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Extraction of natural dye powder from morinda *citrifolia* and its application as antibacterial dyes for cotton fabrics

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Abstract. Since hundreds year ago, the application of natural dyes on batik is unique procedure to obtain special fabrics and colour. Today, this technique is gaining popularity all over the world, because of the increased awareness on environmental, ecological aspects, and pollution control. In this research, extraction of dyes from wood of *Morinda citrifolia* using simple precipitation assisted brine solution has been investigated. The color components extracted and isolated from *Morinda citrifolia* plant were characterized by Thin Layer Chromatography (TLC), IR techniques and reflectance methods. The natural dye extract obtained from the *Morinda citrifolia* was used for the dyeing of cotton fabrics. The results indicated that the extracted dye increase significant improvement in fabrics colour. The treated fabrics showed excellent antibacterial activity Gram-positive bacteria Staphylococcus aureus (S. aureus).

1. Introduction

Batik is a hallmark of Indonesian culture, which is recognized as a world cultural heritage. Batik as a cultural product that has a high added value that needs to be balanced with quality and superior functionality. Batik fabric has antibacterial functional ability with natural color excellence expected to gain consumer interest [1]. Added value of functional textiles with antibacterial capability will increases competitive advantage, so there has been growing interest in application of various natural dyes on fabrics. In the meantime, the use of additional materials that are not environmentally friendly, the waste from it processes become a sensitive issue for the textile industry. Therefore, research on Indonesia natural resources that has antibacterial capability needs to be conducted

The use of noni plants as a source of dye cloth traditionally in Indonesia has been going on for generations [2]. Areas such as Sumatra, Java, Kalimantan, Sulawesi, Bali and Flores have utilised dye from roots and stem of noni for a typical traditional fabric dyeing. Stems and roots are commonly used as part of the main sources of dyes [3]. In addition to having a distinctive color, dye ability of noni also showed antibacterial and antiviral antivity [4, 5, 6]. This ability come from the component of secondary metabolites such as anthraquinone, flavonoids, tannins, which are present in the roots and stems of the plant Morinda citrifolia [7]. Since Noni is a tropical plant, thrives in Indonesia, it ensures the availability and environmental sustainability.

The use of natural dyes as traditional textile dyes generally use simple extraction techniques. These techniques are only able to produce a concentrated liquid dye. However, there are weaknesses in terms

of color stability and shelf life of the product concentrates. When concentrates are used in the dyeing process, product colour differences will always occur due to the high level of difficulty to determine the amount of dye to the fabric to be dyed. Likewise, there is an issue relates to concentrates power savings which cannot be stored longer. Thus, natural dyes should always be made fresh. Manufacture of natural dyes in powder form which is easy to use and can be stored for longer period of time are the objectives of this study. The use of a simple technique using saturated saline fluid will be tested on the dye concentrate from noni roots thus obtained powder dyes, as has been done by Prabhu and Deli to isolate dyes from seed tamarin [8]. In this study, we investigate potential use of natural dyes from roots of Morinda citrifolia for batik dyeing. Also, extensive use of the noni plant in traditional fabric dyeing and its potential economic value derived from batik fabrics will also be investigates.

Identification of phytochemical composition, thin layer chromatography is used to determine the main constituent components of extracted dye. Effect of repetition of dyeing, the resulting color will be verified using reflectance spectroscopy and chromameter. Infrared spectroscopy is used to study the interaction among various dye cotton fabrics. Meanwhile, the performance of the antibacterial measures are conducted to verify antibacterial ability of the resulting dye [9]. At the end of this study we expect to obtain information about characteristics of the dye powder noni roots, the color obtained, its interaction with the fabric , as well as its anti-bacterial ability. These information will then be applied to the next process of making traditional batik clothes.

2. Methods

We use experimental method involving the collection and processing of samples, extraction in distilled water using heating technique, and the application of the extract dyes on cotton fabric. We sterilized equipment and materials, and prepared specific bacterial testing of the pour plate method to gain information on the antibacterial activity of sample. The sample used in this study is the root of Morinda citrifolia Cilacap. Two Hundred grams of dried sample was heated with distilled water to reach ratio of 1: 2. Heating was discontinued to obtain roughly half volume of solution which then followed by filtration to separate precipitate from product. This process is continued until 4 times repetition. The resulting solution allowed settling overnight. After settling solution is then filtered with polyester fabric and steamed to obtain half the volume. Saturated brine solution was added to dye solution until a ratio of 2: 3 was reached. The precipitate obtained was filtered and oven for 72 hours at 80^{o}C. Antibacterial Activity Test of wood root extract of noni against Escherichia coli and Staphylococcus aureus in the amount of repetition immersion. Characterization of the interaction of dyes on cotton fabric and DRUV measured by FTIR. Phytochemical profile test was conducted on the test against the class of alkaloids, flavonoids, phenolics, saponins, steroids and terpenoids. Extracts were tested with various reagents to identify secondary metabolites contained in the noni roots wood.

3. Results and discussion

Extraction is used to separate dyes from natural ingredients. Natural material utilised is noni (Morinda citrifolia) roots. Morinda root timber is separated from the root bark. Characterization of extract dye test was carried out using phytochemicals test. The test show a class of phytochemical compounds secondary metabolites contained in the noni tree roots. Test was conducted on alkaloids, flavonoids test, test phenolic, saponin test, test terpenoids, and steroids test.

Results of phytochemical test are shown on the table below:

Table 1. phytochemistry test results of dye powder from morinda citrifolia tree roots.

Phytochemistry test	alkaloid	Phenolic	saponin	flavonoid	terpenoid	steroid
Noni root extract dye	-	+	-	+	-	+

Result from Phytochemical test indicated that powder dye from root extract of Morinda wood showed a positive reaction for steroid compounds, having marked colour changes to green on the addition of

concentrated H_2SO_4. Flavonoid test gives a positive result with the formation of a reddish orange color in the extract solution. The addition of concentrated HCl and Mg powder was applied in flavonoids test. Addition of HCl aims to detect compounds containing core benzopiron. Red or orange color comes from the salts formed benzopirilium also called salt flavilium. Positive reaction with the blue-black color in the extract solution obtained after addition of $FeCl_3$ on phenolic test. Negative reactions obtained in testing the compounds alkaloids, saponins, and terpenoids.

Separation by TLC (Thin Layer Chromatography) where the use of TLC ¬ Silicagel 60 F254 Merck as a separation medium. Eluent pure or mixture will elute the compounds in the sample along the stationary phase [10]. Eluent used is a comparison between chloroform: Acetone: Ethanol (90:0,45:10) as the mobile phase. TLC separation results obtained a few spots that show the separation of polar compounds by class of secondary metabolites contained in the extract of noni root wood.

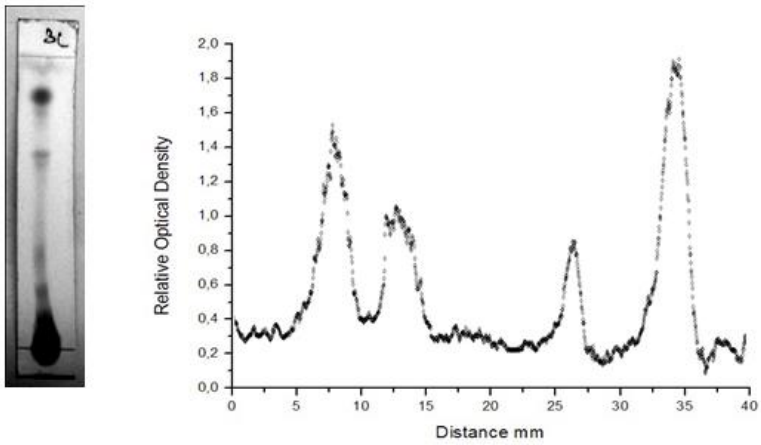


Figure 1. (a) The test results TLC with eluent (chloroform: acetone: ethanol) (90:0,45:10) under UV light (b) The ROD Graph.

Observation under UV light (figure 1 a) obtained spot arising from the chromophore group in the form of a conjugated double bond or aromatic ring. The next spot is converted in the graph obtained ROD (figure 1b) which will be obvious separation divided into four peaks. Differences caused by the high peak concentrations of each compound class of secondary metabolites were dissolved at each spot. Greater the distance spot indicates lower level of secondary metabolite classes polar compounds. On TLC in general compounds that have a low polarity will elute faster than polar compounds as polar compounds bind more tightly to material containing silica silanol ($SiOH_2$) which basically has a strong affinity for polar compound.

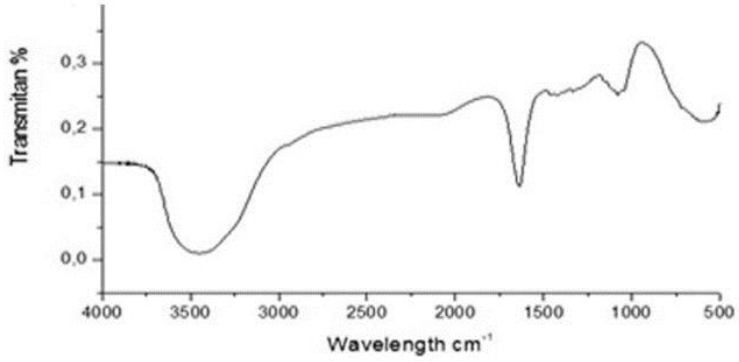


Figure 2. FTIR spectra of powder wood extract noni roots to determine the functional groups contained therein.

Tests using Fourier Transform Infrared (FTIR) was performed to observe the absorption bands identify the functional groups in wood extract noni roots. The spectra have absorption bands characteristic for each molecule functional groups. FTIR spectra of powder wood extract noni roots (figure 2) show the presence of an OH group which is characterized by a peak at 3452 cm^{-1}. There is a second peak in 1634 cm-1 indicating the presence of C = C conjugated with C = O this is possible because there is a flavonoid derivatives or anthraquinone [11]. Most colors in plants due to the presence of flavonoids. An estimated 2% of all carbon is converted into chemicals by plant flavonoids and derivatives [12]. While to mention that the literature anthraquinone anthraquinone secondary metabolites found contained in the Morinda citrifolia roots [13].

Optimization of solubility of the dye in the form of powder to distilled water was conducted to determine the ability of soluble extract dyes from Morinda citrifolia root wood. Dyes from the roots of Morinda citrifolia extract optimum 0.26g/10mL soluble in distilled water with a temperature of 90ºC, heating distilled aims to maximize the solubility of the dye may be dissolved.

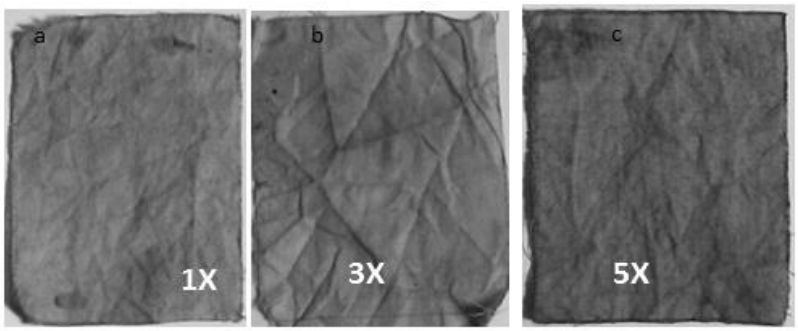
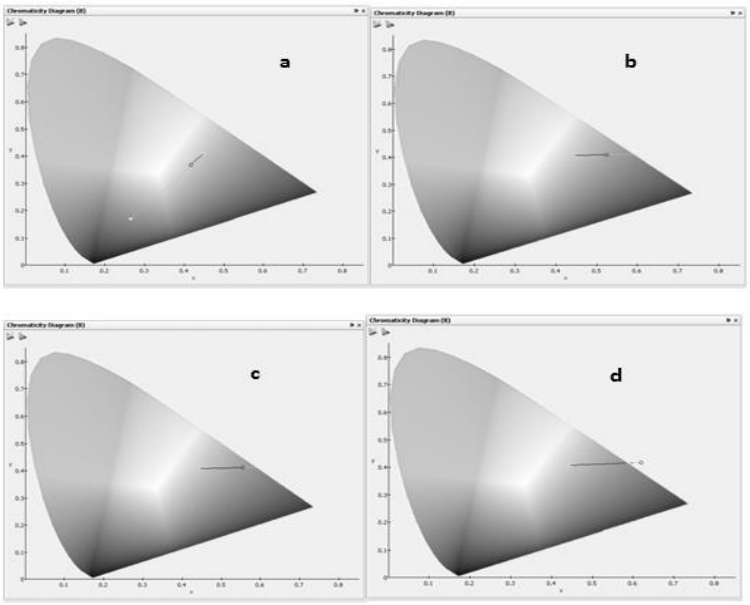


Figure 3. Dyeing cotton fabric using cold dyeing techniques.

Data obtained from the reflectance chromameter fabric using DR-UV instruments Ocean optics DH-2000-BAL on lab instruments UNJ. This information shows the differences in the results of reflectance and color intensity can determine how dark the colour is invisible to the eye.



Figur 4. (a) Diagram of uptake prior to dyeing cotton fabric color as standard (b) Diagram of the absorption color cotton wood root extract of Morinda citrifolia on dyeing 1X (c) Diagram of the absorption color cotton wood root extract of Morinda citrifolia on dyeing 3X (d) Diagram of the color uptake cotton cloth to extract timber in the noni roots dyeing 5X.

Colour absorption data in figure 4 (a) is where the cotton fabric before dyeing cotton fabric that looks white to the eye did not show chromameter intensity. Less white colour may be caused by the presence of protein impurities in cotton fabrics. Fur ther more the figure 4 (a) with figure 4 (b) there is a difference absorption intensity colour indicates that the dye has been absorbed in cotton fabric. While the figure (b) to (c) and (d) visible difference absorption intensity color that appears, where the length of the line from the starting point (0.45) is equal to the left towards the red-brown color on color density diagram showed by chromameter data.

Fabric dyeing results were tested by reflectance to determine dye uptake of many variants. Information that will be obtained is the optimum uptake of variation dyeing cotton fabric to dye. Dyeing is done at room temperature and allowed to stand any immersion for 30 minutes. It is intended that the optimum dye can be absorbed by the fabric.

Figure 5 is a spectrum of cotton fabrics without dye uptake to recognize the difference between before and after dyeing.

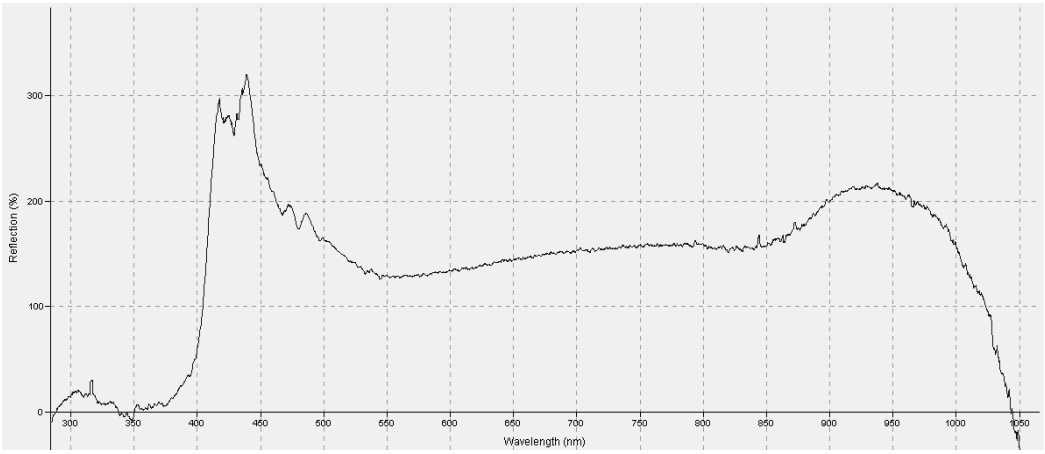


Figure 5. Spectrum of cotton fabrics without dye.

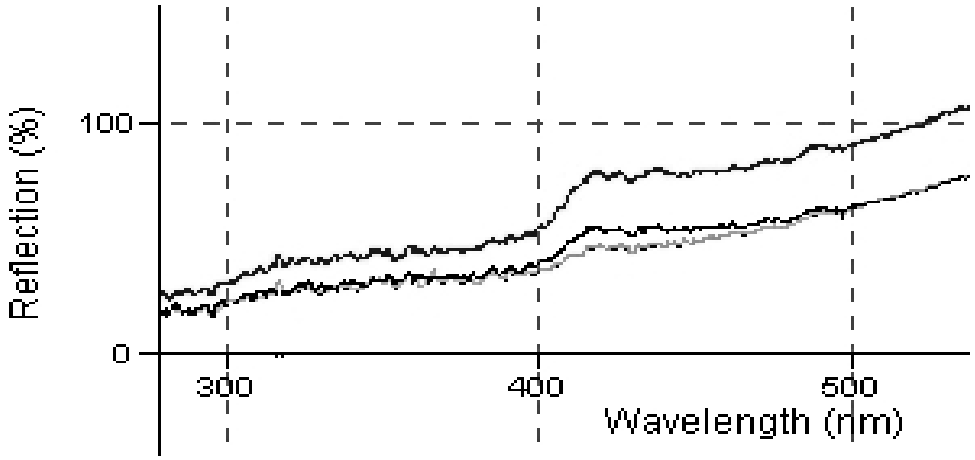


Figure 6. Reflectance spectra in the wavelength dyeing fabric with 1X, 3X, and 5X.

Figure 6 shows reflection differences in color absorption dyeing variations carried out in this experiment. Significant differences occurred between 1X to 3X immersion dyeing, where the absorbance at 1X dyeing produces a high reflectance due to the low absorption of the dye and the fabric light absorbed is proportional to the dye uptake. Because the sample absorbs some of the radiation intensity I is weaker than the intensity of the early I_0. While the lowest was on dyeing reflectance 5X, it can be concluded that the dye uptake was highest in the repetition of immersion to 5 where in each immersion allowed to stand for 30 minutes and dried in the sun without sun for 2 hours.

3.1. Antibacterial test

Antibacterial test method that carried out is the Pour Plate method with Nutrient Agar as medium. Antibacterial test results using a fabric dyeing dye wood noni roots and calculated inhibition zone formed after incubation for 24 hours. The bacteria applied in this test are bacteria commonly infect humans through the skin, which is a gram-positive bacterium Staphylococcus aureus and gram-negative bacteria Escherichia coli [12].

Table 2. Antibacterial inhibition zone test results.

Dyeing process	Bacterial Growth Inhibition Zone Diameter (mm)	
	Escherichia coli	*Staphylococcus aureus*
1x	-	4
3x	-	5,67
5x	-	3,34
Negative control	-	-
Chloramphenicol	8	7,5
Kannaycin	5,5	4

4. Conclusion

Based on the results obtained, it can be concluded that Morinda citrifolia root extract timber containing secondary metabolites phenolic groups, flavonid and steroids. Morinda citrifolia root extract wood has a brownish yellow color on the fabric. Possible interactions that occur between groups of secondary metabolites anthraquinone compounds or flavonoids with cellulose. Wood extract noni roots has an antibacterial effective against gram-positive bacteria, namely Staphylococcus aureus.

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