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Printing with Natural Dye Using Moderate Alum, $FeSO_4$ & $CuSO_4$

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Abstract

Printing is an ancient art and science that dates back to the Bronze Age. The early methods involved rubbing pigments or attaching plants to fabric to impart colour and pattern to fabric. More advanced techniques evolved, utilizing natural dyes extracted from plants, animals, insects, and minerals. While synthetic dyes are most commonly used for their vibrant colors, they pose risks such as carcinogenicity and negative environmental impacts. This has led to a renewed interest in natural dyes today. In this study, Areca nut was used as a natural dye and applied to a pre-mordanted cotton fabric by direct printing. Alum, $CuSO_4$, and $FeSO_4$ were used as mordants. $CuSO_4$ produced a more intense and vibrant color, while $FeSO_4$ resulted in a darker, more subdued hue. These observations highlight how the choice of mordant can affect the final shade of the printed fabric.

Keywords

Natural Dyeing, Areca Nut Extract, Mordants, $CuSO_4$ and $FeSO_4$, Shade Change

1. Introduction

Printing is an art and science that has evolved significantly over the centuries. Initially, the process of printing fabrics was quite rudimentary, with early methods involving rubbing pigments or attaching plant materials directly onto textiles. These early techniques gradually progressed into more sophisticated processes, and colorants extracted from plants, animals, insects, and some marine sources were used for printing on fabrics [1]. These natural dyes, when applied to fabric, provided color with some light and water resistance. Synthetic dyes emerged in the 19th century and quickly became popular due to their ability to produce vibrant, long-lasting colors at a lower cost [2]. However, the environmental impact and potential health risks of synthetic dyes, such as carcinogenic properties and harmful effects on aquatic ecosystems, have raised significant concerns in recent decades [3]. Considering these concerns, the textile industry has experienced a resurgence in natural dyes, which are biodegradable and generally less harmful to the environment compared to their synthetic counterparts. Furthermore, natural dyes are often gentler on the skin and produce a range of unique, rich colors that are difficult to replicate with synthetic dyes [4]. However, printing processes using natural dyes can be more complex and less predictable compared to synthetic dyes [5]. One of the key factors that influence the outcome of natural printing is the use of mordants. Mordants are substances that help fix the dye to the fabric, improving the intensity, color retention, and durability of the printed fabric. The most used mordants are salts of metals like aluminum, iron, copper, and tin. These metal salts react with the dye and the fiber, forming a complex that helps the dye bond more strongly with the fabric [6]. The choice of mordant can have a significant impact on the final color, as different mordants often produce different shades when used with the same dye [7].

The use of alum as a mordant is well-known in natural dye printing [8]. Alum, which is a compound of aluminum sulfate, is commonly used for pre-mordanting fabrics to improve the uptake and fixation of dyes [9]. It is a relatively mild mordant, producing lighter and brighter colors, and is often preferred for its safety and low environmental impact. The addition of other mordants, such as copper sulfate ($CuSO_4$) and ferrous sulfate ($FeSO_4$), can result in different shades and improve the overall color strength of the dye [10]. While $CuSO_4$ tends to produce vibrant, intense colors, $FeSO_4$ can yield darker, more subdued shades. These differences in shades are due to the distinct chemical reactions that occur between the mordants and the natural dye.

In this study, the focus is on printing cotton fabric using a natural dye derived from the seeds of the Areca palm (Areca catechu). It is traditionally used in some cultures for dyeing and printing fabrics, and its application in modern textile research is growing due to its eco-friendly properties [11]. The objective of this research is to explore the impact of different mordants, specifically $CuSO_4$ and $FeSO_4$, on the color characteristics of cotton fabric printed with Areca nut extract. The study investigates how the separate addition of $CuSO_4$ and $FeSO_4$, incorporated into separate starch thickeners, influences the resulting shades of the printed fabric.

The results show distinct shade differences between the two treatments, highlighting the effect of these metal salts on the final color of the print. The findings offer valuable insights into how different mordants can be used to influence the color palette available in natural printing, providing textile manufacturers and artisans with additional options for producing sustainable and vibrant printed fabrics [12]. Moreover, the study highlights the importance of understanding the interactions between mordants and natural dyes, which can be crucial for optimizing printing processes and achieving desired color outcomes [13].

The increasing interest in sustainable and eco-friendly practices in the textile industry has brought natural dyes and mordants back into the spotlight. This study aims to shed light on the specific effects of $CuSO_4$ and $FeSO_4$ as mordants on Areca nut-dyed cotton fabrics, while also providing insights into optimizing textile dyeing and finishing processes for improved energy efficiency and sustainability in fabrics, as reported in previous studies on fleece knitted textiles [14].

2. Materials and Methods

2.1. Material

Areca nut powder used as the natural dye source was obtained from Karnataka, India. Alum (potassium aluminum sulfate), corn starch, ferrous sulfate ($FeSO_4$), and copper sulfate ($CuSO_4$) were purchased from M/s S. D. Fine Chem Pvt. Ltd. Ready-for-dyeing (RFD) woven cotton fabric was procured from M/s Piyush Syndicate. The structural parameters of the cotton fabric, including ends per inch (EPI) and picks per inch (PPI), were recorded before experimentation.

2.2. Method

Extraction of Areca Nut Dye

The dye extract was prepared using an aqueous extraction method. Approximately 10 g of areca nut powder was accurately weighed and mixed with 200 mL of distilled water. The mixture was heated and boiled until the volume was reduced to approximately 100 mL to obtain a concentrated dye extract. The resulting solution was filtered through bolting cloth to remove insoluble particles, and the filtrate was used for the preparation of the printing paste.

Pre-mordanting of Cotton Fabric

For mordanting, 5 g of alum was dissolved in 200 mL of water and heated until the volume was reduced to 100 mL to obtain a concentrated mordant solution. The solution was filtered before use.

The RFD woven cotton fabric samples were treated with this mordant solution at the boiling temperature for 15 minutes. After treatment, the fabric samples were removed, squeezed to eliminate excess liquid, and dried under ambient conditions. The pre-mordanted fabric was then used for subsequent printing experiments.

Preparation of Print Paste and Experimental Design

A starch-based print paste was prepared using 10% corn starch as a thickening agent, into which the previously prepared areca nut dye extract was incorporated. The paste was thoroughly mixed to obtain a uniform consistency suitable for printing.

To investigate the influence of different metal salts on shade development and color fastness, the prepared print paste was divided into two equal portions. In one portion, copper sulfate ($CuSO_4$) was added, while in the second portion, ferrous sulfate ($FeSO_4$) was incorporated.

Thus, three experimental conditions were evaluated:

Sample A: Cotton fabric pre-mordanted with alum and printed with areca nut dye paste (control).

Sample B: Alum pre-mordanted cotton printed with areca nut dye paste containing copper sulfate.

Sample C: Alum pre-mordanted cotton printed with areca nut dye paste containing ferrous sulfate.

Each experiment was conducted with replicate samples to ensure the reproducibility of the results. During the study, key parameters such as fabric type, mordanting conditions, dye extraction method, printing procedure, drying temperature, and steaming conditions were kept constant so that the effect of the metal salts could be clearly evaluated.

Printing and Fixation

The prepared print pastes were applied to the pre-mordanted cotton fabric using a laboratory printing method. After printing, the samples were dried at 120˚C, followed by steaming at 105˚C for 20 minutes to facilitate dye fixation. The printed fabrics were subsequently washed thoroughly with water to remove unfixed dye and thickener, and then dried before further evaluation.

2.3. Fastness Testing

The printed fabric is tested for various fastness properties, such as washing, rubbing, and light fastness. Similarly, shade was also evaluated by K/S values using a computer colour matching system.

Fastness to washing (C01 - C06)

Sample preparation

Washing fastness was determined according to the ISO 105-C01 standard. The test specimen was prepared by sandwiching the printed cotton sample between two undyed adjacent fabrics, one cotton and one wool.

The composite specimen was washed in a launder-ometer under the prescribed conditions of the standard. After washing, the specimens were dried and evaluated for color change and staining using the grey scale rating.

Fastness to light

Light fastness was evaluated according to ISO 105-B02 (Xenon arc lamp method). The test specimen and a set of eight blue wool reference standards were mounted together.

One third of the specimens were exposed to the xenon arc light source for 24 hours, while the remaining portion was covered. After exposure, the fading of the specimen was visually compared with the blue wool standards and rated accordingly.

Fastness to rubbing.

Rubbing fastness of the printed fabrics was evaluated according to ISO 105-X12 using a crock meter. Both dry and wet rubbing tests were carried out. In this method, the printed specimen was mounted on the rubbing tester and rubbed against a standard white cotton cloth under controlled pressure and several rubbing cycles. For wet rubbing, the rubbing cloth was wetted with distilled water before testing.

After the test, the staining of the rubbing cloth was assessed using the grey scale for staining, and the results were reported accordingly.

Fastness to perspiration

Perspiration fastness was determined according to the ISO 105-E04 standard. The printed specimen was placed between adjacent undyed fabrics and treated with acidic and alkaline perspiration solutions.

The composite samples were kept under controlled pressure and temperature for a specified duration. After treatment, the specimens were dried at room temperature and evaluated for color change and staining using the grey scale rating.

3. Results and Discussion

Color fastness is usually assessed separately with respect to:

Changes in the colour of the specimen being tested, that is, colour fading:

Staining of undyed material, which is in contact with the specimen during rubbing, fastness

The test, which is bleeding of color.

3.1. Washing Fastness

This test measures the resistance of printed colors to washing, which is a key factor for the durability of textiles in real-life usage. The test followed ISO 105-C01 standards, using a launderometer or washing machine at 40˚C for 30 minutes with standard detergent. The printed samples were stitched to cotton and wool fabrics to check for staining, and gray scales were used to evaluate both color change and staining. Both Areca nut dye prints were tested with alum, $FeSO_4$, and $CuSO_4$ mordants. The results showed that the prints had moderate to good fastness ratings. Alum-mordanted samples displayed better performance, especially when tested on cotton, with minimal staining and less noticeable color change. $FeSO_4$ and $CuSO_4$ showed more staining on wool fabrics, indicating slightly lower fastness. Overall, washing fastness was acceptable, with alum-mordanted samples showing the best balance of stability and resistance [15].

As summarized in **Table 1** and shown in **Figure 1**, the washing fastness results indicate that the type of mordant significantly influences the durability of the printed color on cotton fabric. Among the three mordants, alum exhibited the best performance, showing good fastness ratings on both cotton (4)-(5) and wool (4), which suggests minimal color change and staining. In contrast, fabrics treated with $FeSO_4$ showed moderate fastness on cotton (3)-(4) and only fair performance on wool (3), indicating increased color bleeding, especially on protein fibers. Similarly, $CuSO_4$-treated samples demonstrated moderate fastness on cotton (3) and fair performance on wool (2)-(3), reflecting comparatively lower resistance to washing. Overall, alum proved to be the most effective mordant in maintaining color stability during washing, followed by $FeSO_4$ and $CuSO_4$.

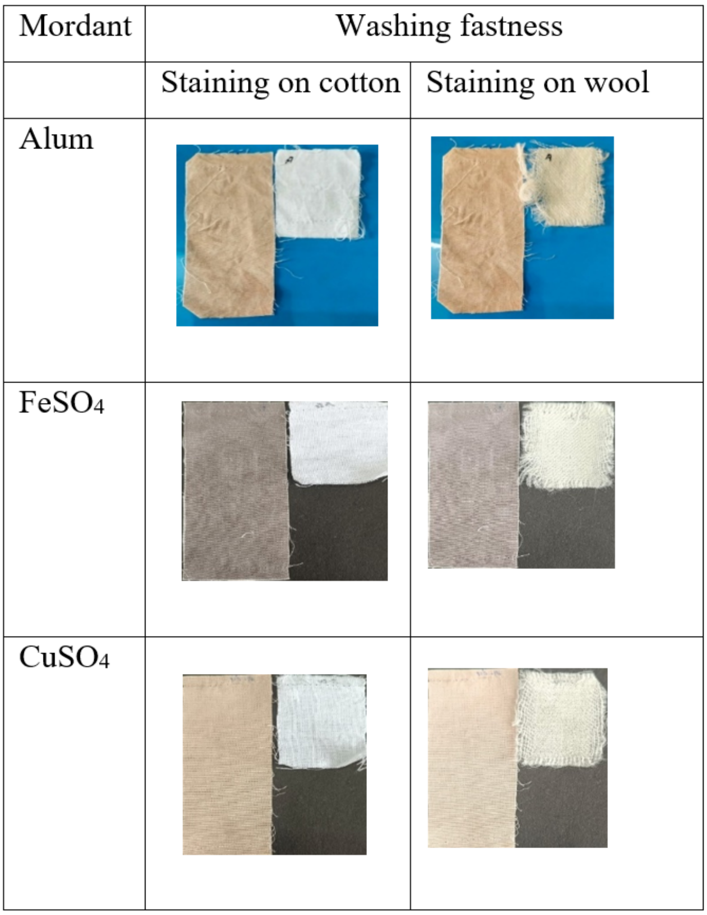


Figure 1. Image containing the test evaluation, Washing to Fastness test.

Table 1. Rating of fastness to washing tests.

Name of the test	Mordant	Rating
Washing fastness	Alum	Cotton: Good (4)-(5), Wool: Good (4)
	$FeSO_4$	Cotton: Moderate (3)-(4), Wool: Fair (3)
	$CuSO_4$	Cotton: Moderate (3), Wool: Fair (2)-(3)

3.2. Rubbing Fastness Test

The rubbing fastness test evaluates how well the printed color resists rubbing, both in dry and wet conditions. This is important to assess how the fabric will behave during wear and use. The test was conducted using a crockmeter with a white test cloth. For dry rubbing, the sample was rubbed 10 times using a dry white cloth, and the amount of color transferred was evaluated using a gray scale ranging from 1 (poor) to 5 (excellent). In wet rubbing, the same procedure was repeated, but the cloth was first wetted with water before rubbing. Results varied depending on the dye and mordant used. Areca nut dyes were tested with three mordants: alum, ferrous sulfate ($FeSO_4$), and copper sulfate ($CuSO_4$). Generally, alum-treated samples showed better rubbing fastness than those treated with $FeSO_4$ and $CuSO_4$, particularly in dry conditions. However, wet rubbing fastness was slightly lower across all samples, which is typical due to the dye's tendency to bleed when moisture is present [16].

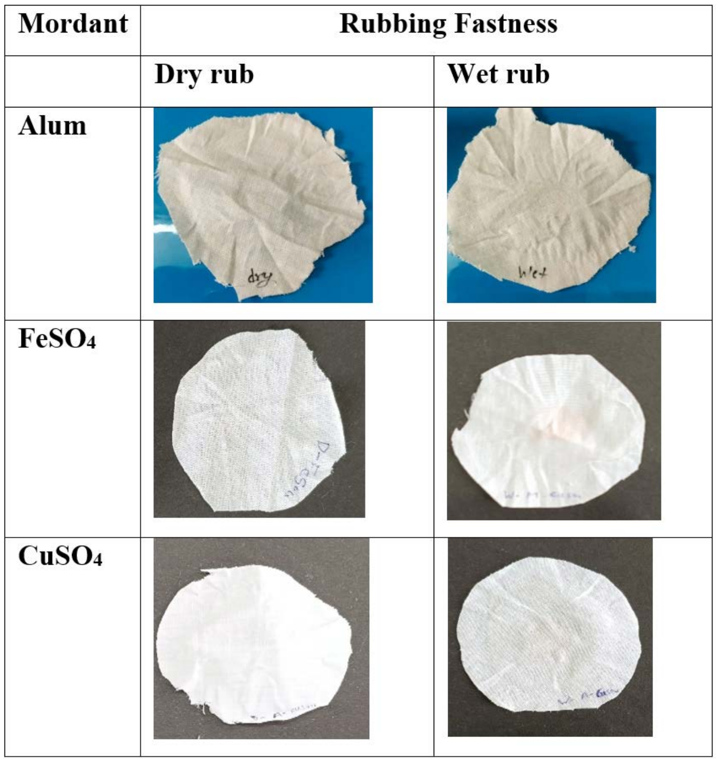


Figure 2. Image containing the test evaluation, rubbing to fastness test.

As presented in **Table 2** and illustrated in **Figure 2**, the rubbing fastness results show a clear dependence on both the mordant used and the testing condition (dry vs. wet). Alum-treated samples exhibited the best performance, with good dry rubbing fastness (4)-(5) and moderate wet rubbing fastness (3)-(4), indicating strong dye fixation and relatively low color transfer. In comparison, $FeSO_4$-mordanted fabrics showed moderate resistance in dry conditions (3)-(4) but only fair performance in wet conditions (2)-(3), suggesting increased dye bleeding when moisture is present. Similarly, $CuSO_4$-treated samples demonstrated moderate dry rubbing fastness (3) and fair wet rubbing fastness (2)-(3), indicating comparatively weaker dye adherence. Overall, the results confirm that rubbing fastness is generally higher in dry conditions, while wet conditions reduce performance due to the increased mobility of dye molecules, with alum providing the most stable results among the tested mordants.

Table 2. Rating of fastness to rubbing tests.

Name of the test	Mordant	Rating
Rubbing fastness	Alum	Dry: Good (4)-(5), Wet: Moderate (3)-(4)
	$FeSO_4$	Dry: Moderate (3)-(4), Wet: Fair (2)-(3)
	$CuSO_4$	Dry: Moderate (3), Wet: Fair (2)-(3)

3.3. Light Fastness Test

Results showed that Areca nut dyes both responded differently to light depending on the mordant used, as summarized in **Table 3**. In general, $CuSO_4$-treated samples exhibited slightly better resistance to fading, while alum and $FeSO_4$ showed moderate light fastness. The dye samples had slightly higher grades overall compared to Areca nut, especially when alum was used. This test confirmed that while natural dyes can fade under prolonged exposure, selecting the right mordant can significantly improve their stability under light [17]. The visual assessment of light fastness for different mordants is shown in **Figure 3**.

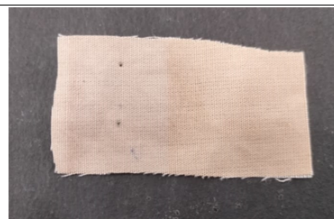
Mordant	Light fastness
Alum	
$FeSO_4$	
$CuSO_4$	

Figure 3. Image containing the test evaluation of light fastness test.

The light fastness results indicate that the resistance of the printed fabrics to fading under light exposure varies with the type of mordant used. Among the tested samples, $CuSO_4$ exhibited the best performance with a good rating (4), suggesting enhanced stability of the dye under light conditions. Alum-treated fabrics showed moderate light fastness (3)-(4), indicating acceptable resistance to fading with relatively stable color retention. In comparison, $FeSO_4$-mordanted samples displayed slightly lower performance with a moderate rating (3), suggesting a higher tendency for color degradation upon prolonged light exposure. Overall, while all mordants provided acceptable light fastness, $CuSO_4$ proved to be the most effective in improving resistance to photodegradation.

Table 3. Rating of fastness to light tests.

Name of the test	Mordant	Rating
Light fastness	Alum	Moderate (3)-(4)
	$FeSO_4$	Moderate (3)
	$CuSO_4$	Good (4)

4. Shade Evaluation

Measurements included CIE tristimulus values (X, Y, Z), CIELAB coordinates (L*, a*, b*), chroma (C*), hue angle (H*), and color strength (K/S), as presented in **Table 4**.

Table 4. Table of shade evaluation.

	ARECA NUT—Alum	1: ARECA NUT—$FeSO_4$	2: ARECA NUT $CuSO_4$
X	41.033	35.019	41.188
Y	40.226	36.198	40.559
Z	31.079	35.967	30.838
L*	69.63	66.671	69.866
a*	9.148	2.44	8.608
b*	15.337	3.634	16.087
C*	17.858	4.377	18.245
H*	59.162	56.099	61.824
dE*	-	13.81	0.954
DL*	-	−2.959	0.236
Da*	-	−6.708	−0.54
Db*	-	−11.703	0.75
DC*	-	−13.481	0.387
DH*	-	−0.473	0.839
K/S	10.24	23.843	5.053
RFL	28.19	20.411	39.692
%	100	232.829	49.342
Parts	100	42.95	202.668

4.1. Areca Nut

The color characteristics of cotton fabrics printed with areca nut extract were analyzed using a spectrophotometer. Measurements included CIE tristimulus values (X, Y, Z), CIELAB coordinates (L*, a*, b*), chroma (C*), hue angle (H*), and color strength (K/S), calculated according to the Kubelka–Munk theory at the wavelength of maximum absorption (λmax).

The alum-mordanted sample exhibited a moderate K/S value (10.24), corresponding to a medium shade depth. Its L* value (69.63) indicated a relatively light tone, while positive a* (9.148) and b* (15.337) values confirmed a red–yellow hue, giving a warm orange-brown appearance. The chroma (C* = 17.858) suggested moderate color saturation, and the hue angle (H* = 59.162˚) further verified the orange-yellow character of the shade.

The $FeSO_4$-mordanted fabric showed the highest K/S value (23.843), indicating the deepest shade. Its slightly lower L* value (66.671) reflected a darker tone, and the reduced a* (2.44) and b* (3.634) values suggested a muted red-yellow intensity. This deeper brown color is attributed to the stronger interaction between iron ions and the dye molecules, which enhances dye uptake and color fixation.

In comparison, the $CuSO_4$-mordanted fabric had the lowest K/S value (5.053), indicating a lighter shade. The higher L* (69.866), a* (8.608), and b* (16.087) values demonstrated greater brightness and more vivid red-yellow tones, resulting in a lighter, warmer orange-brown appearance.

These findings confirm that the type of mordant strongly influences shade depth and chromatic properties: $FeSO_4$ produces the darkest, more muted shades; alum yields intermediate shades; and $CuSO_4$ generates lighter, brighter tones. Overall, areca nut dye can impart visually appealing and technically satisfactory colors on cotton fabric, with mordant selection providing a practical method to adjust shade intensity and hue for sustainable textile printing applications.

4.2. Areca Nut Mordant $FeSO_4$

The spectrophotometric analysis of cotton fabric printed with areca nut dye and mordanted with ferrous sulfate ($FeSO_4$) revealed distinct colorimetric characteristics compared to alum-mordanted or $CuSO_4$-mordanted samples. The tristimulus values were X = 35.019, Y = 36.198, and Z = 35.967, indicating a slightly darker and cooler tone. The L* value of 66.671 shows a subtle reduction in brightness compared to the alum-mordanted sample (L* = 69.63), suggesting a mild darkening effect imparted by $FeSO_4$. The a* (2.44) and b* (3.634) values indicate a significant decrease in red and yellow contributions, resulting in a cooler, more subdued orange-brown shade. The *chroma* (*C = 4.377*)* further confirms a duller, less saturated color, while the *hue angle* (*H = 56.099˚*)* positions the shade in the muted orange-yellow range. These changes align with the known behavior of ferrous sulfate to subdue the warmth of natural dyes, often producing earthy or muted tones, making it particularly useful when deeper and less vivid colors are desired.

Despite the lower saturation and muted hue, the K/S value reached 23.843, more than twice the value observed in alum-mordanted fabric (10.24), reflecting stronger dye uptake and enhanced color depth. The reflectance (RFL = 20.411%) and total absorption energy (232.829) support this observation, indicating that the fiber absorbed more dye and retained a darker, richer shade. The Δ-values further quantify the effect of $FeSO_4$: ΔL* = −2.959, Δa* = −6.708, Δb* = −11.703, highlighting decreases in brightness and red-yellow intensity, while ΔC* = −13.481 and ΔH* = −0.473 confirm reduced chroma and minor hue shifts. The total color difference, ΔE* = 13.81, underscores a perceptible change in shade compared to alum-mordanted fabric. Overall, $FeSO_4$ mordanting produces darker, deeper, and more earthy tones, demonstrating a strong dye-fiber interaction that enhances color depth while moderating visual saturation, making it an effective choice for creating rich, muted shades in natural dye printing.

4.3. Areca Nut $CuSO_4$

The spectrophotometric evaluation of cotton fabric printed with areca nut dye and mordanted with copper sulfate ($CuSO_4$) revealed a bright and visually appealing shade with distinct colorimetric characteristics. The tristimulus values were X = 41.188, Y = 40.559, and Z = 30.838, indicating high brightness and moderate color depth, closely resembling the alum-mordanted standard. The **L*** value of 69.866 confirms that the fabric retains a light to medium-light tone, slightly higher than the $FeSO_4$-mordanted sample, suggesting enhanced luminance. The **a*** (8.608) and **b*** (16.087) values indicate strong contributions of red and yellow, producing a vivid and warm hue. The *chroma* (*C = 18.245*)* is slightly higher than the alum-mordanted fabric (C* = 17.858), reflecting enhanced color saturation, while the *hue angl*e (*H = 61.824˚*)* positions the shade in the orange-yellow region, characteristic of areca nut but with slightly more brilliance than alum.

The difference values demonstrate minimal shifts from the standard: **ΔL*** = 0.954 shows almost identical lightness, **Δa*** = 0.236 indicates a slight increase in redness, and **Δb*** = −0.540 reflects a small reduction in yellowness. The total color difference (*ΔE = 0.75**) confirms a close match to the standard, while **ΔC*** = 0.387 and **ΔH*** = 0.839 suggest minor increases in saturation and negligible hue change. The **K/S value** of 5.053, lower than $FeSO_4$ (23.843) and alum (10.24), indicates moderate dye absorption, consistent with the higher **reflectance (RFL = 39.692%)** and lower absorption energy (202.668). These observations confirm that $CuSO_4$ mordanting produces a lighter, cleaner, and more luminous shade with excellent visual clarity. Overall, while $FeSO_4$ yields deep, earthy tones and alum provides balanced vibrancy, $CuSO_4$ offers a bright, orange-yellow hue with moderate depth and high aesthetic appeal, making it suitable for applications where lighter and more vivid natural dye shades are desired.

5. Conclusion

Areca nut can serve as an effective natural dye for textile printing, especially when used with suitable mordants to enhance fastness and shade quality. The study compared the effects of three mordants—Alum, Ferrous Sulfate ($FeSO_4$), and Copper Sulfate ($CuSO_4$)—on the color strength, tone, and durability of the printed fabrics. Among them, $FeSO_4$ produced the darkest shades with the highest K/S value, which appeared duller with more muted tones. Alum offered a vibrant and warm orange-yellow shade with a good balance between color depth and brightness, making it aesthetically pleasing and visually traditional. $CuSO_4$ yielded the lightest shade, producing a clean and slightly golden appearance with higher brightness but lower color strength. Fastness tests confirmed that all three mordants improved the durability of the prints to varying degrees, with Alum generally offering the best overall balance of rubbing, washing, and light fastness. In conclusion, Areca nut dye, when combined with appropriate mordants, can produce eco-friendly, appealing, and reasonably durable printed textiles, supporting its potential as a sustainable alternative in natural dye applications for the textile industry.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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