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Article

Characteristics of Mangium *(Acacia mangium)* Bark Extract with Some Extraction Time and Its Application to Cotton Fabric Dyeing

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Abstract. This study aims to determine the effect of extraction time on the characteristics of mangium bark extract and its application to cotton textile coloring. This study used a completely randomized design (CRD) with 5 treatments and 3 replications, A (10 minutes extraction), B (20 minutes extraction), C (30 minutes extraction), D (40 minutes extraction) and E (50 minutes extraction). . The results showed that the difference in extraction time had a significant effect on the yield, moisture content, tannin content and color of the mangium bark extract. Extraction time of mangium bark for 40 minutes is the optimum extraction time based on analysis of yield, tannin content and color of the extract. Yield analysis at extraction time of 40 minutes obtained the highest yield of 10.72% and tannin content of 15.67%. The cotton cloth that had been dyed with mangium bark extract changed color from yellow to red. The results of the color change test on cotton cloth that had been dyed with mangium bark extract showed that the results did not show very small to small changes when soaked in hot water and detergent.

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1. Introduction

Mangium tree (*Acacia mangium*) is a type of tree used in the pulp, paper, particle board, building materials, household appliances and plywood industries. According to [1-3], the use of mangium trees in the pulp industry will produce waste in the form of sawdust and bark.

Processing of mangium bark waste into various products can be done to increase its use value and selling value, as well as efforts to preserve the environment. Based on [4-6], it was found that mangium bark contains tannin compounds. Therefore, mangium bark can be used as a source of vegetable tannins in the industrial world.

Tannins are complex polyphenolic compounds, have large molecular weights and are soluble in water [7-11]. One method that can be used to obtain tannin compounds contained in plants is extraction using polar solvents [12-16]. Solvent extraction can be done in several ways, one of which is using an ultrasonic bath [17-19]. The use of this tool can increase the yield with a shorter extraction time and lower extraction temperature.

Rahman, H, et al, (2020), stated that the extraction time factor is quite important to consider in the tannin extraction process, because it will affect the quality of the extract. The longer the extraction time, the more the tannin compounds produced will increase until it reaches the optimum time [20-23]. According to [24], have conducted research on the extraction of high soga bark as a natural textile dye using the reflux method at a temperature of 70°C for three minutes. 24343.14 ppm compared to extraction with 2 hours only resulted in tannin content of 17186.27 ppm.

According to [25], textile dyes are all dyes that have the ability to dye textile fibers and are easily removed.One of the requirementsmaterials that can be used as textile dyesishave color-producing groups (chromophores), such as Azo (N=N), Nitrozo (NO), Nitro (NO_2), anthraquinone (=O) and have affinity groups for textile fibers (ausochromes), such as amino (NH) and hydroxyl (OH). According to [26], tannins are composed of conjugated double bonds in polyphenols as chromophores and the presence of OH groups as ausochromes which can cause brown color in materials containing tannins, so that tannin extracts can be used as natural textile dyes.

Natural dyes are dyes obtained from plants, animals or from mineral sources [27-30]. The use of natural dyes in the textile industry should be increased to reduce environmental damage as a result of the use of synthetic dyes [31-34]. Dyed calico textiles using natural dyes from mangium bark extract and produced calico textiles with a purplish-blue color range. Textile materials that can be colored with natural dyes are materials derived from natural fibers, for example cotton [35-36]. In addition, cotton fiber is very strong, heat resistant and can be found easily in the market.

The purpose of this study was to determine the effect of extraction time on the characteristics of the tannin extract of mangium bark, to determine the optimum time of extraction of mangium bark using an ultrasonic bath and to determine the color and color resistance of cotton fabrics that have been dyed with natural dyes of mangium bark.

2. Experimental Section

2.1. Materials and Tools

The basic material used in this research is mangium bark which has been powdered using a hammer mill. The mangium tree used is a tree that grows in the Education and Research forest of the Department of Biology, Andalas University. In this study, Acacia mangium trees aged over eight years with characteristics of rough, hard skin and brown to dark brown were used. The textile material used is toyoba cotton cloth purchased from a textile trader at Pasar Raya, Padang. The chemicals used included 96% ethanol, alum, distilled water, indigocarmin solution, 0.1N $KMnO_4$ solution, gelatin solution, acid salt solution, and kaolin powder.

The tools used in this research include ultrasonic bath, beaker, filter paper, container, sieve, 60 mesh sieve, erlenmeyer, aluminum cup, desicator, oven, measuring cup, hot plate, UV-VIS spectrophotometer and Hunterlab ColorFlex EZ spectrophotometer. The experimental design used in this study was a completely randomized design (CRD) with 5 treatments and 3 replications. Observational data were analyzed by means of variance (ANNOVA) if significantly different then continued with Duncan's New Multiple Range Test (DNMRT) at 5% significance level.

2.2. Research Implementation

The implementation of this research was carried out using five steps, starting with the bark preparation step, then the extraction of tannins from mangium bark, followed by the cotton fabric sample preparation, preparation of staining solution, finally the cotton fabric dyeing process.

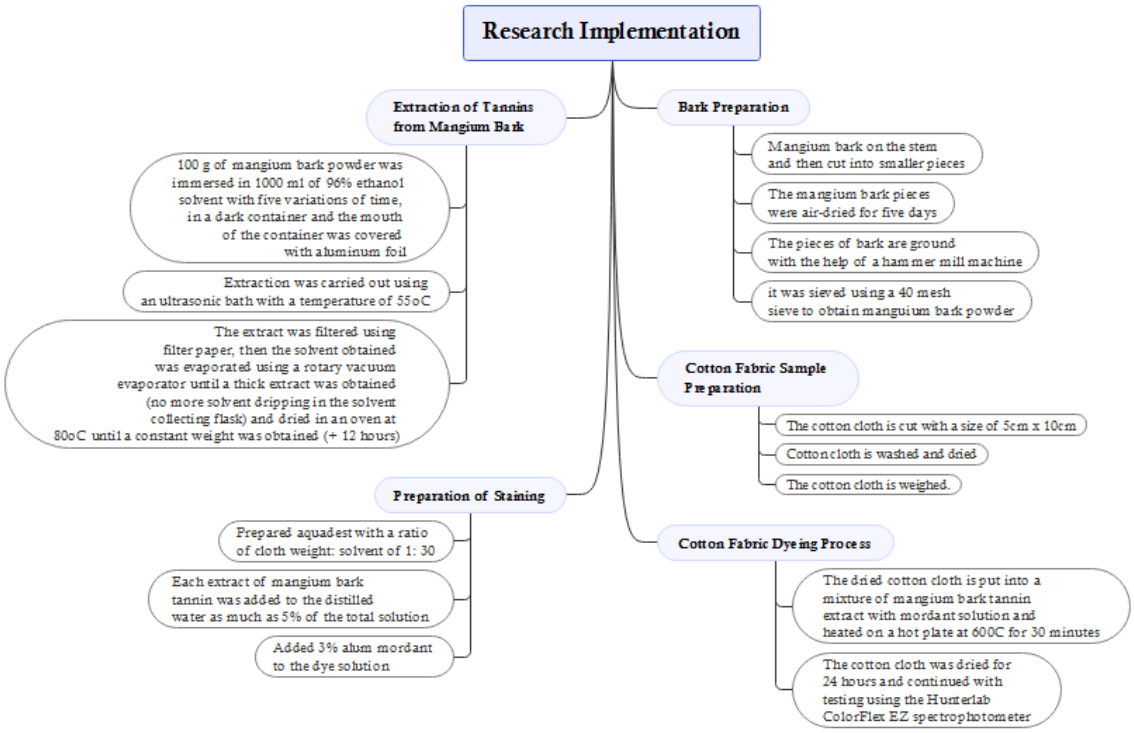


Figure 1. Flowchart of research implementation

2.3 Observation

Observation of raw material for mangium bark, including tannin content, moisture content and color. Observations on mangium bark extract, including yield, tannin content, water content and color analysis. Observations on the results of dyeing cotton fabrics, including the color of the fabric and analysis of differences in the color of the fabric after soaking in plain water, hot water and detergent.

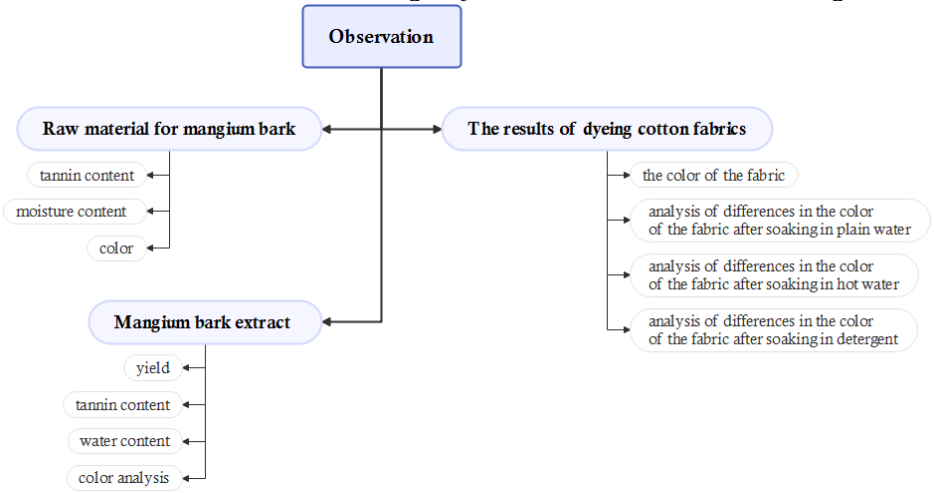


Figure 2. Flowchart of observation research

3. Results and Discussion

3.1 Raw Material Analysis

In this study, the raw material used was mangium bark powder. Observations on raw materials aim to determine the characteristics of the raw materials used. The results of the analysis of raw materials can be seen in Table 1.

Table 1. Average value of mangium bark powder analysis

Analysis	Results (Mean±SD)
Water content (%)	8.31 ± 0.11
Tannin content (%)	10.80 ± 0.23
Color (°Hue)	83.88 ± 0.05 (Red Yellow)

Based on the calculations, the average moisture content of mangium bark powder was 8.31%. The moisture content of the material will affect the effectiveness of the extraction. High water content will complicate the extraction process and require more solvents than materials with low water content. In addition, the moisture content of the material also affects the shelf life and resistance to fungal attack. Raw materials with high moisture content tend to have a relatively short shelf life. According to [35], in his research, obtained a moisture content of 12.67% of Acacia mangium bark powder which was dried in the sun.

Tannin content is the amount of tannin contained in a material and is expressed as a percent. Based on the calculations, the tannin content of the mangium bark was 10.8%. The tannin content obtained was lower than the tannin content obtained which was 15%. However, the tannin content obtained was greater than the tannin content obtained which was 6.25% [37-38].

Tannin levels in plants are influenced by several factors, including differences in species, where they grow and the age of the plant. Differences in acacia plant species and where they grow will affect the tannin content of acacia plants. The older a plant, the lower the tannin content. In this study, 8-9 years old Acacia mangium trees were used with characteristics of rough, hard skin and brown to dark brown in color [38-39].

States that color gives characteristics to various types of wood, but it is difficult to describe in words. Color can be expressed visually, but can also be measured technically. In this study, the color of the mangium bark powder was tested using the Hunterlab ColorFlex EZ Spectrophotometer. Based on the oHue calculation of the bark powder, a value of 83.88 was obtained which refers to the reddish yellow color.

The reddish yellow color of the mangium bark is due to the presence of tannin compounds in the mangium bark. Which states that the bark of mangium acacia is brown which is influenced by the content of tannins. The brown color produced by tannins is caused by the content of phenolic compounds in tannins. One of the properties of materials containing tannins is that they darken when exposed to direct light or left in the open air. There has been no previous research on mangium bark color pigments other than tannins.

3.2 Analysis of Mangium Bark Extract

3.2.1 Analysis of Yield of Mangium Bark Extract

The results of the analysis of the yield of mangium bark extract can be seen in Table 2. The yield was obtained from the calculation of the weight of the dry tannin extract divided by the amount of mangium bark powder used. Extraction of mangium bark powder for 40 minutes resulted in the highest yield of 10.72%. Based on table 2, it can be concluded that the increase in extraction time will significantly affect the yield until it reaches the optimum time.

Table 2. Average yield of mangium bark extract

Treatment	Yield(%) (Average ± SD)
A (extraction time of mangium bark for 10 minutes)	6.64 ± 0.73 a
B (extraction time of mangium bark for 20 minutes)	7.27 ± 0.21 a
C (extraction time of mangium bark for 30 minutes)	8.48 ± 0.40 b
E (extraction time of mangium bark for 50 minutes)	8.57 ± 0.52 b
D (extraction time of mangium bark for 40 minutes)	10.72 ± 0.47 c
KK = 6.05%	

Note: The numbers in the same column followed by lowercase letters are not the same, significantly different at the 5% level according to DNMRT

In the extraction process, there is diffusion of solvent molecules (ethanol) into the pores of the mangium bark, resulting in contact between the ethanol molecules and the molecules in the cinnamon bark. The longer the extraction, the longer the contact time between the solvent molecules and the material molecules, so that the extract obtained will be more and more. However, after passing the optimum time, the addition of extraction time will not increase the yield because the extraction process has reached equilibrium and the solvent has experienced a saturation point [40-41].

The extraction yield obtained is a combination of tannin and non-tannin substances. Extraction of substances containing tannins will usually produce a mixture of heterogeneous substances such as pure tannins and non-tannins such as glucose and hydrocolloids. The use of temperature in the extraction will increase the reaction that occurs so that there is an increase in energy which can cause the decomposition of bioactive compounds and the dissolution of unwanted substances such as starch and sugar [42-43].

3.2.2 Analysis of Moisture Content of Mangium Bark Extract

The results of the analysis of the moisture content of the mangium bark extract can be seen in Table 3. The extract used in this study was a dry extract of mangium bark. Dry extract is a preparation that has a dry consistency and is easily crushed by hand. Dry extracts are obtained from drying and evaporation of materials which should have a water content of not more than 5%. Based on this, the water content of the extract obtained already meets the standard of water content contained in the dry extract.

Table 3. Average value of water content of mangium bark extract

Treatment	Water content (%) (Average ± SD)
E (extraction time of mangium bark for 50 minutes)	1.11 ± 0.50 a
D (extraction time of mangium bark for 40 minutes)	1.56 ± 0.51 a
C (extraction time of mangium bark for 30 minutes)	2.44 ± 0.20 b
B (extraction time of mangium bark for 20 minutes)	2.77 ± 0.18 b
A (extraction time of mangium bark for 10 minutes)	3.10 ± 0.18 b
KK = 16.07%	

Note: The numbers in the same column followed by lowercase letters are not the same, significantly different at the 5% level according to DNMRT

Analysis of the water content of the extract showed that the longer the extraction time, the lower the water content of the material. The longer the extraction time, the water content contained in the material will evaporate. Evaporation of water can occur at all temperatures and does not require a boiling process. Thus, the extraction process carried out at a temperature of 55^{o}C is able to evaporate water in the material, so the longer the extraction time, the more evaporation that occurs so that the water content of the extract becomes lower.

3.2.3 Analysis of Tannin Content of Mangium Bark Extract

The results of the analysis of tannin levels of mangium bark extract can be seen in Table 4. The results of the analysis show that the extraction time affects the amount of tannins extracted. The longer the extraction time, the extracted tannin content increased until it reached the optimum time. In this study, the highest tannin content was obtained at the time of extraction for 40 minutes. Analysis of tannin levels was carried out quantitatively using a UV-VIS Spectrophotometer. The spectrophotometer was operated at a wavelength of 278.5 nm to see the absorbance.

Table 4. Average tannin content of mangium bark extract

Treatment	Tannin Content (%) (Average ± SD)
A (extraction time of mangium bark for 10 minutes)	11.02 ± 0.01 a
B (extraction time of mangium bark for 20 minutes)	13.00 ± 0.00 b
C (extraction time of mangium bark for 30 minutes)	14.05 ± 0.03 c
E (extraction time of mangium bark for 50 minutes)	14.33 ± 0.58 c
D (extraction time of mangium bark for 40 minutes)	15.67 ± 0.50 d
KK = 2.68%	

Note: The numbers in the same column followed by lowercase letters are not the same, significantly different at the 5% level according to DNMRT

Extraction time that is too short will result in low tannin levels, while extraction time that is too long will cause damage to tannin compounds in mangium bark extract. Damaged tannins can decompose into pyrogallol, pyrocatechol and phloroglucinol compounds when extracted at too high a temperature. Therefore, the extraction temperature used must also be considered, because tannins cannot withstand temperatures that are too high, namely above 90^0C [44-45]. In addition, tannins can also be hydrolyzed into glucose and tannic acid.

Tannins are a pigment that gives plants and animals their brown color. The conjugated double bonds in polyphenols as chromophores and the presence of OH groups as ausochromes in the tannin structure can cause brown color in materials containing tannins. The ability to produce brown color in tannins can be used as a source of natural dyes and can be used in natural textile dyes through an extraction process with polar solvents [26, 46].

3.2.4 Color Analysis of Mangium Bark Extract

Color analysis of mangium bark extract was measured using Hunter Lab Color Flex EZ based on parameters L, a*, b*. The L notation shows the brightness with 0 (black) and 100 (white), the a* notation shows the red (positive) and green (negative) chromatic values, the b* notation indicates the yellow (positive) and blue (negative) chromatic values. Furthermore, the calculation of the oHue of the mangium bark extract was carried out. The results of the color analysis of the mangium bark extract can be seen in Table 5.

Table 5. Average color value of mangium bark extract.

Treatment	L*	a*	b*	^{o}Hue (Average ± SD)	Color Range Area
D (extraction time of mangium bark for 40 minutes)	18.81	1.97	0.53	57.25 ± 0.57 a	Reddish yellow
E (extraction time of mangium bark for 50 minutes)	18.87	1.83	0.59	57.53 ± 0.43 a	Reddish yellow
C (extraction time of mangium bark for 30 minutes)	18.93	1.80	0.69	57.56 ± 0.53 a	Reddish yellow
B (extraction time of mangium bark for 20 minutes)	19.19	1.58	0.47	58.22 ± 0.27 ab	Reddish yellow
A (extraction time of mangium bark for 10 minutes)	19.41	1.34	0.35	58.76 ± 0.12 b	Reddish yellow
KK = 0.99%					

The results of analysis of variance showed that the difference in the extraction time of mangium bark had a statistically significant effect on the color of the mangium bark extract. At the L* value, it can be seen that the addition of extraction time resulted in a decrease in the brightness of the mangium bark extract. The longer the extraction time, the darker the color of the resulting extract. The decrease in the brightness of the extract is influenced by the pigments contained in the ingredients.

The more pigment extracted, the darker and denser the color of the extract, so the brightness value decreased [47-48]. The high L* value is an indicator that the pigment has not been extracted completely. In addition, changes in brightness also indicate degradation due to exposure to temperature and light for a long time.

The hue obtained ranged from 57.25 – 58.76, which means that the mangium bark extract is reddish yellow in color. This is caused by the content of tannins which have yellow to brownish pigments. Changes in oHue also reflect changes in tannin levels during the extraction process. The oHue value obtained shows that the longer the extraction time, the more red and darker the mangium bark extract will be.

The dye produced by mangium bark is thought to be used as a natural textile dye. According to [25], one of the requirements for materials that can be used as textile dyes is to have a color-generating group (chromophores), such as Azo (N=N), Nitrozo (No), Nitro (NO_2), anthraquinone (=O) and have anthraquinone (=O) groups, affinity for textile fibers (ausochromes), such as amino (NH) and hydroxyl (OH).

Based on the above requirements, mangium bark tannins can be used as natural textile dyes because they are easily soluble in water, and have a chromophore group, namely C=0 and an ausochrome group, namely OH, which produces a brown to brownish color.

3.3 Cotton Fabric Dyeing Analysis

3.3.1 Cotton Fabric Color Analysis

The results of the color analysis of cotton fabric after being colored with mangium bark extract can be seen in Table 6.

Table 6. Analysis of cotton fabric color

Sample	L*	a*	b*	°Hue	Area Color Range
1A (mangium bark extraction time for 10 minutes)	44.09	5.58	7.98	24.64	Red
1B (extraction time of mangium bark for 20 minutes)	43.69	5.88	8.06	22.08	Red
1C (extraction time of mangium bark for 30 minutes)	43.47	5.95	8.17	20.66	Red
1D (extraction time of mangium bark for 40 minutes)	38.71	6.27	8.66	12.20	Red
1E (extraction time of mangium bark for 50 minutes)	43.21	5.88	8.2	20.70	Red
Control (Cotton Unstained Mangium Bark)	80.08	0.04	0.02	90.04	Yellow

Description: L* = Brightness, a* and b* = Chromaticity Coordinates, °Hue = overall color

The difference between cotton fabrics before and after being dyed with mangium bark extract can be seen in Table 7.

Table 7. Differences in color of cotton fabrics before and after dyeing with mangium bark extract with different extraction times (A=10 minutes, B=20 minutes, C=30 minutes, D=40 minutes, E=50 minutes).

Sample	Before Staining with Mangium Bark Extract	After Coloring with Mangium Bark Extract
1A		
1B		
1C		
1D		
1E		

Dyeing is the process of giving color using a dye which is a combination of organic substances, chromophore as a color carrier and ausochrome as a binder between color and fiber. Dyeing is one of the methods that can be used in textile dyeing. Dyeing is the process of giving color evenly to textile materials in the form of fibers, yarns and fabrics. The absorption of dye occurs due to an exothermic reaction and equilibrium. So in the event of immersion there are three important events, namely [49]:

a. Migration, namely the dissolution of natural dyes and the movement of the dye solution to stick to the material.
b. Adsorption, which is pushing the dye solution to the surface so that it is absorbed and sticks to the material.
c. Diffusion, namely the binding of dye with fibers from the surface of the material into the material.

Cotton fabric before dyeing has a yellow color range with an °Hue value of 90.04. This is in line with [50-51], which also measured the °Hue of cotton fabrics and obtained an °Hue value of 99.12 with a yellow color range.

Based on color analysis, the color of cotton fabric that has been colored with mangium bark extract leads to a red color. This color change is influenced by the content of tannins as natural dyes along with alum mordant which can strengthen the color. The results of the °Hue analysis of cotton fabrics show that the longer the extraction time, the darker the color of the resulting fabric.

The decreasing brightness level is caused by the increasing number of components of dye compounds (tannins) used. This is supported by the research of [52], that the high concentration of dye components in the extract causes the stability of the dye to increase so that the color will be more concentrated and dark.

3.3.2 Analysis of Color Differences in Cotton Fabrics after Soaking in Cold and Hot Water

The results of the analysis of differences in the color of cotton fabric after soaking in cold water can be seen in Table 8.

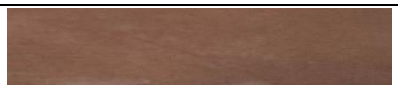
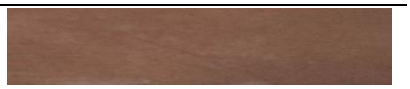
Table 8. Analysis of color differences in cotton fabrics after soaking in cold water

Sample	Before Test			After testing			ΔEE	Change
	L*	a*	b*	L*	a*	b*		
1A	44.09	5.58	7.98	44.08	5.58	7.98	0.01	Not visible
1B	43.69	5.88	8.06	43.69	5.78	8.06	0.10	Not visible
1C	43.47	5.95	8.17	43.46	5.92	8.13	0.05	Not visible
1D	38.71	6.27	8.66	38.41	6.08	8.66	0.19	Not visible
1E	43.21	5.88	8.20	43.21	5.88	8.13	0.07	Not visible

Note: Description of Cloth Color Change Refers to Table 4.

Changes in the color of cotton cloth after soaking in cold water can be seen in Table 9.

Table 9. Changes in color of cotton fabrics before and after soaking in cold water (A=10 minutes, B=20 minutes, C=30 minutes, D=40 minutes, E=50 minutes).

Sample	Before Soaking with Cold Water	After Soaking with Cold Water
1A		
1B		
1C		
1D		
1E		

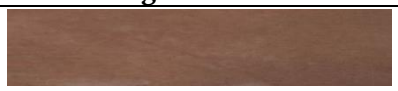
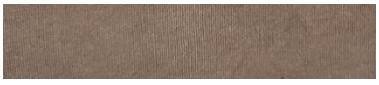
Changes in value indicate the value of the color change. The higher the value, the greater the fastness, and the lower the value, the smaller the color fastness. The color change test of cotton cloth soaked in cold water has a value range between 0.01-0.19, the value is in the invisible category. Based on this, it can be stated that there is a strong bond between mangium bark dye and alum mordant. The mordanting process is able to increase the interaction between the dye and the cotton fiber through the formation of a complex that results in higher dye absorption and dye stability. This condition causes the least amount of dye to be released when soaked in cold water $\Delta E\Delta E\Delta E$EΔEE. The results of the analysis of the color of cotton fabric after soaking in hot water can be seen in Table 10.

Table 10. Color analysis of cotton fabric after soaking in hot water.

Sample	Before Test			After testing			ΔEE	Change
	L*	a*	b*	L*	a*	b*		
1A	44.09	5.58	7.98	44.52	4.97	7.88	0.75	Very small
1B	43.69	5.88	8.06	44.49	5.76	7.66	0.90	Very small
1C	43.47	5.95	8.17	44.09	5.58	7.98	0.75	Very small
1D	38.71	6.27	8.66	40.71	6.02	8.05	2.11	Small
1E	43.21	5.88	8.20	44.89	5.58	7.98	1.72	Small

Changes in the color of cotton cloth after soaking in hot water can be seen in Table 11

Table 11. Changes in color of cotton fabric before and after soaking in hot water (A=10 minutes, B=20 minutes, C=30 minutes, D=40 minutes, E=50 minutes)

Sample	Before Soaking with Hot Water	After Soaking with Hot Water
1A		
1B		
1C		
1D		
1E		

The color change of cotton fabric after soaking in hot water has a value range of 0.75-2.11 which can be categorized into very small to small changes. This is in accordance with the statement of [48], the bonds of dyes with natural fibers form complex compounds so that they are difficult to fade. This very small change is influenced by the mordanting compound, namely alum. The degree of hue of cotton fabrics after soaking in hot water has increased slightly so that the resulting red color is slightly fainter than the color of cotton fabrics before soaking in hot water.

3.3.3 Color Analysis of Cotton Fabric after Soaking with Detergent

The results of the color analysis of cotton fabric after soaking in detergent can be seen in Table 12.

Table 12. Analysis of differences in color of cotton fabrics after soaking in detergent

Sample	Before Test			After testing			ΔEE	Change
	L*	a*	b*	L*	a*	b*		
1A	44.09	5.58	7.98	44.72	4.86	7.72	0.99	Very small
1B	43.69	5.88	8.06	44.67	5.70	7.60	1.10	Small
1C	43.47	5.95	8.17	43.90	5.61	7.52	0.85	Very small
1D	38.71	6.27	8.66	40.80	5.12	7.78	2.54	Small
1E	43.21	5.88	8.20	44.21	5.60	7.63	1.18	Small

Changes in the color of cotton cloth after soaking with detergent ranged from 0.99-2.54 which can be categorized into very small to small changes. According to [26], alum is a compound that has a stronger complex bond than tunjung so that the resulting complex compound is more stable. The more stable the complex, the more difficult the complex. The degree of hue of cotton cloth after soaking with detergent has increased slightly so that the resulting red color is slightly more muted than the color of cotton cloth before soaking in hot water. Changes in the color of cotton cloth after soaking in hot water can be seen in Table 13.

Table 13. Changes in color of cotton fabrics before and after soaking in cold water (A=10 minutes, B=20 minutes, C=30 minutes, D=40 minutes, E=50 minutes).

Sample	Before Soaking With Hot Water	After Soaking With Hot Water
1A		
1B		
1C		
1D		
1E		

4. Conclusion

Differences in the extraction time of mangium bark had a significant effect on the yield, moisture content, tannin content and color of the mangium bark extract. Extraction time of mangium bark for 40 minutes is the optimum extraction time based on the analysis of yield, tannin content and color of the extract. Based on the yield analysis at the extraction time of 40 minutes, the highest yield was 10.72% and the tannin content was 15.67%. The cotton cloth that has been dyed with mangium bark extract changes its color from yellow to red. The results of the color change test on cotton cloth that had been dyed with mangium bark extract showed no visible changes in color when immersed in cold water and very small to small changes when immersed in hot water and detergent.

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