinensis*, and 3.204 cm⁻¹ for *R. mucronata*. The results indicated that the presence of conjugated O=H bonds

is high. Furthermore, the maximum absorbance value at a wavelength between 1,500-1,650 cm^{-1} indicated the presence of conjugated C=C bonds and C=O chromophore (Sari et al. 2015). Tannins are natural polyphenolic compounds containing phenolic hydroxyl, carboxyl, and chromophore groups that generally cause color in a compound. Conjugated C=C and C=O bonds are included in the chromophore group, which supports the assumption that the brown color from the extraction originated from tannins.

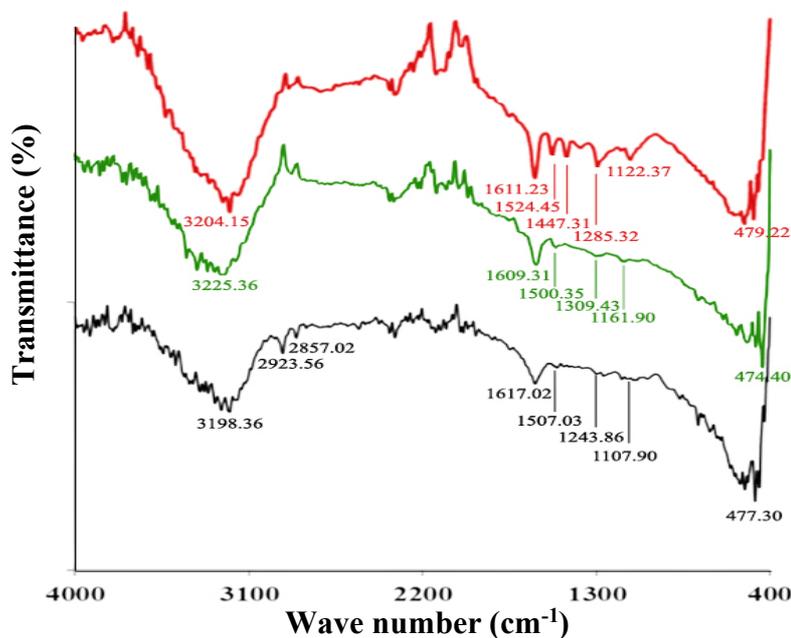


Fig. 1. The results of the FTIR analysis of natural dyes from the extracts of mangrove species (red line= *R. mucronata*, green line= *M. cochinchinensis*, black line= *C. tagal*).

The results of FT-IR analysis on the extraction of *R. mucronata*, *M. cochinchinensis*, and *C. tagal* dyes showed wide absorption with wavenumbers ranging from 1,200 to 1,400 cm^{-1} , namely 12,43.86 cm^{-1} in *R. mucronata*, 1,309.43 cm^{-1} in *M. cochinchinensis*, and 1,285.32 cm^{-1} in *C. tagal*. The presence of absorption indicates the availability of a C-O-C group subsequent absorptions, which occurred in the range of wavenumbers 3,500 and 3,000 cm^{-1} in the three samples, namely 3,198.36 cm^{-1} at *C. tagal*, 3,225.36 cm^{-1} at *M. cochinchinensis*, and 3,204.15 cm^{-1} in *R. mucronata*. The last and weakest absorptions are in wave numbers below 1,200 cm^{-1} . It is because very weak absorption occurred in *C. tagal* extract at 1,107.90 cm^{-1} , followed by 1,161.90 cm^{-1} in *M. cochinchinensis* extract and 1,122.37 cm^{-1} in *R. mucronata* extract. The absorption in the range of 1,200 and below indicated the presence of a C-N group.

Table 4. Band positions and assignments in the infrared spectra of the extracts of mangrove species

No.	Wavenumber (cm^{-1})			Alleged functional group
	<i>R. mucronata</i>	<i>M. cochinchinensis</i>	<i>C. tagal</i>	
1	3204	3225	3198	O-H stretch (3200-3650 cm^{-1})
4	1611	1609	1617	C=O stretch (1580-1650 cm^{-1})
5	1524	1500	1507	C=C aromatic (1500-1600 cm^{-1})
6	1285	1309	1243	C-O-C stretch (1310-1020 cm^{-1})
7	1122	1161	1107	C-N stretch (1020-1250 cm^{-1})
8	479	474	477	Cannot be identified

The three dye extracts of *R. mucronata*, *M. cochinchinensis*, and *C. tagal* showed similar absorption patterns. The presence of absorption in the range of 2,000 to 1,500 cm^{-1} also indicates the presence of a C-O group. The C-O group forms aromatic compounds that are part of the tannins with the O-H and $-\text{CH}_2$ (Paryanto 2020), while the O-H, C=O stretching, and C-O-C ether groups were also present. Moreover, the allegations of these three types of functional groups indicated that flavonoid compounds were present in the extract. This tendency is supported by a previous study (Parubak 2013) which discovered that flavonoid compounds from the flavanone group have bound OH functional groups, aliphatic CH, C=O, C=C Aromatic, C-O, and C-H aromatic. It was also shown that flavanones are the building blocks of proanthocyanin compounds, which are condensed tannins (Chandran et al. 2022).

3.4. Wet Rub Fastness of All Three Kinds of Natural Materials

Table 5 shows that the value of wet rub colorfastness using various extracts from *R. mucronata*, *M. cochinchinensis*, and *C. tagal* has met the SNI 06-0996-1989 (BSN 1989) standard that required a minimum value of 3. The data also showed that the highest wet rubbing color fastness was produced by staining with *M. cochinchinensis* extract with a value of 4-5, which indicated an excellent fastness. Meanwhile, the values of 3-4 were obtained by *R. mucronata* and *C. tagal*, which are good for resisting fastness from wet rubbing. The colorfastness value of wet rubbing in this study followed SNI 06-0996-1989 based on the evaluation standard of wet rubbing testing with a minimum value of 3. A study using an alum mordant showed that alum-type mordant could bind natural colors into the leather fiber. According to Samanta and Konar (2011), mordant alum will form a suitable coordination complex to create affinity between the fiber and the dye or natural dye pigment molecules. The alum metal salts attached to the leather surface will also attract the organic dye/pigment molecules in the leaves to be anchored to the fibers and create a link between the dye molecule and the fiber by removing a coordination complex.

Table 5. The value of the wet rub fastness of ecoprinting leather with natural coloring

No.	Mangrove species	Cycle				
		1	2	3	4	5
1	<i>R. mucronata</i>	3-4	4	3-4	4	4
2	<i>M. cochinchinensis</i>	5	4-5	4-5	4	5
3	<i>C. tagal</i>	3-4	4	3	3-4	4

4. Conclusions

The extracts of *R. mucronata*, *M. cochinchinensis*, and *C. tagal* can be used as a natural leather dye using the ecoprinting technique by producing different colors and having a good to excellent wet rub fastness. Furthermore, the results of ecoprinting leather met the SNI 06-0996-1989 standard.

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