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Dyeing of Cotton Fabric with Natural Dye from *Peristrophe bivalvis* Extract

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Abstract. Indonesia has high potential for biodiversity of flora as a natural dye. Noja leaf (*Peristrophe bivalvis*) has potential as a red natural dye. The major component of the extract of its leaf is phenoxazine. The influence of mordant type, mordant concentration, and dyeing process temperature on the performance of the dyeing process was studied. The dyeing performances of the dyeing process were appreciated by measuring the colour strength parameter (K/S). Optimization of the dyeing process was done with Response Surface Methodology (RSM). Colour fastness to sunlight, washing, and rubbing was performed. Dyeing with noja leaf extract was done on a water bath with variation of mordant type used was alum (potassium aluminium sulphate), ferrous sulphate and calcium oxide with variation concentration 5%, 10%, and 15 % of the weight of the fabric at temperature variations of 50, 60 and 70ºC. The optimum condition of fabric dyed with noja leaf extract is achieved at 61ºC using ferrous sulphate as mordant with 10.96% concentration yielding colour strength value (K/S) of 0.597. The results of the sunlight, washing, and rubbing fastness test indicate that the natural dye from the leaves of the leaves is quite good as a natural textile dye.

INTRODUCTION

Natural dyes are coloured organic compounds obtained from plants, animals, and minerals. Natural dyes have been used as textile, food, and cosmetics dye since long time ago. Since synthetic dyes were discovered, the use of natural dyes began to be abandoned. Synthetic dyes can provide bright colours, excellent fastness, and affordable prices which make it in high demand. However, synthetic dyes are carcinogenic and harmful to the environment, so the interest to use natural dyes as textile dyes begins to rise again.

FIGURE 1. *Peristrophe bivalvis*, known as noja

Indonesia has high flora biodiversity potential as a natural dye, but red natural colour source that can be applied as textile dye is limited. *Peristrophe bivalvis* or known as noja in Indonesia is an alternative to red natural dyes. Noja is an herbaceous perennial plant which grows in cool and humid climate areas. The red natural dye in the noja leaf

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belongs to phenoxazine alkaloid, but chlorophyll and anthocyanin compounds are also found [1]. Derivatives of phenoxazine alkaloid found in noja leaf are peristrophine and perisbivalvine A [2]. Phenoxazine compounds usually are found limited in insects and fungi [3]. The extraction process was carried out based on the optimum conditions obtained from preliminary research which were using water as solvent at 70°C with material and solvent ratio of 0.3721 grams of leaves / mL of solvent for 40 minutes with a stirring speed of 400 rpm [4].

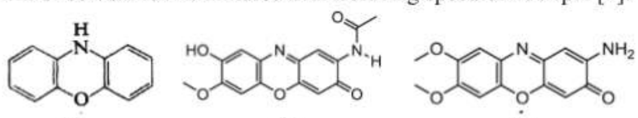


FIGURE 2. Chemical structure of (a) *Phenoxazine*, (b) *Perisbivalvine* A , and (c) *Peristrophine*

The colour fastness of dyed fabric with natural dyes is influenced by the chemical structure of natural dye and mordant used on dyeing process. The structure o 4 atural dye is the main factor determining colour strength of applied fabric. Chemical structure of dyes consists of two main groups of compounds, chromophores and auxochromes. Chromophores determine the colour of the dye while the auxochromes determine the intensity of the colour [5]. Most natural dye compounds that have hydroxyl groups will be more susceptible to oxidation and cause colour to fade [6]. Mordant is a chemical compound which binds dyes to the fabric by forming a chemical bridge from dye to fabric so the colour fastness will be improved [7]. Some natural dyes have no affinity, so they cannot bind to the fabric and have to us 13 mordant to help in binding. Mordant used are commonly metal mordant, such as alum (potassium aluminium sulphate), ferrous sulphate, calcium oxide, and stannous chloride. The process of mordanting on fabric consists of several methods, pre-mordanting, simultaneous mordanting (by simultaneous application of the mordant and the dye), and post-mordanting.

The process of dyeing fabric with natural dyes can be done several times to obtain the desired shade colour intensity. There are several factors that influence the dye absorption into the cotton fabric, colouring process temperature, concentration of dyes, and pH [8].

The present work was conducted to determine the effects of mordant type, mordant concentration, and dyeing 6 ocess temperature on dyeing quality with natural dyes from noja leaf, expressed as colour strength (K/S). The optimization of dyeing process was investigated using Response Surface Methodology. The colour fastness tests of cotton fabric dyed with noja leaf were evaluated. Research on the application of natural dyes from noja leaves as fabric dyes will provide an alternative source of natural dyes that are competitive and affordable.

MATERIAL AND METHODS

Materials

The cotton fabric used in this research was 100% cotton bleached fabric purchased from local trader in Yogyakarta. Mordants used in this research were alum (potassium aluminium sulphate), ferrous sulphate, and calcium oxide were provided by Gamaindigo workshop in Yogyakarta. Noja was grown and cultivated in Yogyakarta.

Methods

Pre-mordanting Process

Pre-mordanting was carried out on cotton fabric using various type of mordants (alum, ferrous sulphate, and calcium oxide) on various concentration (5%,10%, and 15% on fabric weight) at 70°C for 60 minutes keeping material to liquor ratio 1:30. The pre-mordanted cotton then washed thoroughly, squeezed, and air dried.

Dyeing Process

Dyeing was carried out in a dye bath with noja dye extract concentration of 30% v/v at different temperatures (50, 60, and 70°C) for 60 minutes keeping material to liquor ratio 1:100 in a shaker water bath. Then the fabric samples were washed and air dried.

Post-mordanting Process

Post-mordanting was carried out on dyed cotton fabric using the same type of mordants used on pre-mordanting process in same concentration at 25°C for 30 minutes keeping material to liquor ratio 1:30. The post-mordanted cotton then washed thoroughly, squeezed, and air dried.

Measurement of Colour Strength (K/S)

Dyed textile samples were analysed by measuring the reflectance curve between 350 nm and 750 nm using Shimadzu Spectrophotometer UV-2401PC. The colour strength (K/S) values were calculated by Kubelka-Munk equation [9], as shown in Equation (1).

$$\frac{K}{S} = \frac{(1\text{-}R)^2}{2R} \quad (1)$$

Where R is the decimal fraction of the reflectance of dyed fabric, K is the absorption coefficient, and S is the scattering coefficient.

Colour Fastness

The dyed samples were tested according to Indonesian standard methods (SNI). The colour fastness to sunlight was carried out using SNI 08-0289-1989 method, washing fastness using SNI 08-0285-1989 method, and rubbing fastness using SNI 08-0288-1989 method.

Design of Experiment

Optimization of the dyeing process with noja leaves extract from each independent variable (mordant type, mordant concentration, and dyeing process temperature) on the response variable of colour strength values (K/S) was carried out using Response Surface Methodology. The model of the experimental design in the second order reaction is a second order polynomial model with a 2^k factorial design, which is presented in Equation (2).

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum \sum_{i<j=2}^{k} \beta_{ij} X_i X_j + \varepsilon \quad (2)$$

Where β is the regression coefficient.

In the process of the dyeing process with noja leaves extract there were three variables used, type of mordant, mordant concentration, and temperature of dyeing process. The design of experimental was carried out on each type of mordant, so that the remaining 2 independent variables were the concentration of mordant and the temperature of the dyeing process. Based on two independent variables which affect this study, the experimental design is presented in Table 1 and Table 2.

TABLE 1. Coded level of variables for every type of mordant used

Variables	(-)	(0)	(+)
X_1	X_{1a}	X_{1b}	X_{1c}
X_2	X_{2a}	X_{2b}	X_{2c}

TABLE 2. Factorial Design of Experiment

Run	Variables (X)		Response (Y)
	X_1	X_2	Y_p
1	X_{1a}	X_{2a}	Y_{p1}
2	X_{1a}	X_{2b}	Y_{p2}
3	X_{1a}	X_{2c}	Y_{p3}
4	X_{1b}	X_{2a}	Y_{p4}
5	X_{1b}	X_{2b}	Y_{p5}
6	X_{1b}	X_{2c}	Y_{p6}
7	X_{1c}	X_{2a}	Y_{p7}
8	X_{1c}	X_{2b}	Y_{p8}
9	X_{1c}	X_{2c}	Y_{p9}

RESULTS AND DISCUSSION

Colour Strength Value Optimization using Alum as Mordant

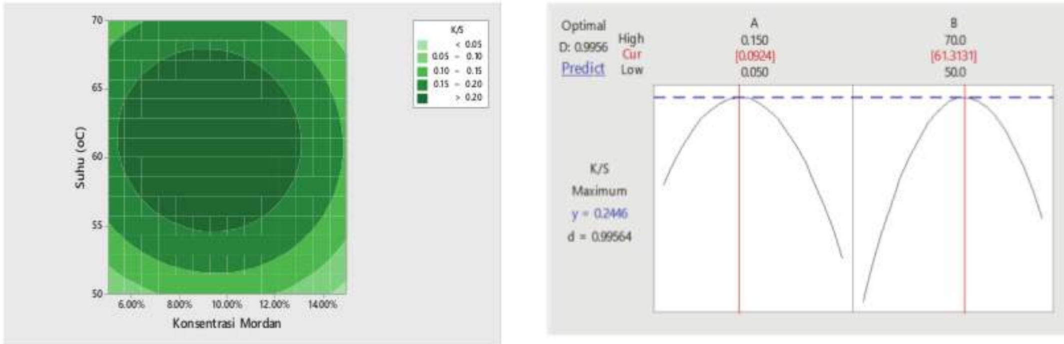
The colour strength value of K/S is obtained by Equation (1) of each independent variable based on design of experiment. Data are shown in Table 3.

TABLE 3. Colour strength (K/S) value using alum as mordant

Concentration of Mordant (A)	Dyeing Temperature (B)	%R	K/S
5%	50	70.93	0.059
		75.63	0.039
	60	51.03	0.234
		53.69	0.199
	70	62.08	0.115
		68.03	0.075
10%	50	64.68	0.096
		63.36	0.105
	60	51.58	0.227
		50.30	0.245
	70	51.58	0.227
		58.53	0.146
15%	50	73.85	0.046
		72.16	0.053
	60	57.65	0.155
		66.71	0.083
	70	68.91	0.070
		71.43	0.057

Optimization was carried out by Response Surface Methodology (RSM) with optimum results obtained at the concentration of alum 9.24% on fabric weight and temperature of 61°C with the maximum K/S value was 0.2446. The model equation is presented in Equation 3 with coefficient of determination R^2 value at 83.91%. Contour plot and optimization of K/S values with alum are shown in Fig. 3.

$$K/S_{alum} = -2.662 + 1.16\,A + 0.0916\,B - 5.12\,A^2 - 0.000757\,B^2 - 0.0053\,AB \quad (3)$$



(a) (b)

FIGURE 3. (a) Contour showing the effects of independent variables on the K/S, (b) Optimization of K/S value on the effect of dyeing temperature and mordant alum concentration

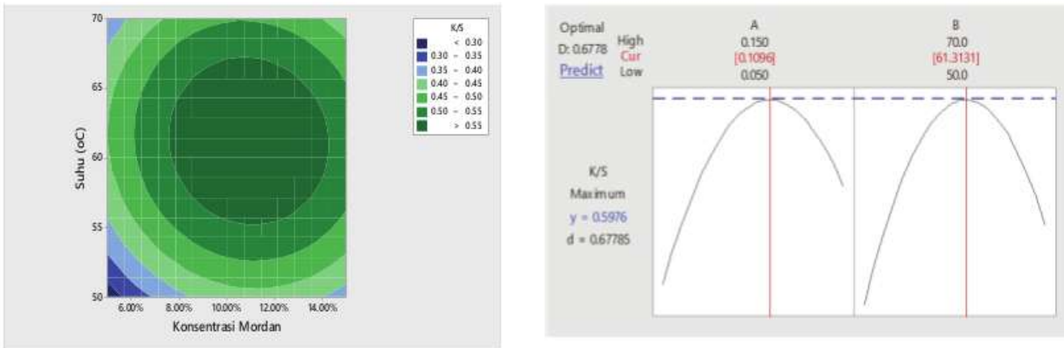
Colour Strength Value Optimization using Ferrous Sulphate as Mordant

The colour strength value of K/S is obtained by Equation (1) of each independent variable based on design of experiment. Data are shown in Table 4. Optimization was carried out by Response Surface Methodology (RSM) with optimum result obtained at the concentration of ferrous sulphate 10.96% on fabric weight and temperature of 61°C with the maximum K/S value was 0.597. The model equation is presented in Equation (4) with coefficient of determination R^2 value at 73.14%. Contour plot and optimization of K/S values with ferrous sulphate are shown in Fig. 4.

$$K/S_{Fe} = -5.00 + 10.83\ A + 0.1636\ B - 42.5\ A^2 - 0.001314\ B^2 - 0.0250\ AB \quad (4)$$

TABLE 4. Colour strength (K/S) value using ferrous sulphate as mordant

Concentration of Mordant (A)	Dyeing Temperature (B)	%R	K/S
5%	50	48.44	0.274
		50.52	0.242
	60	38.63	0.487
		42.29	0.393
	70	44.03	0.355
		42.57	0.387
10%	50	39.88	0.453
		41.71	0.407
	60	31.03	0.766
		39.12	0.473
	70	39.80	0.455
		39.30	0.468
15%	50	43.08	0.376
		43.11	0.375
	60	36.57	0.550
		39.87	0.453
	70	39.89	0.452
		40.99	0.424



(a) (b)

FIGURE 4. (a) Contour showing the effects of independent variables on the K/S, (b) Optimization of K/S value on the effect of dyeing temperature and mordant ferrous sulphate concentration

Colour Strength Value Optimization using Calcium Oxide as Mordant

The colour strength value of K/S is obtained by Equation (1) of each independent variable based on design of experiment. Data are shown in Table 4. Optimization was carried out by Response Surface Methodology (RSM) with optimum results obtained at the concentration of calcium oxide 8.13% on fabric weight and temperature of 60°C with the maximum K/S value was 0.1446. The model equation is presented in Equation (5) with coefficient of determination R^2 value at 75.43%. Contour plot and optimization of K/S values with calcium oxide are shown in Fig. 5.

$$K/S_{Ca} = -2.662 + 1.16\,A + 0.0916\,B - 5.12\,A^2 - 0.000757\,B^2 - 0.0053\,AB \quad (5)$$

TABLE 5. Colour strength (K/S) value using calcium oxide as mordant

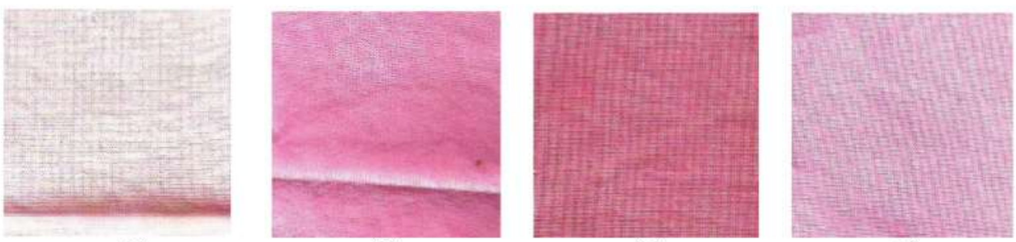
Concentration of Mordant (A)	Dyeing Temperature (B)	%R	K/S
5%	50	69.73	0.065
		70.70	0.060
	60	54.36	0.191
		65.49	0.090
	70	67.70	0.077
		73.64	0.047
10%	50	75.75	0.038
		74.39	0.044
	60	59.67	0.136
		64.73	0.096
	70	65.12	0.093
		69.73	0.065
15%	50	72.39	0.052
		72.18	0.053
	60	59.67	0.136
		64.73	0.096
	70	75.61	0.039
		69.90	0.064

(a) (b)

FIGURE 5. (a) Contour showing the effects of independent variables on the K/S, (b) Optimization of K/S value on the effect of dyeing temperature and mordant calcium oxide concentration

Effect of Type and Concentration of Mordant and Dyeing Temperature on Colour Strength (K/S) Value

Different types of mordant produces different colour shade on cotton dyed with natural dyes from noja leaf extract which shown in Fig. 6. Ferrous sulphate mordant gives darker colouring shade than the original colour without mordant. While alum produce the original colour of the noja leaves extract, and calcium oxide gives a lighter shade colour than the original colour without mordant.



(a) (b) (c) (d)

FIGURE 6. Dyed samples of cotton fabric with noja leaf extract (a) Unmordanted, (b) Alum, (c) Ferrous sulphate, and (d) Calcium Oxide

The colour strength value (K/S) from the un-mordanted dyed sample is 0.1148. In the mordanting method, addition of mordants increase colour strength value (K/S), both with the addition of alum, ferrous sulphate, and calcium oxide. Ferrous sulphate as mordant give results of increasing K/S value significantly, K/S value found to be 0.597. Alum as mordant gives colour strength value (K/S) of 0.2446 and calcium oxide as mordant gives K/S value of 0.1446. The colour strength values (K/S) are shown in Fig. 7. This is in accordance with the research using extracts from *Morinda lucida* plants also showing that mordant is better than mordant alum and calcium oxide [10]. In dyeing of cotton fabric with noja leaf extract, the order of colour strength is found to be ferrous sulphate > alum > calcium oxide.

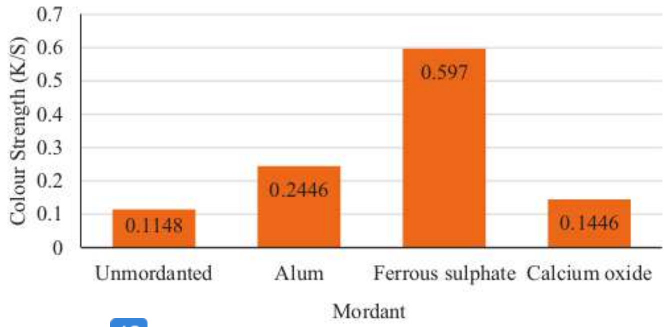


FIGURE 7. Colour strength (K/S) value of dyed cotton fabric with noja leaf

Increased colour strength values (K/S) due to mordanting reveals that phenoxazine compounds are able to form complex compounds with metals. In the mordanting process, there is an ion bond between the metal mordant and cotton fabric. In the dyeing process, the metal complex then binds to natural dyestuff compounds, perisbivalvine A and peristrophine, thus forming bonds between natural dye molecules and cotton fabric. If the dyeing occurs on an unmordanted cotton fabric, there is only one bond between the natural dye molecule and one site on the cotton fabric. However, one mordant molecule can bind two or more ionic bonds with natural dye molecule. Therefore, using mordant will increase colour strength [11].

Ferrous sulphate gives higher colour strength values (K/S) than alum and calcium oxide. Iron salts have a good ability to form coordination complex with natural dyes and give darker shade on fabric. Iron has coordination number of 6, so when iron ions bind to the fabric there are unoccupied sites when they interact with fabric. This empty site then filled with natural dye molecules. This leads to higher values of colour strength and darker shade on fabrics. Possible structure of coordination complex between ferrous sulphate and natural dye from noja leaf is presented in Fig. 8. On the other hand, alum and calcium oxide, with aluminium and calcium metals form weak complex coordination bonds to the fabric, but strong against natural dye molecules. They block the dye and reduce interaction with cotton cloth. This causes lower colour strength values in case of alum and calcium oxide compared to ferrous sulphate [12] .

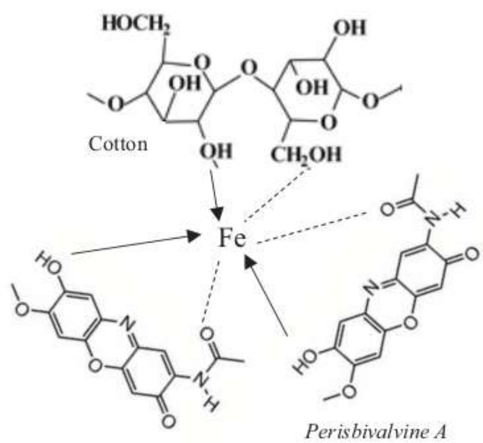


FIGURE 8. Possible structure of Perisbivalvine A with iron (Fe) on cotton

The concentration of mordant will affect the amount of dye attached to the fabric. The higher the concentration of mordant, the more even the mordant will spread on the fabric and provide sufficient bond bridges to the fabric so the amount of dyes absorbed in the fabric will be higher.

This also applies to colour strength, the higher the concentration of mordant, the better the colour produced. However, the results showed that there was a decrease in the K/S value after 10% mordant concentration.

The optimum dyeing temperature was obtained at 61°C. Temperature is very important in the fabric dyeing process. Temperature has a different effect on produced fabric. In maddar plants, the colouring results will get better if the temperature gets higher, while indigo dye is good at room temperature [8]. Natural dyes are very sensitive to high temperatures. There is a possibility that phenoxazine from noja leaf is damaged in high temperature, this will cause the colour strength value decreasing on high temperature.

Colour Fastness Result

The results of colour fastness to sunlight, washing, and rubbing of cotton fabric dyed with noja leaf dye were shown in Table 6. The value ranging from 1 (worst rating) to 5 (excellent rating). Unmordanted fabrics dyed with natural dyes from noja leaf have poor sunlight fastness and washing with a value of 1. The use of mordant in dyeing the fabric with natural dyes from the noja leaf increase the fastness value, both from sunlight, washing, and rubbing. The best fastness is obtained with the use of ferrous sulphate within 2-3 rating sunlight fastness value, sufficient washing fastness value, and good fastness to rubbing value. All of the washing fastness result are increased from 3 to 4-5. So, it can be said that overall ratings of colour change are good.

TABLE 6. Sunlight, washing, and rubbing fastness of dyed cotton fabric with noja leaf

Mordant	Colour Fastness		
	Sunlight	Washing	Rubbing
Unmordanted	1	1	3
Alum	2	2	4 – 5
Ferrous sulphate	2 – 3	2 – 3	4 – 5
Calcium oxide	2	2	4 – 5

CONCLUSIONS

The best colour strength value of K/S from the fabric dyed with natural dyes from the noja leaves was obtained with ferrous sulphate, followed by alum and calcium oxide. The optimum condition of dyeing cotton fabric with noja leaf extract was reached at 61°C using ferrous sulphate as mordant with concentration 10.96% resulting in colour strength value (K/S) of 0.597. The results of sunlight, washing, and rubbing fastness tests showed that the use of mordant in the dyeing cotton fabrics with natural dyes noja leaf gave a significant increase. The best fastness test results were obtained with ferrous sulphate. Natural dyes from noja leaf are quite potential as a source of natural dye for textiles.

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