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Dyeing process optimization of chitosan modified cotton dyed with *Bixa orellana* and *Caesalpinia sappan*

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ABSTRACT

Indonesia is rich in flora diversity, very potential for the development of natural dyes. The advantage of dyeing fabrics with natural dyes is that they contain antioxidants and environmentally friendly. The purpose of this study was to optimize the dyeing process on chitosan-modified fabrics with variations in temperature and solution ratio. The dyes used were kesumba keling seeds (Bixa Orellana) and sappan wood (Caesalpinia sappan). The analysis carried out is the analysis of the value of color differences (ΔE), calculated to determine changes in color intensity. The dyed fabrics were tested for fastness to light, heat, and washing. The best value of ΔE was obtained at a temperature variation of 70 °C and a ratio of fabric weight to volume of solution 1:75 for both dyes from kesumba keling seeds and sappan wood with each ΔE value of 6.22 and 11.84. Supported by the test results of fastness to sunlight and iron heat which gives good results.

1. Introduction

Batik is one of the richness of Javanese culture, especially in Yogyakarta. Long ago, batik craftsmen dyed batik cloth with natural dyes sourced from plant skins, fruit, flowers, or leaves [1]. However, now the dyeing process of batik fabric that is widely used is synthetic dyes. The use of synthetic dyes causes several environmental problems due to their waste, because they contain heavy metals and certain azodyes [2].

Indonesia is rich in plant diversity, very potential for the development of natural dyes. The advantage of dyeing fabrics with natural dyes is that they contain antioxidants that are good for health and are environmentally friendly because they are easily degraded. Plants used as dyes can be obtained around our environment so that they are more economical, especially supported by Indonesia's natural wealth.

However, the use of natural dyes as dyes in fabrics has several problems. The color produced on fabrics dyed with natural dyes is not as bright as the color of synthetic dyes, so to get the desired color requires several repeating steps of dyeing process. In addition, natural dyes do not have good fastness to sunlight and washing when applied on cotton fabric which mostly used by batik craftsmen [3]. As a result, batik craftsmen are not productive because it takes a long time to process cotton fabric with natural dyes into batik, so many batik craftsmen turn to synthetic dyes.

In addition, the colors produced from natural dyes are less diverse. Generally, the dyes used as dyes for cotton fabric of batik are brownish colors derived from the bark which contains tannin. Meanwhile, for other colors, such as the red shades, they have limited resources and have poor quality of fabric dyeing. For example, the seeds of *Bixa Orellana* known as kesumba keling in Indonesia which contains bixin, have orange shades, and the sappan wood (*Caesalpinia sappan*) contains

brazilin which has red shades. Both sources of dye are generally used as food coloring. The weakness of those two dyes is that they are easily oxidized so that the results of the dyeing on the fabric are very easy to fade [4]–[6].

Several attempts have been made to increase the absorption of dyes and increase their fastness to fabrics, by adding UV absorbers [7], TiO_2 [8], etc. One of them is by modifying the fabric with chitosan. Chitosan has begun to be used as an additive in fabrics to improve various functions of fabrics, including increasing the absorption of dyes into fabrics, increasing anti-bacterial properties, and anti-wrinkle [9]–[12]. In addition, chitosan comes from the skin of crustaceans, so it is environmentally friendly. Researchers have conducted research on the use of chitosan as a textile enhancer. The effect of using chitosan as a pre-mordanting on fabric dyeing with natural dyes from noja leaves is that it darkens the color produced on the fabric compared to without chitosan treatment [13]. Preliminary research has been carried out, and the results show positive results, but the chitosan is still not evenly distributed on the fabric, so the resulting color is uneven. The purpose of this research is to optimize the dyeing process on fabrics that have been modified with chitosan by varying the temperature and the ratio of the solution to the color difference variable (ΔE).

2. Research Methodology

2.1. Materials

The materials used in this study included primisima cotton fabric, chitosan (80 degree of acetylation) from Chem Mix Yogyakarta, NaOH, acetic acid (CH_3COOH) and aquadest. The source of the natural dye is the kesumba seeds grown in Yogyakarta and the sappan wood is obtained from the Ngasem market in Yogyakarta.

2.2. Methodology

Cotton fabric was dipped in NaOH solution with a concentration of 5%. Chitosan was dissolved in 0.5% v/v acetic acid solution with a variation of 2.5 g/l. In the pre-mordanting treatment, the cotton fabric was soaked for 60 minutes in a chitosan solution at a temperature of 60 °C. Then the modified fabric was dipped in the kesumba dan sappan wood extract. The ratio between the weight of the fabric and the volume of the solution is 1:25, 1:50, 1:75. The dyeing process was carried out at various temperatures of 50, 60, and 70 °C for 1 hour, then the fabric was dried by aerating. Then the color is fixated by 10% o.w.f Al_2O_3 (of weight fabric).

The analysis carried out is the analysis of the value of color differences (ΔE), calculated to determine changes in color intensity based on the parameters of CIELab L*, a*, and b*. Each value of L*, a*, and b* represents brightness, red-green shades, and yellow-blue shades. The difference in color compared is the fabric with the addition of chitosan and catechins to the fabric without the addition of additives. The value of color difference (ΔE) is calculated by equation (1).

$$\Delta E = \left[(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2\right]^{1/2} \quad (1)$$

Dyed fabrics are tested for fastness to light, rubbing and washing. The test was carried out based on SNI 08-0289-1989 regarding the test of fabric fastness to light. The washing fastness test was carried out based on SNI 08-0285-1989, and the rubbing fastness test was carried out based on SNI 08-0288-1989. Test results in visual form are expressed as greyscale.

3. Result and Discussions

3.1. Dyeing Results

The results of the dyeing of the cotton fabric dyed with kesumba and secang are presented in Tables 1 and 2. From the two tables, it can be seen that the cotton cloth that received the chitosan treatment had a much more intense color than the untreated fabric. This proves that the use of chitosan in the fabric dyeing process can increase the absorption of dye into the fabric. In this case, chitosan functions as a mordant to bridge the dye with the cotton fabric, thereby increasing the affinity of the fabric for the dye and increasing the dye absorbed into the fabric. According to research conducted by Dessie using chitosan as a mordant to increase the absorption of dye into the fabric by 13.1% [14], [15].

Table 1. Dyeing Results on Fabrics with Kesumba Seed Dye

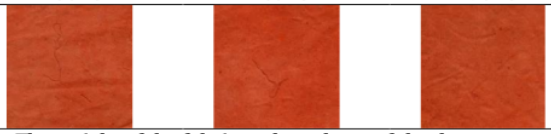
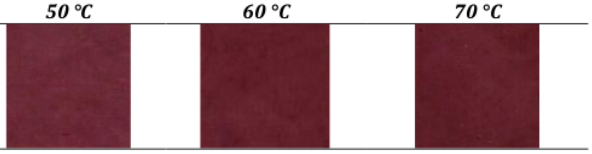
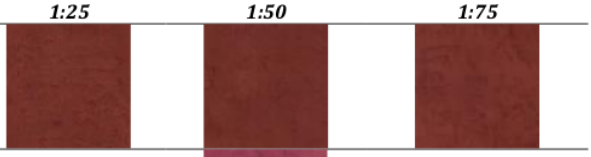
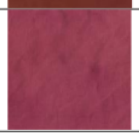
Variation	Kesumba Extract		
	Temperature Variations		
	50 °C	*60 °C*	*70 °C*
Chitosan			
	The weight of the fabric to the volume of the dye extract ratio		
	1:25	*1:50*	*1:75*
			
Untreated			

Table 2. Dyeing Results on Fabrics with Sappan Wood Dye

Variation	Sappan Wood Extract		
	Temperature Variations		
	50 °C	*60 °C*	*70 °C*
Chitosan			
	The weight of the fabric to the volume of the dye extract ratio		
	1:25	*1:50*	*1:75*
			
Untreated			

From the results of the dyeing of chitosan-modified fabrics with natural dyes of kesumba seeds and sappan wood which are presented in Tables 2 and 3, there was no significant visual change in color with variations in temperature and the ratio of fabric weight and dye volume. Therefore, a visual color test was used using the color difference value (ΔE) to be able to see the results of dyeing fabrics with natural dyes.

3.2. Effect of Temperature on Chitosan Treated Cotton Fabric Dyeing

In this study, the effect of the use of temperature variations on the dyeing results of chitosan-modified cotton fabric with natural dyes from kesumba and sappan wood will be seen. The effect of temperature variation is seen from the value of color difference (ΔE) on the fabric without chitosan modification. The results of the analysis of the value of ΔE are presented in Table 3.

Table 3. The effect temperature on dyeing results of chitosan treated fabrics with natural dyes

Natural Dye	Temp	R G B Value			L A B Value			ΔE
		R	*G*	*B*	*L*	*A*	*B*	
Kesumba	50 °C	187.32	68.645	37.08	45.59	46.22	42.96	6.00
	60 °C	190.2	70.93	39.91	46.48	46.34	42.61	5.84
	70 °C	189.80	67.9	36.31	45.87	47.44	43.80	6.22
Secang	50 °C	96.71	33.46	43.11	22.87	29.99	8.87	11.72
	60 °C	97.44	35.09	45.47	23.55	29.25	8.00	11.64
	70 °C	94.95	32.975	42.81	22.63	29.12	8.41	11.84

The results showed that the use of temperature variations affected the dyeing of fabrics with natural dyes. And it can be seen in the data above that the ideal temperature for staining with more concentrated results is indicated by the greater the value of the color difference (ΔE) is at a temperature of 70 °C. On fabrics that use kesumba dye at a temperature of 50 °C, the E value is 6.00, at 60 °C the ΔE value is 5.84, at 70 °C the ΔE value is 6.22, so based on the data above the best value with the best color density is at a temperature of 70 °C. In fabrics that use a sappan dye at a temperature of 50 °C, an ΔE value of 11.73 is found, at a temperature of 60 °C, an ΔE value of 11.64 is found, at a temperature of 70 °C, an ΔE value of 11.84. The best result is at a temperature of 70 °C.

Compared to the results of Ohama's research which carried out dyeing of fabrics with natural dyes from sappan wood using $FeSO_4$ metal mordant where the dyeing results produced a dark lavender color so as quite different from the original red color from sappan wood [5]. Meanwhile, in a study conducted by Zaman who dyed fabrics with natural dyes from kesumba seeds using various metal mordant ($FeSO_4$, $FeCl_3$, and $CuSO_4$) the results of fabric coloring with a light brown final result [16].

Temperature is very influential on the process of dyeing the fabric. Vankar's research states that temperature will have a different effect on the dyeing results. In maddar plants, the dyeing results will be better if the temperature is higher, while indigo dye is good at room temperature [13]. Natural dyes are compounds that are very sensitive to high temperatures. The possibility of structural damage to compounds containing bixin and brazilin dyes in kesumba and sappan causes the absorption of natural dyes into the fabric at high temperatures is not optimal [17].

3.3. Effect of Fabric Weight to Volume Ratio on Chitosan Treated Cotton Fabric Dyeing

This study will examine the effect of variations in the ratio of the weight of the fabric to the volume of the extract on the results of dyeing fabrics with natural dyes. From the analysis of the color difference data (ΔE), the results are shown in Table 4.

Table 4. The effect fabric weight to extract volume ratio on dyeing results of chitosan treated fabrics with natural dyes

Natural Dye	Ratio	R G B Value			L A B Value			ΔE
		R	*G*	*B*	*L*	*A*	*B*	
Kesumba	1:25	205	102.3	55.2	54.88	37.33	44.33	1.03
	1:50	205.4	104.5	57.1	54.04	41.06	47.71	3.68
	1:75	200.1	91.1	52.5	51.88	40.94	42.26	3.49
Secang	1:25	102.2	41.4	35.6	25.47	26.88	28.29	1.78
	1:50	111.7	48.1	41.2	28.53	27.37	18.04	2.74
	1:75	98.8	39.7	34.5	24.48	26.34	16.59	3.48

Table 4 shows the results with the best concentration of each natural dye. In fabrics that use kesumba dye at a volume of 1:25, the ΔE value is 1.03, at a ratio of 1:50, the ΔE value is 3.68, at a ratio of 1:75, the ΔE value is 3.49, so on based on the above data the best value with the best color result is at 1:50. On fabrics that use sappan dye at a ratio of 1:25, the ΔE value is 4.79, at a ratio of 1:50 the ΔE value is 2.75, at a ratio of 1:75 the ΔE value is 3.48, so based on the data above the best value is at a weight-to-volume ratio of 1:75.

It can be seen that the higher the volume, the higher the yield is produced because the contact surface between the solvent and the dye is wider. The yields produced at ratio of 1:25, 1:50 and 1:75 have very little difference. In order to use it more efficiently, the optimum condition was taken at a volume of 1:75. Theoretically, the dye content of kesumba seeds and sappan wood, but from the

experiment, a much larger yield was obtained. It is possible that this happens because at the time of extraction, other components are also extracted. And the higher the level of variation in the volume, the lower the absorption, this is due to the higher concentration of dye, so that it can cover the entire surface of the fabric [18], [19].

3.4. Fastness Test Results

The dyed fabric is then tested for fastness. There are three stages of the fastness test, namely the sun resistance test, the soap washing test, and finally the iron heat resistance test. The test results are presented in Table 5.

Table 5. Fastness Test Results

Natural Dye	Color fastness test		
	Sun	*Soap Washing*	*Iron Heat*
Kesumba	3 (Fair)	2 – 3 (Fair)	4 – 5 (Good)
Secang	4 (Good)	2 – 3 (Fair)	4 – 5 (Good)

Fabrics that are dyed with natural dyes from kesumba have good value for fastness to sunlight and washing. Meanwhile, the fabric colored with sappan dye has a good value for fastness to sunlight on a scale of 4 and fastness to washing with a fair value. Both fabrics dyed with kesumba and sappans extracts have good ironing heat fastness on a 4-5 scale or good result.

4. Conclusions

Temperature and the ratio of the weight of the fabric to the volume of the solution affect the dyeing results on chitosan-modified cotton fabrics with natural dyes from kesumba seeds and sappan wood, where the results of the color difference value (ΔE) give a fairly good difference in dyeing results compared to the dyeing results on untreated fabrics. The best value of ΔE was 11.84 obtained at a temperature variation of 70 °C and a ratio of fabric weight to volume of solution 1:75 for both dyes from kesumba seeds and sappan wood. Supported by the test results of fastness to sunlight and iron heat which gives good results.

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