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by Adhi Kusumastuti

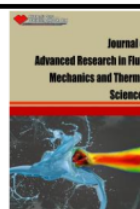
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Utilization of Ash from Thermal Decomposition of Rice Straw as a Natural Dye

Adhi Kusumastuti^{1,*}, Miftahiyah Nuru'Udhma Hidayati¹

¹ Faculty of Engineering Universitas Negeri Semarang, Kampus UNNES Sekaran, Gunungpati, 50229 Semarang, Indonesia

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ABSTRACT

Indonesia is among the largest countries in global rice production. Annual generation of rice straw of about 20 million tons were reported. It is therefore, utilization of rice straw as natural dye is considered to minimize pollutant problem. Rice straw composed of 48.7% carbon that generate black color. In this research, natural dyes of rice straw ash were applied in cotton and rayon fabrics, followed by mordanting using lime, alum, and ferrous sulphate. The color intensity and color fastness of cotton and rayon fabrics dyed using rice straw ash extract was investigated. It was found that the system using ferrous sulphate mordant produced the lightest color; alum mordant treatment produced a darker color while the darkest color was provided by system using lime as mordant.

1. Introduction

Color is among the most important character in textile and fashion. Commercially, synthetic dyes are applied to generate various colors at high intensity and color fastness. Synthetic dyes are also easily and quickly applicable at relatively low costs. However, continuous utilization of synthetic dyes triggers negative impact to water body. This is due to the fact that in Pekalongan, well-known as batik city, only about 0.6% of batik industries provide own installation of wastewater treatments [1]. Direct disposal of untreated wastewater of dyeing process is a common practice. It was reported that annually, worldwide textile industries involve about 1.3 million tons of dyes, pigments, and dye precursors which worth around \$23 billion [2]. It implies the major application of synthetic dyes in the textile processing. The generation of toxic and hazardous chemical wastes may impose environmental pollution as well as health problems. Some studies on the removal of synthetic dyes have also been reported [3-5].

The raising of environmental concern, enhance the utilization of natural dyes as the alternative of synthetic dyes. The exhibition of higher biodegradability as well as environmental compatibility strengthens the potential of natural dyes for textile application. Natural resources offer the existence of natural dyes from plant, animal, mineral, and microbial dyes. Natural dyes are applicable in dyeing

* Corresponding author.

E-mail address: adhi_kusumastuti@mail.unnes.ac.id

process of almost all types of natural fibers. As tropical country, Indonesia provides abundant types of natural resources having potential to be applied as natural dyes. The dyes contain coloring matters having affinity to natural fiber. To ensure the sustainability of textile production, the material must be available in large quantities throughout the year. The easiness of handling and storing, the high dyestuff content as well as the easiness of water extraction are the basic requirements of natural dyes selection [6]. It is therefore, the application of low-utilize materials is recommended for textile dyes. Previous studies regarding the exploration of weeds, invasive plants, or unused plant parts have been carried out [7-9]. The studies revealed the favorable prospective of natural resources as textile dyes at various colors.

Rice is a food crop commodity that is widely cultivated in Indonesia. Nevertheless, it is among the most important agricultural products. This is due to rice (*Oryza sativa*) has become the staple food for almost all Indonesians. It was reported that rice production in 2020 was 54.65 million tons of milled dry grain showed an increase of 45.17 thousand tons or 0.08% when compared to 2019 which was 54.60 million tons of milled dry grain. Rice straws are side products generated in significant quantities on a global basis. The minimum utilization of rice straw releases another problem of waste thus causing environmental pollution. To date, insufficient effective waste management are applied to the large quantity of rice straw. After harvesting, the rice straw is only burnt by 75–80% of farmers. The utilization of rice straw in Indonesia is still limited to compost and organic feed while the energy sector only processes about 20% of straw waste for bioethanol production whereas the utilization in creative industry is relatively very low [10]. Hidalgo *et al.*, [11] reported the annual production of rice straw was 67 million tons worldwide.

Rice straw consists of ash of about 14.65% [12] while Van Hung *et al.*, [13] reported ash content of about 18.67% in their study. Carbon content of 38.24% in the rice straw was also revealed by Van Hung *et al.*, [13]. Considered as a lingo-cellulosic biomass, rice straw is composed of 38% cellulose, 25% hemicellulose, and 12% lignin [14]. As potential coloring matter, the carbon content determines the intensity of the generated black color. Rice straw with higher carbon content will produce colorant with higher black intensity [10]. Comparable carbon content in the range of 11.10% to 16.75% offer the potential of rice straw as natural dyes [13]. Carbon is among the sources of black color [2].

Considering the coloring matter inside rice straw ash as well as the abundantly availability, study on the exertion of rice straw ash as natural dyes was done. Rice straw ash was extracted, the obtained dye extract was applied in the dyeing of pre-mordanted cotton and rayon fabrics. The dye quality was determined using color intensity and color fastness tests.

2. Research Method

Aqueous extraction was used to extract the coloring matter of rice straw ash. Prior to extraction, the raw material is broken into small pieces or powdered to enlarge the surface area. The fine material was then brought to boil and filtered to remove the impurities. Despite the process simplicity, this technique suffers from the possibility of dye decomposition due to the application of high temperature. It is therefore; the requirements of high temperature resistance as well as water solubility are inevitable for this method.

2.1 Materials

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Rice straw ash was used as sample of natural dyes. Deionized water was used for all of the solutions preparation. Commercial cotton and rayon fabrics, sodium carbonate, Turkey red oil (TRO), alum, ferrous sulphate, and lime were purchased in local stores.

2.2 Procedure

TRO was dissolved in the deionized water at concentration of 2 g/L, the obtained solution was then used to soak the fabrics for 24 hours, followed by washing and air drying. Preparation of mordanting solution was carried out by dissolving the mordanting agents, i.e., alum, ferrous sulphate, or lime in the deionized water at the concentration of 50 g/L. The solutions were allowed to settle for 24 hours, the clear solutions were taken for mordanting process. The fabric was soaked in each mordanting solution for 12 hours, and then washed and air dried.

The burnt to ash-rice straw was sifted to separate the impurities and to ensure the evenness size. Ash was diluted into deionized water in the ratio of 1:5. The obtained solution was brought to boil till the volume reached half of the initial volume. Furthermore, the solution was let to settle. The clear solution was taken and filtrated to assure the solution was free of ash. Extract of rice straw ash was then used to soak the fabrics at volume ratio of 1:20 for 15 minutes. The fabric was drained and allowed to dry, then it was soaked back in the dyeing bath, this process was repeated 15 times.

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The color intensity (R %) of the dyed fabric was measured using a spectrophotometer (UV-PC). Color fastness to washing was measured using grey scale. Color fastness to wet rubbing of the dyed fabric was tested using a crock meter and the value was measured using a staining scale.

3. Results and Discussion

3.1 Color Intensity

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The amount of dye absorbed in the material expressed by K/S. The reflectance (% R) of test sample was measured at a wavelength of 400 – 700 nm with an interval of 20 nm to determine the maximum wavelength at the lowest % R value. The lower value of R% generates darker color of fabric, on the contrary, higher value of R% indicates lighter color, towards white. The reflectance value was converted to a color intensity value (K/S) based on the Kubelka-Munk equation, Eq. (1) as follows [15]

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

4
where

K: Coefficient of light absorption

S: Coefficient of light scattering

R: Reflectance (%)

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Study on the color intensity of cotton and rayon fabrics dyed using rice straw ash solution is shown in Table 1. Based on Table 1, it can be observed that each type of mordant generated different color intensity, indicated by reflectance (R %) values that vary insignificantly. Almost all experiments generated color in category of dark, indicated by reflectance (R %) value in the range of 21-40. A variation of ferrous sulphate as mordanting agent with cotton fabric resulted in average color intensity. The lightest color was resulted by cotton fabric given pre mordanting treatment of ferrous sulphate (R% of 47.58), while the darkest color was resulted by rayon fabric given pre mordanting

treatment of lime (R% of 28.16). In all mordanting agent, rayon fabric generated darker color rather than that of cotton fabric. This is related to the moisture content of each fabric. Cotton fabric with lower moisture content (6%) than that of rayon fabric (13%) leads to the lower absorption capacity to natural dye solution.

The K/S value indicates the amount of dye absorbed, shown in Table 1 that this study generated higher K/S value rather than study of Wang *et al.*, [16] about dyeing cotton fabric with anthocyanin dyes extracted from mulberry (*Morus rubra*) fruits. In the dyeing process of cotton fabric using reactive dye, Ramaiah and Ari [17] succeeded in reaching much higher K/S of 49.235. In term of mordanting agents variation, lime mordant generated the darkest color, followed by alum mordant. Ferrous sulphate mordant generated the lightest color in all experiments.

Table 1

| Color intensity | | | |
|-----------------|-------|----------|-------|
| Code | R% | Category | K/S |
| F1M1 | 39.09 | Dark | 18.56 |
| F1M2 | 47.58 | Average | 22.80 |
| F1M3 | 37.11 | Dark | 17.57 |
| F2M1 | 37 | Dark | 17.51 |
| F2M2 | 39.48 | Dark | 18.75 |
| F2M3 | 28.16 | Dark | 13.10 |

F1 = cotton fabric
F2 = rayon fabric
M1 = alum mordant
M2 = ferrous sulphate mordant
M3 = lime mordant

3.2 Color Fastness to Washing

Color fastness test to washing of textile fabrics dyed using natural dyes of rice straw ash extract under various types of mordanting agents were carried out. The value of color fastness to washing was measured based on the value of grey scale in CD units (color difference) between 1-5, where 1 is the lowest value and 5 is the highest value. Grey scale was used to indicate the level of color difference or color contrast in the sample, before and after treatment. The value of color change (grey scale) was considered good by the minimal color change after soap washing. Table 2 describes the results of color fastness test to washing of six samples.

Table 2

| Color fastness to washing | | |
|---------------------------|-----|-----------|
| Code | CD | Category |
| F1M1 | 2.1 | Good |
| F1M2 | 1.5 | Very good |
| F1M3 | 2.1 | Good |
| F2M1 | 2.1 | Good |
| F2M2 | 1.5 | Very good |
| F2M3 | 2.1 | Good |

Based on Table 2, it can be observed that all experiments of the color fastness tests to soap washing revealed the results of good to very good. Both types of fabrics, rayon and cotton, provided the same trend of color fastness to washing. Application of each mordanting agent generated the same results on both fabrics. Ferrous sulphate generated the best color fastness in the value of very

good. Alum and lime generated color fastness in the value of good. This finding was comparable to that of Zarkogianni *et al.*, [18]. They compared the application of some types of mordanting agents in the dyeing of cotton and wool fabrics using some natural dyes. Complex formation of metal salts mordant to colorant and fiber resulted in the insoluble precipitate compound. Natural dyes, having no affinity with textile fiber, could only bond to mordanting agent. Thus, mordanting process could improve color fastness to washing. Similar result was also obtained by Prabhavathi *et al.*, [19] in the utilization of some natural dyes in cotton dyeing.

3.3 Color Fastness to Rubbing

Color fastness to wet rubbing was carried out, the results are presented in Table 3. This process was done to measure the color fastness of dyed fabric towards wet rubbing treatment. The value of color fastness to wet rubbing was determined from the staining scale in CD (color difference) units.

Table 3
Color fastness to rubbing

| Code | CD | Category |
|------|-----|----------|
| F1M1 | 5.6 | Average |
| F1M2 | 6.4 | Average |
| F1M3 | 5.6 | Average |
| F2M1 | 5.6 | Average |
| F2M2 | 5.6 | Average |
| F2M3 | 5.6 | Average |

In Table 3, it can be observed that in all experiment, the results of color fastness test to wet rubbing generated the same results of average. There was no effect of application of different mordanting agents and fabric types to the color fastness to rubbing. In wet condition, textile fiber suffers from strength degradation, it is therefore the color fastness to wet rubbing was in the level of average. Study of Tayyab *et al.*, [20] on application of tangerine extract for lyovell dyeing obtained the same level of wet rubbing fastness. In their research, ferrous sulphate was also utilized as mordanting agent. Zarkogianni *et al.*, [18] revealed that the worse color fastness to wet rubbing was contributed by the superficially adsorbed excess of pigment which could be more easily removed in wet condition. The comparable result was also reached in the color fastness to wet rubbing test.

4. Conclusions

Study on the application of rice straw ash as natural dye was carried out. The research was succeeded in extracting coloring matter of rice straw ash. In term of color intensity, rayon fabric provided better result rather than cotton fabric, while in term of mordanting agent, the color intensity was in the order of ferrous sulphate < alum < lime. In the color fastness to washing test, mordanting agents provided the same trend for both types of fabric. The best color fastness was provided with system using ferrous sulphate as mordanting agent. Performance test of rice straw ash extract as natural dyes was also observed based on color fastness to wet rubbing test. All experiments resulted in average level of wet rubbing fastness.

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