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Extraction and Dyeing Behavior of Natural Dye Obtained from *Areca catechu* Powder Waste for Cotton Fabric

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***Abstract*—** Natural dyes derived from plant materials have the potential to replace synthetic dyes due to their ecological compatibility and biodegradability. This study focuses on extracting and characterizing the natural dye from powder waste of *Areca catechu* and developing a dye recipe for the cotton fabric dyeing process. The ethanol extraction method was used to extract the *Areca catechu* dye. Two different mordants (Alum and Ferrous sulfate) and two mordanting techniques (pre-mordanting and post-mordanting) at 3% and 5% concentrations were used to set the extracted dye on the cotton fabrics. The dye exhibited a pH value of 4.0, presenting a distinct red-brown hue, and had absorption spectra in the UV-A and visible light regions. The extraction was analyzed by FT-IR to determine the presence of the functional groups in each extract. Dyed cotton samples displayed good colorfastness properties, particularly in washing and light fastness. Color intensity values were measured in terms of color strength and coordinate values. Pre-mordanting the dyed cotton fabrics with 5% Ferrous sulfate proved more effective, leading to optimal color strength. Thus, the dye extracted from *Areca catechu* powder waste can be successfully utilized with ferrous sulfate to dye the cotton fabrics. The future study of *Areca catechu* natural dye should focus on the economic viability of producing the dyes commercially.

***Keywords*:** Natural Dye, Ethanol, Areca nut, textile, extraction, characterization

I. INTRODUCTION

The historical and cultural significance of color in human societies is a manifestation of artistic expression, identity, and tradition. Natural dyes derived from plant, animal, or mineral sources have been instrumental in shaping these expressions by imparting hues to fabric such as cotton, silk, and wool (Verma & Gupta, 2017). Natural dyes are highly valued due to their ecological suitability and biodegradability, in contrast to synthetic dyes that often contribute to environmental pollution (Pizzicato et al., 2023). They are increasingly favored by consumers due to the rich, smooth, and soft color they impart, and unique aesthetic qualities that synthetic dyes cannot replicate (Chipambwa & Nyathi, 2017). The global demand for natural dyes exceeds 10,000 tons annually, equivalent to approximately 1% of the worldwide consumption of synthetic dyes (Chipambwa & Nyathi, 2017).

Among all natural dyes, those derived from plant sources hold significant potential due to their widespread availability. Plants such as indigo, madder, weld, and henna have been extensively utilized (Samanta, 2020). *Areca catechu* nuts are rich in sugars, lipids, and polyphenols (Jung, 2017). These polyphenols encompass condensed tannins, hydrolyzed tannins, lignans, stilbenes, flavanes, flavonoids, anthocyanins, and simple phenolic acids (Pawar et al., 2018). In the context of *Areca catechu* processing factories in Sri Lanka, a waste product in the form of powder/dust is generated. This agricultural waste includes unripe and molded nuts, which are typically disposed of by burning. Effectively utilizing this agricultural waste would address disposal issues and provide an additional source of income for farmers and processing industries that generate this waste. The presence of tannins in *Areca catechu* waste offers opportunities for dye extraction, potentially mitigating waste accumulation while providing economic avenues for agricultural communities.

II. BACKGROUND OF STUDY

Consumer awareness of the environment and health has recently grown as a result of the numerous negative effects of synthetic colors. Discarding dyes incorporated wastewater into water bodies decreases the passage of sunlight, prevents photosynthesis, and inhibits plant growth (Ardila-Leal et al., 2021; Patel, 2011). Furthermore, synthetic dyes have been shown to be poisonous, mutagenic, carcinogenic, bio-accumulative, and recalcitrant (Ardila-Leal et al., 2021). Moreover, even at low concentrations, these dyes cause harmful effects on the environment. Many synthetic dyes, particularly azo dyes have been found to be potentially carcinogenic (Verma & Gupta, 2017). In addition to being carcinogenic, certain chemicals used in the synthesis of dyestuffs are also regarded to be mutagenic as well as sensitizing or allergenic (Patel, 2011).

Due to these environmental and human health impacts of synthetic dyes, there is an increased interest in environmentally friendly coloring options, including safer synthetic and natural dyes (Pawar et al., 2018). Above all, natural dyes have the benefit of being biodegradable and sourced from renewable resources, meaning they have less of an adverse effect on the environment (Singhee, 2020). Consumers favor fabric dyed with natural dyes because of the deep, smooth color they impart. Natural dyes are also valued for their ability to express cultural history, status, and unique aesthetic features that synthetic dyes cannot seem to replicate (Selvaraj & Prathiba Devi, 2023).

Areca nut (*Areca catechu*) is widely distributed in tropical Asia, East Africa, and the central Pacific regions, and can successfully extract natural dye for the textile industry. *Areca catechu* nuts are rich in sugars, lipids, and polyphenols, with polyphenols representing the most bioactive constituents (Jung, 2017). These polyphenols encompass tannins and dyes extracted from *Areca catechu*, are part of the tannin group, and are highly concentrated within *Areca catechu* nuts (Jannah et al., 2021).

The extraction of natural dyes is a complex process because these are not comprised of a single chemical entity. To extract natural dyes, various methods are employed, reflecting the complexity of the task. These methods include alkali or acid extraction, aqueous extraction, solvent extraction, microwave and ultrasonic-assisted extraction, fermentation and enzymatic extraction, and supercritical fluid extraction (Saxena & Raja, 2014). Depending on the nature of the substance, the extraction technique, the solvent, and the optimum parameters of the extraction process are chosen.

These extracted dyes are typically integrated through the dyeing process. The crucial factors that influence the dyeing process of textile substrates are fiber structure, temperature, dyeing duration, dye concentration in %, the salt concentration in %, pH of the dye bath, and the characteristics of the dye molecules (Gupta, 2019). However, textile fibers typically lack a strong affinity for most natural dyes, and as a result, an additional step, known as mordanting, becomes necessary.

Mordants act as intermediaries with an affinity for both textile fibers and dyes, bridging the connection between them, although the mechanism of action varies depending on the type of fiber (Pizzicato et al., 2023). Various mordants, either applied individually or in combinations, can be employed on textile fabrics to achieve different colors or shades, enhance dye uptake, and improve colorfastness behavior with natural dyes (Arora et al., 2017). Mordant application methods are categorized into three main approaches based on their timing in the dyeing process: pre-mordanting, post-mordanting, and meta-mordanting or simultaneous mordanting (Ramli et al., 2021).

III. PROBLEM SPECIFICATION

A substantial waste is generated at *Areca catechu* processing factories which includes dust/powder and unripe and molded nuts. Typically, this waste is disposed of by burning. However, if this agricultural waste is effectively utilized, it could help address disposal issues and provide an extra source of income for farmers and processing industries that generate this waste.

Although previous investigations have conducted on natural dye extraction from *Areca catechu*, they have predominantly focused on laboratory-scale experimentation. Therefore, a substantial research gap exists concerning utilizing these findings in industrial-scale applications for fabric dyeing. This research aims to extract and characterize the natural dye from the powder waste of *Areca catechu* and to explore the feasibility of integrating *Areca catechu* waste-derived natural dye into industrial fabric dyeing processes.

IV. METHODOLOGY

A. Materials

In this experiment, Areca nut (*Areca catechu*) powder formed as a waste product in *Areca catechu* processing factories was collected from a factory in Matale, Sri Lanka. A 100% mercerized cotton fabric (weight of 80 g m-2 with 58 ends per inch (EPI) and 72 picks per inch (PPI)) was purchased from Janasalu, Department of Industrial Development, Kurunegala, Sri Lanka. The laboratory-grade chemicals of Ethanol (C2H5OH, Scientific Limited, Northampton, UK), Ferrous sulfate (FeSO4, Sigma Aldrich Co., Germany), and Aluminum potassium sulfate (KAl(SO4)2.12H2O, Alum, Fluka, Germany) were purchased from chemical suppliers in Colombo, Sri Lanka. Distilled water (laboratory water distiller- wall mounted type, Bionics Scientific Technologies, India) was used in dye extraction and chemical preparation. All the chemicals were used without any additional purification.

B. Dye extraction:

The collected *Areca catechu* powder underwent a sieving process using a 250 µm sieve to achieve a consistent particle size (Figure 1). 10 g of *Areca catechu* powder was combined with 200 ml of ethanol (1:20) in a soxhlet apparatus, as shown in Figure 2.

The solution obtained through extraction was then concentrated to enhance the concentration of the extracted compounds. Subsequently, the solution was poured into Petri dishes and oven-dried. Then, the dye was scratched out of the petri dishes and collected in powder form (Figure 3). The obtained dye extract was then characterized and used for further dyeing studies.

Figure 1: Sieved *Areca catechu* powder

Figure 2: Soxhlet extraction of *Areca catechu* dye

Figure 3: Powdered *Areca catechu* dye

C. Characterization of the dye

1) pH value analysis:

The pH of the dye was measured using a digital pH meter (S-610H pH meter, PEAK Instruments, USA). Here, the powdered dye is dissolved in distilled water to prepare the dye solution and three pH readings of the dye solution were taken using the pH meter. Then the first reading was removed and the average pH value was calculated using the second and third readings.

2) Fourier-transformed infrared spectroscopy (FTIR) analysis:

The determination of specific functional groups of *Areca catechu* extracted dye was measured by Fourier transform infrared (FTIR) spectroscopy (Vertex 80, Bruker Corporation, USA). The samples were placed in FTIR-ATR mode at 600-4000 cm^{-1}.

3) Ultraviolet-visible (UV-Vis) spectroscopy analysis:

The maximum absorption of the wavelength (λ_{max}) of the aqueous extract dye sample was determined using Ultraviolet-visible (UV-Vis) spectroscopy (UV-3600i Plus, Shimadzu Corporation, Japan). The extracted dye was then diluted by dissolving 1 ml in 50 ml of Deionized water to measure its absorption spectrum under a range of wavelengths (200 nm–800 nm).

D. Mordanting and dyeing

Dyeing was carried out with different mordants and without any mordants. The procedure consisted of several steps. As mordants, ferrous sulfate ($FeSO_4$) and Aluminum potassium sulfate ($KAl(SO_4)_2$/Alum) were used. The mordanting methods employed were Pre-mordanting and Post-mordanting, both conducted at a temperature of 80 °C for 2 hours.

To pre-mordant the fabric samples, two solutions were prepared by adding the mordant at 3% and 5% concentrations (% of the fabric weight) with distilled water. The Material-to-Liquor Ratio (MLR) utilized was 1:30. The samples were squeezed thoroughly to remove excess water and allowed to dry for a few minutes. The dyeing process was carried out at its initial pH value. MLR used was 1:40. 1.0 g of prepared dye powder was mixed with 200 ml water for 30 minutes using a magnetic stirrer. Dyeing was carried out for 5.0 g of cotton fabrics at 70 °C for 2 hours.

To prepare for post-mordanting, the fabric samples were initially dyed at its' initial pH value using a MLR of 1:40. Two dye solutions were prepared by mixing 1.0 g of dye powder with 200 ml of water for 30 minutes using a magnetic stirrer. Then the dyeing was carried out for 5.0 g of cotton fabrics at 70 °C for 2 hours. Then the dyed fabric was mordanted using the Alum and Ferrous Sulfate at 3% and 5% concentrations with distilled water. MLR utilized was 1:30.

E. Evaluation of dyed fabric color characteristics

1) Evaluation of colorfastness:

The dyed fabric samples were subjected to colorfastness assessment under different conditions, including exposure to light, rubbing, and perspiration. *Areca catechu* dyed fabric was assessed according to the corresponding international standards, that is, fastness to washing (ISO-105-C06-2010), fastness to rubbing (ISO-105-X12-2016), and fastness to light (ISO-105-B02-2014). The changes in the color and the staining of the adjacent multi-fiber fabric were assessed using grey scales (Glogar et al., 2020).

2) Evaluation of color strength and color space of dyed fabric samples:

Color strength is a measure of the ability of a dye to bring color to materials. The color strength is evaluated by light absorption in the visible region of the spectrum. The ratio of K/S values for samples compared to a standard at the same wavelength, expressed as a percentage is known as the relative color strength. 'K' and 'S' are a dyed sample's absorption and scattering coefficients. The higher the K/S value is, the darker the color is. The color strength (K/S) values of the dyed fabric samples were determined using the "Kubelka-Munk" equation (Equation 01) (Shimo & Smriti, 2015).

$$\frac{K}{S} = \frac{(1-R)^2}{2R} \text{ ---------- (Equation 01)}$$

Where R is the reflectance of the dyed fabric, K is the absorption coefficient, and S is the scattering coefficient. The reflectance of the dyed samples (R) was measured using the Data Color spectrophotometer (DC 800 Spectrophotometer, Datacolor Technologies Suzhou Corporation, China). The color strength values of the dyed fabric samples were determined using the ratio of absorption (K) and scattering (S) coefficients (K/S) in the Data Color tools plus software (the built-in software in the spectrophotometer). The color strength values of the dyed fabric were used to quantify and express the color characteristics of the dyed fabric samples. They were measured using the Commission International de l'Eclairage (CIE) color space. The CIE Lab color space values are expressed as three color values: L* represents the perceptual lightness from black to white, a* represents the green–red colors, and b* represents the blue–yellow colors (Ji et al., 2023).

V. RESULTS AND DISCUSSION

A. Dye characterization

1) pH value:

The pH level of the dye solution influences the dyeing behavior during dyeing, impacting solubility and color outcome. It can also affect the fastness properties, the colour strength of the dyed fabric (Yasassri et al., 2019). Without any mordanting agents, the ethanol extract solution of *Areca* catechu dye maintains a pH level 4.0, displaying a deeper red-brown shade.

2) UV–Vis spectroscopy analysis:

The UV spectrum of the *Areca catechu* dye solution with a pH value of 4.0 is presented in Figure 4. The characteristic spectrum has shown the absorptions in 316nm and 442nm regions. The peaks of 316 nm show tannin compounds in the dye (Naveed et al., 2020). This spectrum also shows a peak at 442 nm in the visible zone, indicating the presence of a yellow color in *Areca catechu* ethanol extract (Dhanania et al., 2021).

It can be observed that the *Areca catechu* dye molecules can easily absorb radiations in the UV-A region (310-400 nm) and the visible light region (400-700 nm). UV-A makes up about 95% of the UV radiation that reaches the Earth and it is the primary cause of photo aging, wrinkling, skin cancer, and eye damage because of it can penetrate the dermis layer of skin (Bashari et al., 2018). As a result, the absorption peaks in the UV-Vis zone for *Areca catechu* dye extract shows its preferential UV absorption, giving good light fastness properties and high UV protection (Dhanania et al., 2021).

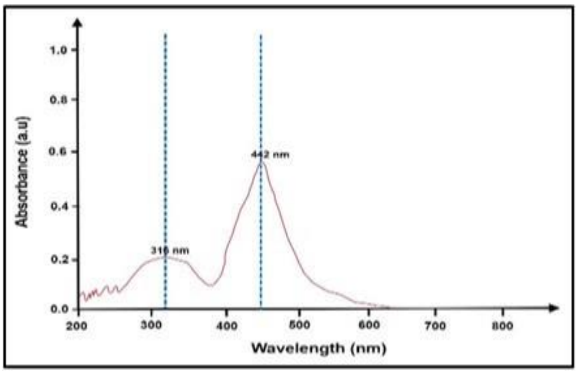

Figure 4: UV-Vis spectral analysis of *Areca catechu*

1) Fourier-transformed infrared spectroscopy (FTIR) analysis:

The FTIR spectra obtained for *Areca catechu* dye extracted using ethanol extraction is shown in Figure 5. The broad absorption band 3376 cm^{-1}, which appeared in the 3500–3100 cm^{-1} range, indicates the presence of –OH groups (Sadeghi-Kiakhani et al., 2020). Again, the spectrum near 2900 cm^{-1} corresponds to the stretching of the –CH– group (Singh et al., 2021), and 1673 cm^{-1} corresponds to the conjugated C=C stretching mode of vibration (Adedokun et al., 2018). The peaks at 1416 cm^{-1} are attributed to the vibrations of the aromatic C=C bond (Singh et al., 2021). The spectrums at 1237 cm^{-1} 1050 cm^{-1} were attributed to the C-O group, and the C=C bond is found at 793 cm^{-1} wavenumber (Handayani et al., 2019).

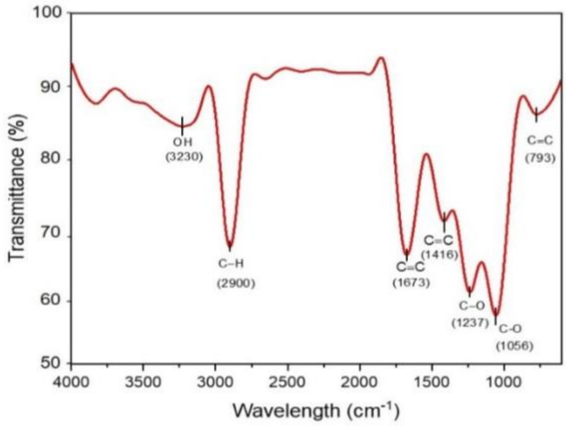

Figure 5: Fourier transform infrared spectra of *Areca catechu* dye

B. *Dyed fabric characterization*

1) *Color evaluation:*

The fabrics readily pick up a brownish color as soon as it is immersed in both dye bath, but in the presence of a mordant, the color changes accordingly. The dyeing depth was improved using different metal salt mordants in both methods. The color chart of the dye is shown in Table 1.

Table 1: Color evaluation of *Areca catechu* dye extracted by Ethanol extraction

Mordant type		**Colour shade**	
Non-mordant			
		Mordanting method	
		Pre	Post
Ferrous sulfate	3%		
	5%		
Alum	3%		
	5%		

1 Table 2: Colorfastness Ratings for dyed cotton fabric samples

Mordant	**Mordant concentration**	**Mordanting method**	**Wash fastness**		**Light fastness**	**Rubbing fastness**	
			Color Change	**Color Staining**		**Dry staining**	**Wet staining**
Non	-	-	3	4-5	3	4-5	4-5
$FeSO_4$	3%	Pre	4	5	4	4	4
		Post	4	5	4	4	4-5
	5%	Pre	4	4-5	4	4	4
		Post	4	4-5	4-5	4	4-5
Alum	3%	Pre	4	4-5	3-4	4	4
		Post	4	5	3-4	4	4-5
	5%	Pre	4	5	3-4	4	4
		Post	4	4-5	4	4-5	4-5

2

The fabric's color has become darker after dyeing and mordanting because of ferrous sulfate. The presence of Alum as the mordant caused a brighter yellowish shade on the fabric. Typically, when mordants are used, coordinate covalent bonds are formed between the fiber and the dye molecules, resulting in complex formations that may cause color changes. Alum is a neutral mordant that doesn't affect the depth of color change, whereas iron and copper intensify it, making the color darker (Kabir et al., 2020).

2) *Fastness properties of Areca catechu dye:*

The fastness properties of dyed fabric samples can be altered based on the type of mordants used, the method of mordanting, and the concentration of the mordant. To determine how much the dyed cotton sample can withstand washing, light, and rubbing, the color fastness test must be carried out. Table 2 shows the colorfastness ratings of cotton fabric samples.

In wash-fastness, significant improvement was observed in the case of all mordants, mordanting concentrations, and mordanting techniques. The rating for colorfastness to washing for both substrates appears to be above four for color change and the adjacent fabric's staining. The mordanting method has the same effect on the colorfastness of washing for both the mordanting agents. This indicates excellent fastness to washing.

Colorfastness to light for cotton dyed with both dyes without any mordanting agent was improved upon applying ferrous sulfate and Alum as the mordanting agents for both the mordanting methods. The colorfastness to light is higher for ferrous sulfate than Alum in the dye. In both mordanting agents, the colorfastness to light is relatively higher for the post-mordanted fabrics of 5% concentration.

Colorfastness to rubbing improved by using ferrous sulfate and Alum as the mordants. The ratings for dry staining appear to be four, except in the 5% post-mordanted sample. In wet staining, the post-mordanted samples exhibited higher fastness properties than the pre-mordanted fabric samples in both mordant concentrations. This indicates excellent fastness to rubbing for both mordants and mordanting concentrations.

3) *The Color Characteristics of the dyed fabrics:*

Relevant data in Table 3 show the effects of variation in the dyeing process conditions on color strength (K/S) and CIE L* a* b* values of dyed fabric samples. The color characteristic values of dyed cotton fabrics produced through the ethanol extraction of *Areca catechu* dye are shown in Figure 6. It shows the dye uptake of the dyes in terms of K/S values at different concentrations and mordanting methods. It was found that using a higher concentration of the mordant during the dyeing process significantly improved the K/S values of the fabric. The pre-mordanted fabric with 5% Ferrous sulfate had the strongest color (K/S = 3.3), while the post-mordanted with 5% Alum had the weakest color (K/S = 1.15). The pre-mordanted samples with 3% and 5% Ferrous sulfate exhibited excellent K/S values compared to non-mordanted ones. However, for post-mordanting, both concentrations of Alum resulted in lower values. Improvement in the K/S value of the dyeing indicates a higher amount of dye adsorption, interaction, and bridging with the mordanted substrate via different conjugated bonds (Dhanania et al., 2021). This indicates that pre-mordanting with a higher concentration of mordant is the best method for Ferrous sulfate. In contrast, pre-mordanting is the best method for Alum to achieve optimal dye absorption and interaction.

There are variations in hue color as well as significant changes in K/S values, L* values, and brightness index values with the type of mordant used. Figure 7 shows the CIE L* a* b* values of dyed cotton fabrics with *Areca catechu*. The CIEL*a*b* color coordinates L*, a*, and b* represent lightness (L* = 0 yields black and L* = 100 indicates white), redness–greenness of the color, and yellowness–blueness of color, respectively (Jiang et al., 2019).

It can be seen that the fabric samples with lighter shades give a higher value of L*, while those with a darker shade have a lower L* value. The L* values were lower for the fabric samples dyed using ferrous sulfate, corresponding to deeper shades. The L* value was higher for non-mordanted dyed samples, corresponding to lighter shades. Similarly, using Alum, the L* values were also higher, leading to lighter shades.

Red and yellow hues are represented by high values of a* and b*, respectively, which also indicate brightness. The a* value varied from 4.4 to 13.71, where those positive values show that the dyeing colors obtained from all the dyeing processes were red. It also can be seen that the b* value of all the dyed fabrics ranged from 10.38 to 20, representing the yellow color. Using Alum as the mordant has increased the quality of the red and yellow tones, while Ferrous sulfate has caused a reduction in both.

Table 3: K/S and CIE L*a*b*values of dyed samples with *Areca catechu* dye

Mordant concentration	Mordant	Mordanting method	K/S %	L*	a*	b*
5%	Alum	Pre	2.69	55.22	13.84	18.79
		Post	1.99	60.25	12.28	19.61
	$FeSO_4$	Pre	4.50	42.81	8.93	10.91
		Post	6.44	38.83	4.76	12.68
Non			1.74	58.17	9.86	12.39
3%	Alum	Pre	2.03	58.75	12.10	17.64
		Post	1.55	62.03	11.73	16.26
	$FeSO_4$	Pre	2.39	52.01	6.24	10.38
		Post	2.60	50.78	4.87	10.67
5%	Alum	Pre	3.32	52.73	13.71	20.00
		Post	1.15	66.15	11.04	15.58
	$FeSO_4$	Pre	3.98	45.20	6.95	11.79
		Post	3.11	48.65	4.40	11.49

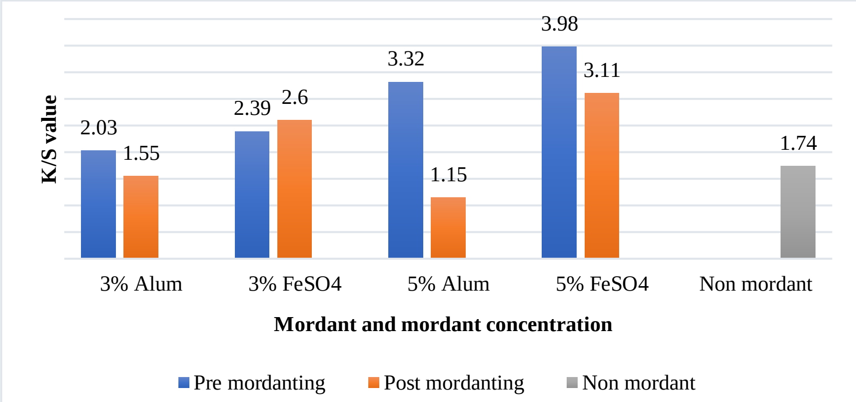

Figure 6: K/S value comparison of cotton fabric dyed with *Areca catechu*

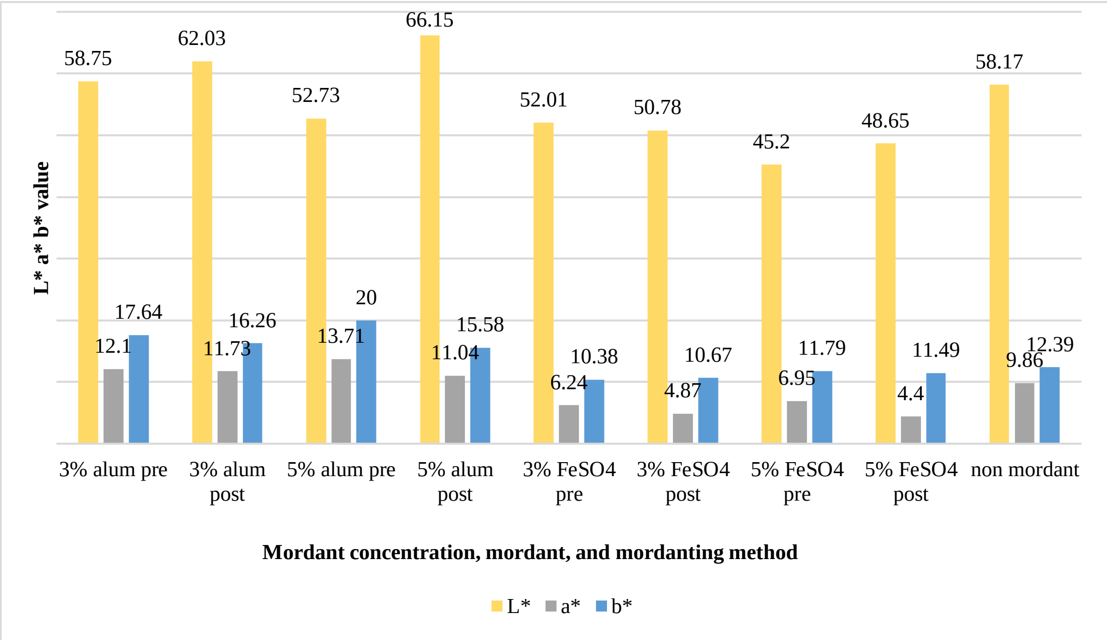

Figure 7: L*a*b* value comparison of cotton fabric dyed with *Areca catechu* dye

VI. CONCLUSION

In this study, the extraction and the dyeing behavior of natural dye from powder waste of *Areca catechu* using Ethanol extraction method was studied. Without mordanting agents, the dye exhibited a pH of 4.0, presenting a distinct red-brown hue. The dye has absorption spectra in the UV-A region and visible light region. The structural analysis of the functional groups in the dye was established using FTIR spectroscopy. For the cotton fabrics mordanted using 5% Ferrous sulfate by, post-mordanting gave the optimum color strength with good fastness properties. Pre-mordanting the dyed cotton fabrics with 5% Ferrous sulfate proved more effective, leading to optimal color strength. The data generated through this work may be used to study the economic viability of producing the dyes on a commercial scale.

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