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Extraction and application of coloring agent of shrubby morning glory leaves (*Ipomoea carnea* subsp. *fistulosa*) as dye in batik

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Abstract. *Zulfiyah, Anggarwulan E, Sari SLA. 2015. Extraction and application of coloring agent of shrubby morning glory leaves (*Ipomoea carnea *subsp.* fistulosa*) as dye in batik Nusantara Bioscience 7: 160-165*. Natural dye is natural materials whose production is influenced by environmental factor. Natural material content affects the quality and intensity of products' color. Shrubby morning glory (*Ipomoea carnea* Jacq. subsp. *fistulosa* (Mart. ex Choisy) D.F. Austin) has potency as a natural dye since it contains high tannin. The objective of the research is to determine the difference of tannin contents in the extract of shrubby morning glory leaves grow in field or rice field, and also the quality of batik's color by using dyes extracted from these leaves using fixators iron sulphate ($FeSO_4$), alum ($Al_2(SO_4)_3$), and calcium oxide ($CaCO_3$). Soil conditions observed are soil texture, pH, nitrogen content (N), organic materials content, C-organic content. Data of the soil quality are analyzed descriptively. The result of fastness test is analyzed by Kruskal Wallis test and continued by Mann Whitney test with the significance rate of 95%. Data of tannin level are analyzed by t test-independent. The result of the study shows that the leaves of shrubby morning glory from rice-field contain high tannin, which is 1.52%. This type of soil is more fertile than field soil, with better soil texture, pH, N level, organic level, and C-organic. The fastness test shows that using extract shrubby morning glory dye which contains higher tannin is better. Batik's dyeing using the extract of the plants growing in rice field with fixator of sulfate iron ($FeSO_4$) has the best fastness.

Keywords: Batik, fixator, *Ipomoea carnea* subsp. *fistulosa*, natural dye

INTRODUCTION

Dyeing process of batik industry nowadays tends to use types of synthetic dyes which are easily obtained and applied with some color variations and relatively high brightness. Producers still ignore the dangers arising from the use of those synthetic dyes, like environmental pollution and carcinogenic property which cause allergic to the skin which will develop into skin cancer (Wilujeng et al. 2010; Kulkarni et al. 2011).

Some of the advantages of natural dye which can balance synthetic dyes are *biodegradable* means environmentally-friendly; its waste can be used as fertilizers for plants. Natural dye is not poisonous, doesn't cause allergic to skin, and can be found around the neighborhood which makes it cheaper, even with no cost at all (Kulkarni et al. 2011; Kumaresan et al. 2011).

One of the other natural resources which have potency as natural dye is shrubby morning glory or *kangkungan* (*Ipomoea carnea* Jacq. subsp. *fistulosa* (Mart. ex Choisy) D.F. Austin). According to Irvianti (2004), tannin is one of the chemical substances of shrubby morning glory leaves. Tannin can be used as natural dye because it produces brown color on fabrics (Kwartiningsih et al. 2009). According to Maryana (2012), natural dye doesn't always have the same coloring. It is caused that natural dye is strongly affected by the place in which it grows, and the effect resulted is in the form of color brightness.

The color fastness to the washing plays an important role as the determiner of textile quality. To make natural coloring result has good fastness, it is necessary to hold fixation process. Fixation process can function as color director and improve colorfastness of the fiber (Ruwana 2008). Fixator is a substance which can be used to fixate. Some fixators which are often used are ferrous sulfate ($FeSO_4$), alum ($Al_2(SO_4)_3$, and calcium oxide ($CaCO_3$).

This study is intended to do a process of natural dye extraction of shrubby morning glory leaves from different growing locations to learn the effects of different growing locations to the level of tannin extract, and to do a test of batik's quality (color fastness to the washing), the result of natural dye coloring of shrubby morning glory leaves with fixator variations of ferrous sulfate, alum, and calcium oxide.

MATERIALS AND METHODS

Plant materials

The materials used in this study cover: shrubby morning glory leaves taken from two places, from the field area in Ngoresan, Surakarta and from rice field area in Palur, Karanganyar, all is from Central Java Province, Indonesia. Some other materials needed are cotton fabric which has been given batik motive, alum ($Al_2(SO_4)_3$, calcium oxide ($CaCO_3$), and ferrous sulfate ($FeSO_4$), and TRO (Turkish Red Oil) Solution.

Research design

This study uses Completely Randomized Design of factorial pattern with two factors. The first factor is the type of location where shrubby morning glory leaves are taken which consists of 2 levels, as follows:

P_1 = field location

P_2 = rice field location

The second factor is a type of fixator which consists of three levels, as follows:

F_1 = alum fixator ($Al_2(SO_4)_3$

F_2 = calcium oxide fixator ($CaCO_3$)

F_3 = ferrous sulfate fixator ($FeSO_4$)

Each treatment is given 3 repetitions. The treatment combinations obtained are as follows:

Types of location	Types of fixator		
	F_1	F_2	F_3
P_1	P_1F_1	P_1F_2	P_1F_3
P_2	P_2F_1	P_2F_2	P_2F_3

Procedures

Dye extraction

A total of 500 g shrubby morning glory leaves is boiled into 5 liters of water. The boiling is done until the volume becomes 2.5 liters. Next, the solution of extraction result is filtered. After being cool down, the solution is ready to use (Fitrihana 2007).

Dye application

Dye application is preceded by mordanting process by dissolving 2 g TRO in 1 liter of water. Then, the cloth is soaked for 2 hours. After that, it is washed and wind-dried. Next, put the cloth into the natural dye and soaked for 30 minutes. Drying is done in the shade (dipping process). After the cloth was colored, fixation process is carried out by dissolving 50g of fixation materials (alum, calcium oxide, and ferrous sulfate) into 1 liter of water, and take the clear solution. The cloth then dipped into fixator solution. After being dry, the cloth is boiled in the boiling water to remove the wax in the cloth (Java: *nglorod*). After being wind-dried, the cloth is ready to be tested its fastness (Fitrihana 2007).

Tannin test

The test is preceded by the preparation of standard solution (STA solution*)*. Next, 10 g shrubby morning glory leaves extract is heated for 30 minutes using reflux. A total of 0.5 mL extract is put and added by Na_2CO_3 0.5 mL, folin 0.25 mL and aquadest 3.75 mL, the solution is then vortexed and read its absorbance in λ 725 nm (Makkar et al. 1993).

Soil quality test

Soil quality test covers soil texture, nitrogen content (N), and acidity level (pH), organic material level, and soil C-organic. Soil texture test, nitrogen content (N), and acidity level (pH) are carried out based on the method of Soil Research Institute (2009), while the measurement of organic material and C-organic contents are carried out using Walkley dan Black method (Page 1982).

Coloring agent (dye) quality test

The test of color fastness level is carried out in Laboratory of Commodity Test of Craft and Batik Industry Yogyakarta, Indonesia. It is used two types of scales in the process of measurement, namely *Gray Scale* and *Staining Scale*. Table 1 is the value level in measurement stated in CD (*Color Difference*) (Ruwana 2008).

Data analysis

The data of soil quality are analyzed descriptively. The data of tannin level are analyzed by *t-test* independent, while the result of color fastness is analyzed using Kruskal Wallis (1952). If $p<0.05$, it can be continued by advanced test using Mann and Whitney (1947) test to compare two treatments with significance rate of 95%.

RESULTS AND DISCUSSION

Soil quality and tannin level of shrubby morning glory leaves extract

Shrubby morning glory leaves used are taken from two different locations which have the same climate condition. The first location is a field area in Ngoresan, Surakarta City and the second location is a rice field area in Palur, Karanganyar. Both of them in Central Java Province, Indonesia. The soil condition observed covers: soil texture, nitrogen content (N), organic materials content, and carbon content (C-organic).

Based on the result of soil texture test, rice field location has the texture which can support higher soil fertility (Table 2). The comparison of dust fraction in rice field area is higher (51.00%), also on the fraction of clay (31.73%). Field area has higher sand content of 65.23%. Clay fraction has the ability to keep more water and plant's nutrient. Generally, essential nutrients are mostly found in dust fraction, therefore, the soil which has higher clay and dust texture has better fertility compared to the soil with higher sand fraction.

Nitrogen is the main macronutrient needed by plants in a high amount. The result of the study shows that the soil of the rice field area has more N level of 0.19% compared to the soil of the field area which has N level of 0.17%.

Table 1. Evaluation scoring of color fastness and color staining

Score	Color difference (in CD)		Note
	Gray scale	Staining scale	
5	0	0.0	No fastness/staining
4-5	0.8	2.0	Almost no fastness/staining
4	1.5	4.0	Very little fastness/staining
3-4	2.1	5.6	Little fastness/staining
3	3.0	8.0	Fastness/staining exists
2-3	4.2	11.3	Enough fastness/staining
2	6.0	16.0	Pretty much fastness/staining
1-2	8.2	22.6	Much fastness/staining
1	12.0	32.6	Very much fastness/staining

Note: CD = Color Different (Ruwana 2008)

Table 2. The result of soil testing

Location	Soil Texture			N	pH	Organic material	C organic
	% dust	% clay	% sand	%		%	%
Field	23.18	11.59	65.23	0.17	6.06	2.46	1,43
Rice field	51.00	31.73	17.27	0.19	7.14	2.92	1,69

Table 3. Acidity level (pH) of the soil (Hardjowigono 1987)

pH	Very acidic	Acidic	Little acidic	Neutral	Little alkalis	Alkalis
	< 4.5	4.5-5.5	5.6-6.5	6.6-7.5	7.6-8.5	> 8.5

Table 4. The level of C-organic of the soil

Organic material	Very low	Low	Medium	High	Very High
	< 1.00	1.00-2.00	2.01-3.00	3.01-5.00	> 5.00

Table 5. The average of tannin level on shrubby morning glory leaves extract from different location

Location	Tannin level (%)
Field	1.14
Rice field	1.52

Nitrogen plays an important role in stimulating vegetative growth of the plant, making the plants leaves dark green and it is also the composer of cell plasma (Fauzi 2008). The element of nitrogen is also able to role as composers of many essential compounds like protein, amino acid, amide, nucleate acid, nucleotide, coenzyme, and many important compounds for metabolism (Marschner 1986). Therefore, the improvement of nitrogen level on plants can influence in the forming of tannin compound of shrubby morning glory plant.

The effect of pH is pretty high on the availability of nutrients in the soil. According to Sarief (1997), soil pH affects the exchange capacity value of cation, base saturation, attachments of P element, and also affects microorganisms' development.

Based on the level of soil acidity on Table 3, field soil belongs to the type of soil which is a little acidic with pH value of 6.06. Meanwhile, rice field soil belongs to neutral condition, with pH value of 7.14. It is found that rice field soil has higher fertility than field soil.

Organic materials affect the soil's fertility especially of the soil's nutrients (Stevenson 1982). The level of organic materials of the sample from field soil is 2.46%, and from the sample of rice field soil is 2.92%. Based on the results, it is found that rice field soil has higher support in the productivity of shrubby morning glory plants.

Based on the level of soil's C-organic content done by Hardjowigono (1987), C-organic level on the result of the test belongs to low category (Table 4). The field soil is 1.43% and the rice field soil is 1.69%. It is found that rice field soil has higher level of fertility than field soil.

Based on the study, it is found that the content of tannin extract of shrubby morning glory leaves taken from rice field location is higher than the leaves extract taken from field location (Table 5). The result of *t-test independent* shows significant value of (2-*tailed*) < α, which is 0.031< 0.05. It shows that there's tannin level difference in the growing location of field and rice field. Based on the result, it is found that tannin level which is polyphenol in shrubby morning glory plants is higher in amount on more fertile soil.

The variations of tannin concentration in the plants' tissue have been proven to be depended on many factors, including types of plants, parts of plants, plants' maturity, planting time, and soil fertility (Hassanpour et al. 2011). Mudau et al (2007) research stated that polyphenol content contained in *Athrixia phylicoides* L. is in line with the treatment of nitrogen fertilizer. The concentration improvement of nitrogen fertilizer will increase polyphenol level. Venkatesan et al. (2004) reported that the use of nitrogen in tea plants can increase polyphenol content. Based on the research, it is found that *Carbon Nutrition Balance* hypothesis can't be applied on polyphenol content of *Athrixia phylicoides* L plants, tea plants (*Camellia sinensis* L.), and shrubby morning glory plants. CNB hypothesis stated that the improvement of the amount of carbon to form defending element occurs when the plants are in the condition of less nutrition and minerals (Zhang et al. 2009).

The quality of batik as the result of dyeing

This study is carried out in the variation of fixator types on each batik cloth which is dyed with the extract of shrubby morning glory leaves taken from different growing location. Besides increasing colorfastness, fixation process also functions to direct color and generate color after being dyed. Fixators used in this study are ferrous sulfate ($FeSO_4$), alum ($Al_2(SO_4)_3$, and calcium oxide ($CaCO_3$).

Cotton fiber or cellulose arranged in *Primisima* cotton cloth consists of glucose straight polymer. Glucose which is placed by turns in water marination will expand pretty high, that the pores can be added by coloring agent, and have many groups of OH where O is electro-negative and H is low electro-positive (Suheryanto 2010). The bonds between tannin and fibers are in the form of covalent bond, ionic bond, hydrogen bond, and van der Walls bond. These bonds make fibers can be colored permanently.

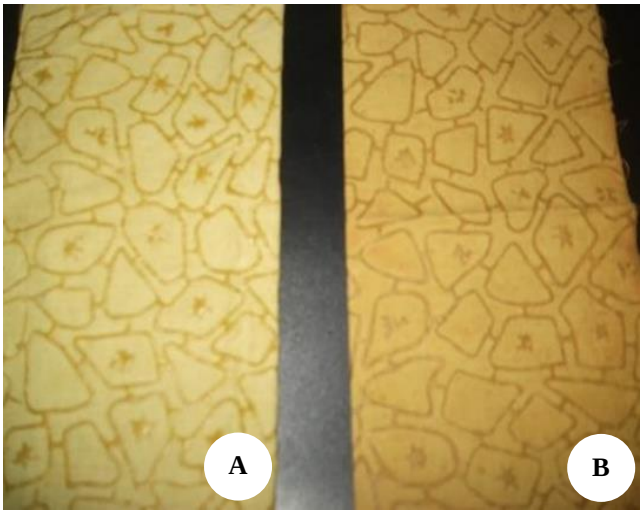

Figure 1. The result of cloth coloring before fixation. A. P1 = field location, B. P2 = rice field location

Figure 2. Batik cloth resulted from fixation. Note: A. P_1F_1: field location, alum fixator B. P_1F_2: field location, calcium oxide fixator, C. P_1F_3: field location, ferrous sulfate fixator D. P_2F_1: rice field location, alum fixator, E. P_2F_2: rice field location, calcium oxide fixator, F. P_2F_3: rice field location, ferrous sulfate fixator.

Table 6. The average result of color change based on grey scale

Location	Fixator	Color change (*Grey scale*)
Field	Alum	4.2
Field	Calcium oxide	3.0
Field	Ferrous sulfate	2.1
Rice field	Alum	3.0
Rice field	Calcium oxide	2.1
Rice field	Ferrous sulfate	1.5

The dipping result of shrubby morning glory leaves extract taken from field and rice field location has different color sharpness (Figure 1). The more natural dye (tannin) contained the more coloring agent bond to the fabrics. After fixation process is carried out, batik cloth which is dyed with the extract from rice field location has more sharp color in all aspects (Figure 2). Therefore, the fabrics resulted from the dye of shrubby morning glory leaves extract from rice field area has higher color sharpness.

The fastness test to washing is measured based on 2 standards, namely *Grey scale* to determine cloth's fastness, and *staining scale* to determine the level of staining on other fabrics. The average result of color change based on grey scale can be seen on Table 6.

The result of statistic test using Kruskal Wallis test with significance rate of 95% to colorfastness based on the standard of Grey scale found that the significant value of 0.004 means lower than 0.05. It shows that there is a difference in color change in the variation of shrubby morning glory growing area and fixator variation. The test is then continued by Mann Whitney test to see the difference among treatments. The result of Mann Whitney shows that P_1F_2 treatment (field location, calcium oxide fixator) >< P_2F_1 (rice field location, alum fixator) and P_1F_3 (field location, ferrous sulfate fixator) >< P_2F_2 (rice field location, calcium oxide fixator) don't have a significant fastness difference. It is shown by the value of α for 1.000 ($\alpha > 0.005$).

Staining scale shows how high the cloth staining is from shrubby morning glory leaves appeared in various types of textile. Meanwhile, the types of fabrics used in staining scale measurement are acetate, cotton, polyamide, polyester, acrylate, and wool (Table 7). Based on Table 7, acetate cloth, acrylate, and wool have the same staining of 2.0 that the statistical test Kruskal Wallis is just carried out to cotton, polyamide, and polyester. The result of Kruskal Wallis test to those fabrics is obtained each α of 0.004 ($\alpha < 0.05$). It shows that there's a difference of color change in the location variation of the growth of shrubby morning glory and fixator variation. The test is then continued by Mann Whitney test to see the difference among treatments (Table 8).

The result of color fastness measured by Grey scale and staining scale treatments on Tables 6 and 7 can be taken the average to determine the location where the leaves are taken which have the lowest color change value. The lower the average of the color change obtained, the higher the fastness level of fabric to washing process.

Based on the average value of color fastness measured using grey scale and staining scale (Table 9), it is found that the cloth with the extract of shrubby morning glory leaves taken from rice field area has better fastness based on Grey scale measurement, where the average of the color change obtained is 2.2. The average result of color change using staining scale also shows that cloth extracted by shrubby morning glory leaves taken from rice field area has lower staining value compared to field area.

Table 7. The average result of color change based on the standard of staining scale

Location	Fixator	Color change (staining scale)					
		Acetate	Cotton	Polyamide	Polyester	Acrylate	Wool
Field	Alum	2.0	4.0	4.0	4.0	2.0	2.0
Field	Calcium oxide	2.0	2.0	4.0	4.0	2.0	2.0
Field	Ferrous sulfate	2.0	4.0	4.0	4.0	2.0	2.0
Rice field	Alum	2.0	4.0	4.0	5.6	2.0	2.0
Rice field	Calcium oxide	2.0	4.0	4.0	4.0	2.0	2.0
Rice field	Ferrous sulfate	2.0	2.0	2.0	4.0	2.0	2.0

Table 9. The average result of color change value ofgGrey scale and staining scale

Location	Color change (Grey scale)	Staining scale					
		Acetate	Cotton	Polyamide	Polyester	Acrylate	Wool
Fields	3.1	2.0	3.3	4.0	4.0	2.0	2.0
Rice fields	2.2	2.0	3.3	3.3	3.3	2.0	2.0

Table 8. The test result of Mann Whitney. Data on color change based on the standard of staining scale to cotton, polyamide, and polyester

The difference of color change	Sig. value of cotton	Sig. value of polyamide	Sig. value of polyester
P_1F_1 >< P_1F_2	0.025	1.000	1.000
P_1F_1 >< P_1F_3	1.000	1.000	1.000
P_1F_1 >< P_2F_1	1.000	1.000	0.025
P_1F_1 >< P_2F_2	1.000	1.000	1.000
P_1F_1 >< P_2F_3	0.025	0.025	1.000
P_1F_2 >< P_1F_3	0.025	1.000	1.000
P_1F_2 >< P_2F_1	0.025	1.000	0.025
P_1F_2 >< P_2F_2	0.025	1.000	1.000
P_1F_2 >< P_2F_3	0.025	0.025	1.000
P_1F_2 >< P_2F_1	1.000	1.000	0.025
P_1F_3 >< P_2F_2	1.000	1.000	1.000
P_1F_3 >< P_2F_3	0.025	0.025	1.000
P_1F_3 >< P_2F_3	1.000	1.000	1.000
P_2F_1 >< P_2F_3	0.025	0.025	1.000
P_2F_2 >< P_2F_3	0.025	0.025	1.000

Note: sig. value <0.05 shows that there is significant difference among treatments. P_1F_1 : field location, alum fixator, P_1F_2 : field location, calcium oxide fixator, P_1F_3 : field location, ferrous sulfate fixator, P_2F_1 : rice field location, alum fixator, P_2F_2 : rice field location, calcium oxide fixator, P_2F_3 : rice field location, ferrous sulfate fixator.

From the result of fastness quality test based on grey scale (Table 6) and staining scale (Table 7), it is found that the treatment of shrubby morning glory leaves extract from rice field area with ferrous sulfate fixator (P_2F_3) is the best combination. The value of color change in standard measurement of Grey scale shows P_2F_3 treatment has the lowest value of color's difference. It also occurs on the measurement using staining scale, especially seen from the staining test result of polyamide cloth.

P_2F_3 treatment becomes the best fastness treatment because tannin element has hydroxyl group as polar group which can ionize in water medium and makes tannin becoming reactive. When a metal is added (in the form of fixator), it will form ionic bond with hydroxyl group from tannin (Ismininingsih 1979). Therefore, coloring agent (dye) and fixator will form complex bond which is formed by fixator of metal ion and dye. One electron of fixator of metal iron will bond ionically with the dye, and one more electron will bond ionically with cotton molecule.

Ferrous sulfate fixator ($FeSO_4$) will ionize into ion Fe^{3+} and SO_4^{2-}. Iron (Fe) is transition element which has metal character as in all other transition elements. This metal character is affected by the element's ease to release valence electron. The existence of electron in block D which hasn't been full yet causes a stronger metal bond compared to the main group of element, including Al (alum fixator ($Al_2(SO_4)_3$) and Ca (calcium oxide fixator ($CaCO_3$) (Riyana, pers. comm. 2012). Therefore, alum fixator has lower color change and stronger fastness.

In conclusion, the difference of location where shrubby morning glory is taken affects tannin element level contained in the extract of shrubby morning glory leaves. Tannin content is found more in the extract of shrubby morning glory leaves growing in rice field area. Batik quality as a result of coloring using shrubby morning glory leaves extract from rice field location produces better fastness. The best fastness is shown in shrubby morning glory leaf extract from rice field by using Ferrous sulfate as fixator.

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