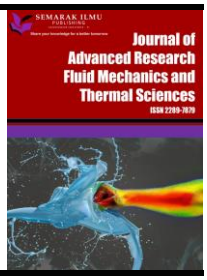
SEMARAK ILMU PUBLISHING

Journal of Advanced Research in Fluid Mechanics and Thermal Sciences

Journal homepage:
https://semarakilmu.com.my/journals/index.php/fluid_mechanics_thermal_sciences/index
ISSN: 2289-7879



Quality Results of Cotton Fabric Coloring with Mangsi (Phyllanthus Reticulatus) Fruit Extract

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ARTICLE INFO

Article history:
Received 15 June 2024
Received in revised form 22 September 2024
Accepted 1 October 2024
Available online 20 October 2024

Keywords:
Natural dyes; mangsi fruit; solvent; color fastness

ABSTRACT

The research was done to analyze the quality of the coloring result of mangsi (phyllanthus reticulatus) fruit extract as natural dyes in cotton fabric dyeing. Extraction method was carried out through maceration process using some solvents namley water, ethanol, acidic ethanol, methanol, and acidic methanol in a ratio of 1:10 with pre-mordanting and fixation using mordant alum, calcium carbonate, and ferrous sulphate. The conducted observations included color direction test, color opacity test and color fastness to soap washing test. The result of color direction was brown to yellow-orange and blue to dark blue. The value of color opacity on cotton fabrics was in the "medium" to "very opaque" color category. The resulting value of color fastness to soap washing test was in the "good" to "poor" category. The dyeing results on cotton fabric generated an even color.

1. Introduction

Natural dyes have been used by people since 3500 BC (BC). Natural dyes can be obtained from plants such as leaves, stems, fruit, flowers, roots, and sap [1]. As technology are developing, synthetic dyes are more widely being used because they are considered cheaper, ready to use, and do not easily fade. More than 700,000 tons of synthetic dyes are produced globally [2]. Excessive use of synthetic dyes causes water pollution and waste disposal problems [3]. Waste resulting from synthetic dyes is categorized as B3 waste (Hazardous and Toxic Materials) [4]. Methylene blue is one of the aniline-based dyes widely used in the textile industry, producing hazardous waste. The treatment of methylene blue is not effective because it requires a relatively high cost [5]. Compared to synthetic dyes which are harmful to the environment, natural dyes have various advantages, including being more environmentally friendly, non-toxic, and producing distinctive and beautiful colors.

In order to support the efforts of reusing natural dyes (back to nature), exploration of plants that can be used as natural dyes is required. One plant that has the potential to be a source of natural dye

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https://doi.org/10.37934/arfmts.122.2.94101

is mangsi (Phyllanthus reticulatus) fruit. Mangsi fruit is a wild plant that habitually grows on vacant land. Mangsi fruit contains anthocyanin pigments which are the source of the orange, red, blue, purple, and black pigments [6]. Currently, mangsi fruit has only been used as a medicinal plant as well as a bonsai plant and its use as a natural dye is still less than optimal. Mangsi fruits that grow wild are cut down and are considered a nuisance because they grow as vines. Up to now, many studies on weeds are focusing on the substances or the methods of eradicating the weeds [7]. Meanwhile, weeds have the potential for fabric dyeing [8].

The processing of natural dyes is carried out by extraction method using solvents. Extraction produces pigment in liquid form which then can be used to dye textile materials. Extraction of dyes in plants is usually done using the boiling method. However, high heat can cause anthocyanin stability and dye resistance to fluctuate and result in damage [9]. So far, people are more aware of water being used as a solvent in the natural dye extraction process. Meanwhile, in reality there are many types of solvents that can be used for extraction. Organic solvents commonly used besides water are ethanol and methanol. Organic solvents produce pigments better than water [10]. The addition of acid to organic solvents, both ethanol and methanol, can increase polarity of the solution.

Natural dyes require auxiliary substances as color generators and binders called mordanting. Apart from mordants, textile materials that are dyed using natural dyes are textile materials resulting from cotton fibers such as cotton, silk, and rayon because they have good absorbency [11]. The washing results are then subjected to chemical testing which aims to see the durability of the fabric based on how long a fabric can or cannot last over a long period of time.

2. Method

Reviewing from the purposes and objectives, this study constitutes as experimental research. The experimental method is a method used in conducting experiments to investigate cause and effect in finding two controllable factors [12]. Data collection process carried out in this study employes laboratory tests. The data analysis technique uses quantitative descriptive analysis. The collected data in the form of single numbers are then described.

The independent variables in this study were certain types of solvent consisting of water, ethanol, acidic ethanol, methanol, and acidic methanol with 1.5% HCl as an acidic mixture and the certain types of mordant, which are alum ($A_{12}(SO_4)_3$) with a concentration of 70g/ℓ, calcium carbonate ($Ca(OH)_2$) with a concentration of 50g/ℓ, and ferrous sulphate ($FeSO_4$) with a concentration of 20g/ℓ. The dependent variables in this study were color direction, color opacity, color fastness to soap washing, and color evenness.

Extraction of mangsi fruits was executed using the solvents with a ratio of 1:10 through the maceration (soaking) method. 100 grams of mangsi fruits were crushed using dry blender then put into a glass jar containing solvents (water, ethanol (96%), acidic ethanol, methanol (99%), and acidic methanol) with 32% HCl, then being diluted in ethanol/methanol with a concentration of 1.5% as an acidic mixture. The extraction was carried out for 5 hours at room temperature. The mordanting techniques used were pre-mordanting and post-mordanting. Pre-mordanting was done to cotton fabric which had been soaked in detergent overnight, boiled for 1 hour then left overnight, rinsed and then being air-dried. The dyeing was carried out for 30 minutes continued with post-mordanting process using each mordant for 10 minutes. The frequency of dyeing process was 2 times.

Color direction and color opacity tests were performed using a spectrophotometer. The color direction test was carried out based on the level of brightness (lightness), color saturation (chroma), and color complexion (hue). The color difference test was conducted using the CIELAB method which then generated L*, a*, b*, and dE*ab values. Meanwhile, the color opacity test was indicated by the

darkness and lightness value of a color. The color fastness to soap washing test was carried out using a standard gray-scale. The gray-scale was used to examine color changes by determining the level of color difference or contrast from the lowest level to the highest level. The gray-scale standard consisted of nine pairs of gray standards. The color evenness test was determined from the average color opacity value and then the coefficient of variation value was calculated. The smaller the value of the variation coefficient of the average color opacity, the more even the color would be.

3. Result and Discussion

This study aims to determine the quality of the dyeing results of mangsi fruit extract applied to cotton fabric based on variations in the type of solvent and mordant. The quality tests in this study include color direction tests, color opacity tests, color fastness tests towards washing, and color evenness tests. The resulting test data is presented in Table 1.

The color direction results in Table 1 show that samples with alum mordant treatment during extraction using water, ethanol, acidic ethanol, methanol, and acidic methanol resulted in brown colors which in their Spanish names are white, bone, malta, ecru, and gurkha. Calcium carbonate mordant treatment during extraction using water, ethanol, acidic ethanol, methanol, and acidic methanol resulted in yellow colors namely medium wood, putty, tan, fallow, and barley corn. Ferrous sulphate mordant treatment during extraction using water, ethanol, acidic ethanol, methanol, and acidic methanol resulted in blue to dark blue colors namely charcoal, karaka, nero, and night rider.

The color direction results indicated that mangsi fruit natural dyes produced different color directions according to the types of solvents used in the extraction process and also types of mordant treatment. This study was in line with the research that the use of solvents in the extraction of anthocyanin from red dragon fruit peel affected the pH value, brightness level (L), redness level (a+), and yellowness level (b+) [13]. It also stated that the ethanol concentration could cause changes in polarity which influenced the extraction results of natural dyes [14]. The extraction of seaweed as natural dyes for cotton and silk fabrics with variations using alum mordant produced a yellowish color, lime mordant treatment produced a reddish color; and ferrous sulphate mordant treatment produced a blue color [15].

Ferrous sulphate mordant had proven to bind color strongly thus the resulting color was darker [7]. The coloring process using alum mordant produced a lighter color compared to ferrous sulphate mordant. In ferrous sulphate, the color binding agent is salt which contains metal Fe^{3+}. Tannins condensed in Fe^{3+} salt solutions will produce a blackish green color [16]. Each method and type of mordant produced different colors. $Al_2(SO_4)_3$ mordant tended to generate a lighter color, CaO mordant generated a red brown color resulting in a lighter color, while $FeSO_4$ produced a darker color [9]. The results of previous studies confirmed that the type of solvent used in extraction influenced the direction of the resulting color. Different results were also indicated in the mordant type treatment.

Table 1
Color catalogue of mangsi fruit extract dyeing

Sample Codes	Result Colors	Color Direction and Catalogue
Water, Alum		Brown Spanish White
Ethanol, Alum		Brown Bone
Acidic Ethanol, Alum		Brown Malta
Methanol, Alum		Brown Ecru
Acidic Methanol, Alum		Brown Gurkha
Water, Calcium carbonate		Yellow Medium Wood
Ethanol, Calcium carbonate		Yellow Putty
Acidic Ethanol, Calcium carbonate		Yellow Tan
Methanol, Calcium carbonate		Yellow Fallow
Acidic Methanol, Calcium carbonate		Yellow Barley Corn
Water, ferrous sulphate		Dark Blue Charcoal
Ethanol, Ferrous sulphate		Dark Blue Karaka
Acidic Ethanol, Ferrous sulphate		Dark Blue Charcoal
Methanol, Ferrous sulphate		Dark Blue Nero
Acidic Methanol, Ferrous sulphate		Dark Blue Night-Rider

3.1 Color Opacity Test Results

The results of the color opacity test using mangsi fruit extract on cotton fabric generated different R% and T% values. The T% value of treatment using alum mordant ranged from 45.48 to 83.21; "medium" category for water and acidic ethanol, "opaque" category for ethanol and methanol, and "very opaque" category for acidic methanol. Meanwhile, the T% value of treatment using calcium carbonate mordant ranged from 47.54 to 72.04; "medium" category for water, ethanol, and acidic ethanol and "opaque" category for acid methanol. The T% value of treatment using ferrous sulphate mordant ranged from 97.07 to 99.38; for water, ethanol, acidic ethanol, methanol and acidic methanol were considered in the "very opaque" category.

The treatment using alum mordant in water and acidic ethanol solvents produced a lighter color compared to methanol, ethanol, and acidic methanol. Furthermore, the treatment with calcium carbonate mordant in water, ethanol, and acidic ethanol solvents produced a lighter color compared to methanol and acid methanol.

Meanwhile, the ferrous sulphate mordant treated with water, ethanol, acidic ethanol, methanol,

and acidic methanol produced almost the same color opacity. The treatment using alum mordant produced the lightest color, while the treatment using ferrous sulphate mordant produced a darker/opaque color. This was because ferrous sulphate contains iron which causes the color to become opaque/dark, while alum is a colorless chemical compound so it can only accentuate the color [17]. Based on the explanation above, it can be concluded that mangsi fruit can be used as natural dyes for textiles with color quality that falls within the category of medium, opaque, and very opaque depending on the type of solvent and mordant.

Color changes in dyeing results using secang and jambal wood extracts were in accordance with the type of mordant added [10]. Alum generated a bright to brighter color, while the addition of ferrous sulphate mordant generated a color that tended to be more opaque or black. Other studies stated that mordant can be used to improve color opacity and color fastness of natural dyes characteristics on textiles [18]. The data is presented in Table 2.

Table 2
Color opacity of mangsi fruit extract dyeing

Sample Codes	Color Opacity Test Values	
	R%	T% (100%-R%)
STD-White Fabric		
Water, Alum	54.22	45.78
Ethanol, Alum	29.96	70.04
Acidic Ethanol, Alum	54.52	45.48
Methanol, Alum	37.19	62.81
Acidic Methanol, Alum	16.77	83.23
Water, Calcium carbonate	45.74	54.26
Ethanol, Calcium carbonate	47.47	52.53
Acidic Ethanol, Calcium carbonate	52.46	47.54
Methanol, Calcium carbonate	27.98	72.02
Acidic Methanol, Calcium carbonate	30.70	69.30
Water, Ferrous sulphate	2.96	97.07
Ethanol, Ferrous sulphate	1.32	98.68
Acidic Ethanol, ferrous sulphate	2.45	97.55
Methanol, Ferrous sulphate	0.62	99.38
Acidic Methanol, Ferrous sulphate	1.17	98.83

3.2 The Results of the Color Fastness

The results of the color fastness to soap washing test indicated that the color fastness of the alum mordant treatment in water had a color difference of 2.1 ("fairly good" category), alum mordant treatment using ethanol, acidic ethanol, methanol, and acidic methanol had a color difference of 1.5 ("good" category). The treatment using calcium carbonate mordant in acidic ethanol had a color difference of 2.1 ("fairly good" category), moreover the calcium carbonate mordant treatment using water, methanol, and acidic methanol had a color difference of 3.0 ("fair" category), while the treatment in ethanol had a color difference of 6.0 ("poor" category). The ferrous sulphate mordant treatment using water, ethanol, and acidic ethanol had a color difference of 6.0 ("poor" category), compared to the treatment using methanol and acidic methanol which had a color difference of "3.0 ("sufficient" category).

Based on the description, it indicated that mangsi fruit can be used as natural dyes with the quality of color fastness to washing being in the poor to good category depending on the type of solvent and mordant used. The treatment using alum mordant produced "good" average score of

fastness quality; the treatment using calcium carbonate mordant produced "fair" average score of fastness quality; while treatment using ferrous sulphate mordant produced "poor" average score of fastness quality.

This was in line with the results of examination on mangsi fruit extract dyeing using calcium carbonate post-mordanting which indicated a "sufficient" evaluation [18]. The color fastness obtained from the alum mordant treatment was better than the calcium carbonate and ferrous sulphate mordant treatments [19]. The resulting data is presented in Table 3.

Table 3
Color fastness to soap washing test results

Sample Codes	Scores	Grayscale (CD)	Category
Water, Alum	3-4	2.1	Fairly Good
Ethanol, Alum	4	1.5	Good
Acidic Ethanol, Alum	4	1.5	Good
Methanol, Alum	4	1.5	Good
Acidic Methanol, Alum	4	1.5	Good
Water, Calcium carbonate	3	3.0	Fair
Ethanol, Calcium carbonate	2	6.0	Poor
Acidic Ethanol, Calcium carbonate	3-4	2.1	Fairly Good
Methanol, Calcium carbonate	3	3.0	Fair
Acidic Methanol, Calcium carbonate	3	3.0	Fair
Water, Ferrous sulphate	2-3	4.2	Poor
Ethanol, Ferrous sulphate	2-3	4.2	Poor
Acidic Ethanol, Ferrous sulphate	2-3	4.2	Poor
Methanol, Ferrous sulphate	3	3.0	Fair
Acidic Methanol, Ferrous sulphate	3	3.0	Fair

3.3 Color Evenness Test Results

The color evenness test results were obtained from the average value of color opacity test results and then were calculated using the coefficient of variation formula in percentage. If the coefficient of variation result was close to 0, meaning that the resulting color tended to be even. Conversely, if the coefficient of variation calculation result was close to 100, hence the resulting color tended to be uneven. The resulting data is presented in Table 4.

The treatment using alum mordant in water resulted in a coefficient of variation of 0.07%, 0.09% for ethanol, 0.07% for acidic ethanol, 0.05% for methanol, and 0.06% for acidic methanol; which indicated that the use of methanol and acidic methanol solvents produced more even color compared to water, ethanol, and acidic ethanol. The treatment using calcium carbonate mordant in water resulted in a coefficient of variation of 0.07%, 0.04% for ethanol, 0.07% for acidic ethanol, 0.05% for methanol, and 0.11% for acidic methanol, which indicated that the calcium carbonate treatment with ethanol and methanol solvents generated a more even color compared to water, acidic ethanol, and acidic methanol. The treatment using ferrous sulphate mordant in water as a solvent resulted in a coefficient of variation value of 0.24%, 0.31% for ethanol, 43% for acidic ethanol, 0.39% for methanol, and 0.37% for acidic methanol, which indicated that the dyeing results using water as a solvent were better than ethanol, acidic ethanol, methanol, and acid methanol.

Table 4
Resulting values of color evenness

Sample Codes	Color Opacity Test Values		Coefficient of Variation (KV)
	R%	T% (100%-R%)	
Water, Alum	54.22	45.78	0.07%
Ethanol, Alum	29.96	70.04	0.09%
Acidic Ethanol, Alum	54.52	45.48	0.07%
Methanol, Alum	37.19	62.81	0.05%
Acidic Methanol, Alum	16.77	83.23	0.06%
Water, Calcium carbonate	45.74	54.26	0.07%
Ethanol, Calcium carbonate	47.47	52.53	0.04%
Acidic Ethanol, Calcium carbonate	52.46	47.54	0.07%
Methanol, Calcium carbonate	27.98	72.02	0.05%
Acidic Methanol, Calcium carbonate	30.70	69.30	0.11%
Water, Ferrous sulphate	2.96	97.07	0.24%
Ethanol, Ferrous sulphate	1.32	98.68	0.31%
Acidic Ethanol, Ferrous sulphate	2.45	97.55	0.43%
Methanol, Ferrous sulphate	0.62	99.38	0.39%
Acidic Methanol, Ferrous sulphate	1.17	98.83	0.37%

Research shows that dyeing is better based on water without the addition of electrolytes [20]. The color direction of natural dyes changes with changes in the pH value. Mordant has a significant effect on color evenness [21]. Based on the type of mordant being used, the dyeing results using alum and calcium carbonate as mordant produced a more even color than ferrous sulphate as mordant.

4. Conclusion

Based on the data analysis results and discussion of this study regarding the potential of Mangsi Fruit (Phyllanthus reticulatus) as natural dyes for cotton fabric using water, ethanol, acidic ethanol, methanol, and acidic methanol as solvents with the application of alum, calcium carbonate, and ferrous sulphate as mordant, it can be concluded that the treatment of the mangsi fruit extract with alum mordant produced a yellow to red-orange color direction. The calcium carbonate mordant treatment produced a yellow-orange to orange color direction. The ferrous sulphate mordant produced a blue to blue-green color direction. The results of color opacity test indicated color opacity in the "medium" to "very opaque" category. The results of the color fastness to soap washing test resulted in fastness quality in the "good" to "poor" category. The results of the color evenness test generated an even color from all treatments.

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