

Dyeing properties and color fastness of eri silk yarn dyed with soaked red kidney bean water

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Abstract

The purpose of this research was to study the dyeing of eri silk yarn using soaked red bean water. The optimal condition for preparing dye solutions was a ratio of 1:5 of dried red bean to water and dyeing at 100°C for 45 min. The dyeing process was conducted with and without mordants, using three different mordanting methods; pre-mordanting, meta-mordanting, and post-mordanting. The effects on silk analyzed are color fastness to washing, wet and dry rubbing, acid and alkali perspiration, and color characteristics on CIE L*a*b* color coordinates. Without mordants, the dyed silk yarn gave a rosy brown color. Each mordant caused shade changes on dyed yarns and mordant techniques affected the color intensity and color fastness of dyed eri silk yarn.

1. Introduction

Traditionally, natural dyes are colorants derived from plants, fungi, insects, animals or minerals, and were used for coloring silk, wool, and cotton fibers. However, over time, cheaper synthetic dyes were invented and commercialized to replace natural dyes. Various plant parts namely; roots, barks, leaves, and seeds can be used as a majority of natural dye sources [1]. The advantages of synthetic dye stuffs over natural dyes are color fastness, good reproducibility of shades, brilliance of color, and ease of use [2]. However, synthetic dyes are poisonous and can cause allergies and are carcinogenic and detrimental to human health [3,4]. Natural dyes derived from plants have recently gained economic advantage over synthetic dyes because of their extraction from renewable sources, nonhazardous nature, and biodegradability [5]. Due to the current environmental consciousness of people about natural products, the researchers' attention has been shifted to the use of natural dyes for dyeing textile materials in the past few years [5-7].

Color fastness in the majority of natural dyes can be enhanced by mordants, a chemical species that facilitates the binding of dye to fabrics by means of forming a chemical bridge between the fiber and the dye. This bridge essentially increases the dye's ability to stain the fabric as the color fastness properties are enhanced. For this reason, the mordanting method, which is already applied to the dye used in the study, will inevitably vary the characteristics and, in particular, the color fastness depending on the source of dye [8].

Many natural materials from plant and animal sources are identified as possible colorants, one such established colorant is red kidney bean, *Phaseolus velgaris* L. Generally, the red kidney beans should be soaked in water for 6-8 h to make them swell up and ripen

quickly when cooked. The soaking water is often discarded and is not used for any other purpose. In a recent study, it has been reported that the red color pigments in the seed coats of kidney beans are an attractive potential source for natural dye and food colorants [9,10]. The extracts from *Phaseolus velgaris* L. plant species revealed the presence of anthocyanin in the seed coat of red kidney beans [9]. The most common anthocyanidins distributed in plants are cyaniding, delphinidin, pelargonidin, malvidin, and petunidin [10], as illustrated in Figure 1.

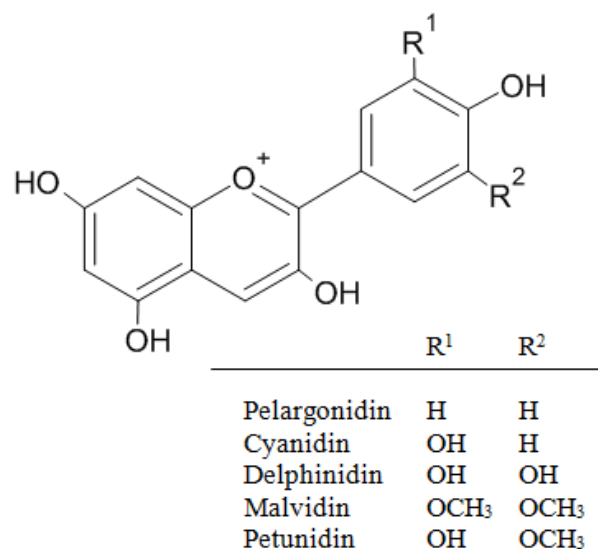


Figure 1. Structure of anthocyanidins: pelargonidin, cyaniding, delphinidin, malvidin, and petunidin.

Although some reports are available, research on the exact method of the red kidney bean dyeing is limited. Moreover, eri silkworm is a silkworm that is categorized as a wild silkworm which eats a castor or mostly cassava leaf [11]. In Thailand, eri silkworm could be reared on cassava leaves, which are its major host plants, and create local employment opportunities to increase income substantially instead of discarding them [12,13]. In addition, the process of obtaining eri silk fiber does not require the immersion of the cocoons in boiling water to kill the silkworms. Therefore, the research is conducted to study the dyeing of eri silk yarn with the residual water from red kidney beans which have been soaked, in order to find use for the residual water which is only currently seen as waste. The various selected mordants and mordanting methods were used and color fastness to washing, rubbing, perspiration and color characteristics were determined.

2. Experimental

2.1 Materials

The ESY Nm 15/3 eri silk yarns were purchased from Spun Silk World Co., Ltd, Thailand. Red kidney beans were acquired from Thanya farm Co., Ltd, Thailand. Three different analytical grade mordants which were ferrous sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$), copper sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$), and stannous sulfate (SnSO_4), were used as received without any further purification, and were obtained from Sigma-Aldrich. The standard soap without optical brightening agent was purchased from SDC Enterprises Limited, UK.

2.2 Dye extract preparation

The dye extraction was performed by mixing dried red kidney beans and water in the weight ratio of 1:1 to 1:9 and soaking them overnight. After that, the resulting solution was filtered to remove the residue and red kidney beans. The crude dye extract was measured for maximum absorbance with the UV-Vis spectrophotometer to obtain the optimum ratio of dried red kidney bean and water. The experiments were performed in triplicate and the results were reported as mean \pm standard deviation.

2.3 Dyeing and Mordanting

To investigate the effects of dyeing time, eri silk yarns were dyed for 30-90 min with the soaking water from the ratio of dried red kidney beans and water at 1:5 using OD-501 UniDye Infra-red dyeing machine (Union TSL Limited, Thailand). The liquor ratio of yarn to water was 1:30 at temperature of 100°C.

The eri silk yarns were dyed at optimum conditions using the red kidney bean soaking water with three different mordanting methods, i.e. pre-mordanting, meta-mordanting, and post-mordanting, at a liquor ratio of 1:30 at 100°C for 45 min. The three mordant types with a concentration of 5 g·L⁻¹ for 45 min were used to improve color strength and color fastness of dyed eri silk yarns.

After dyeing, the dyed samples were rinsed with water, soaped with 2 g·L⁻¹ of standard soap at a liquor ratio of 1:40 at 50°C for 15 min, and finally rinsed with water and air-dried.

The yarn samples were subject one of the following mordanting processes:

Pre-mordanting : mordanting – dyeing – soaping – rinsing and drying

Meta-mordanting : mordanting and dyeing – soaping – rinsing and drying

Post-mordanting : dyeing-mordanting – soaping – rinsing and drying

2.4 Scanning electron microscopy analysis

The surface morphology of the eri silk yarns before and after the application of mordant and dye were analyzed with a JSM-6610LV-Jeol scanning electron microscope (Jeol Ltd., Japan) at 15 kV accelerating voltage. The yarns were adhered on brass stubs and then sputter coated with gold using a Q150RS Quorum sputter coater (Quorum Technologies Ltd, UK) for 3 min in an argon atmosphere.

2.5 Color measurement

The color strength value (K/S) and CIE L*a*b* of all dyed yarns were evaluated using a Datacolor Check II spectrophotometer, Datacolor, USA. The UV-Vis spectrum of the crude red kidney bean extract dye in aqueous solution is presented in Figure 2. All measured samples showed the maximum absorption wavelength value at 490 nm. The K/S is a function of color strength and is calculated by the Kubelka-Munk equation.

$$K/S = (1 - R)^2 / 2R$$

Where R is the reflectance of the dyed yarns at maximum wavelength

The influence of the mordants and mordanting methods on the color of the dyed fabrics in terms of depth of shade and color was analyzed.

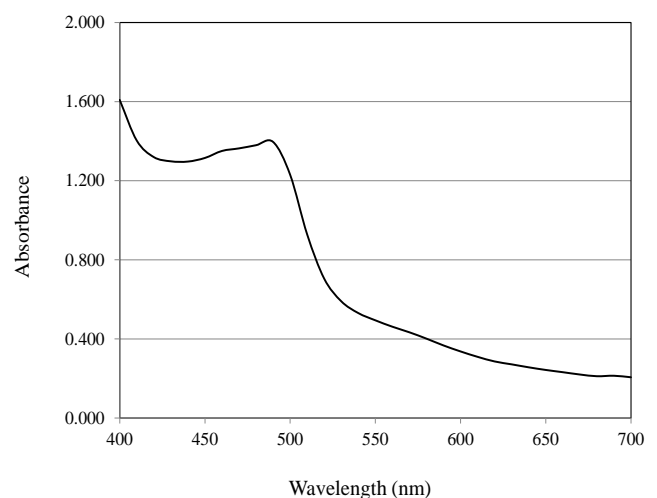


Figure 2. UV-Vis spectrum of crude red kidney bean extract dye in distilled water.

2.6 Color fastness determination

Color fastness to washing, acid and alkaline perspiration, and wet and dry rubbing was rated in terms of color change and color stain properties using the grey scale. The color fastness to washing and perspiration of the dyed samples was determined according to ISO 105-C10 A1:2006 and ISO 105E04: 2013, respectively. Both dry and wet rubbing fastness of dyed samples was tested according to ISO 105-X12:2016 test method.

3. Results and discussion

3.1 Optimum ratio of red kidney bean and water for dye extract preparation

The effect of a ratio of red kidney beans and water on the absorbance of the dye solution is shown in Figure 3 and Figure 4. It was observed that the absorbance decreases with the increase of the red bean to water ratio. The soaked water showed a yellow to pinkish orange color which is because red beans contain the flavonoid compound, a type of phenolic compound [14]. The red kidney bean and water in a 1:1 ratio could not be measured for absorbance due there being no water left after the overnight soaking process. The ratio of red kidney beans and water at 1:2 exhibited the highest absorbance compared with others showing the highest color strength; however, the dye solution with the lowest soaking ratio was not suitable for dyeing. On the other hand, the dye solution with the highest soaking ratio of red kidney beans to water of 1:9 displayed the lowest color strength. In this research, the 1:5 red kidney bean and water ratio was used for dyeing as an optimum condition for preparing the dye solution because of a sufficient amount of water and good colorants from the red beans.

3.2 Effect of dyeing time on color strength and shade

The color strength of eri silk yarns dyed with red kidney bean extracts is related to the dyeing time. The results given in Table 1 showed that the color of dyed silk yarn was a rosy brown, a medium light shade of red. The affinity of the red kidney bean dye toward silk yarn may be due to the presence of both amino and carboxylic

groups present in the silk fiber. Red kidney bean dye contains anthocyanidins, which can form hydrogen bond or ionic bond with silk fiber (Figure 5). Increased color strength could be achieved by keeping the dyeing bath at 100°C for 45 min. The color strength of dyed silk yarn was not significantly changed after dyeing for 45 min. This was a similar result to Mongkholrattanasit, *et al.* [15] which studied the dyeing of cotton fabric with mangrove bark extract. They found that a longer dyeing time gave higher color strength until the dyeing equilibrium was reached at approximately 60 min.



Figure 3. Images of red kidney bean soaking water with a ratio of red beans and water at 1:2 (left) to 1:9 (right).

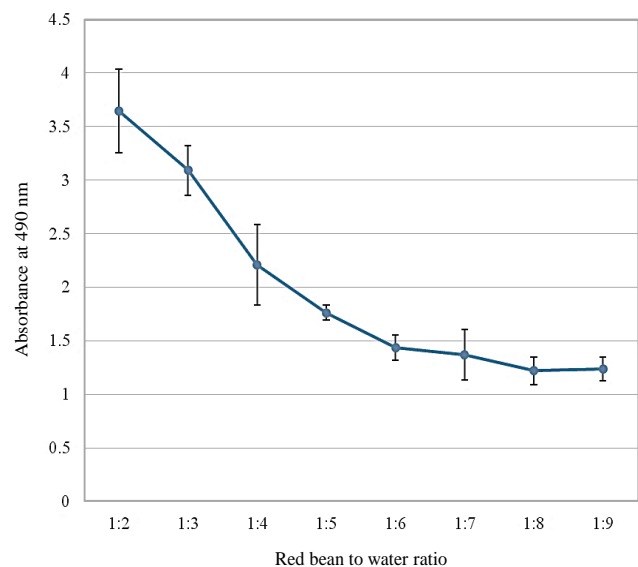


Figure 4. Absorbance of red kidney bean soaking water with a different ratio of red beans and water.

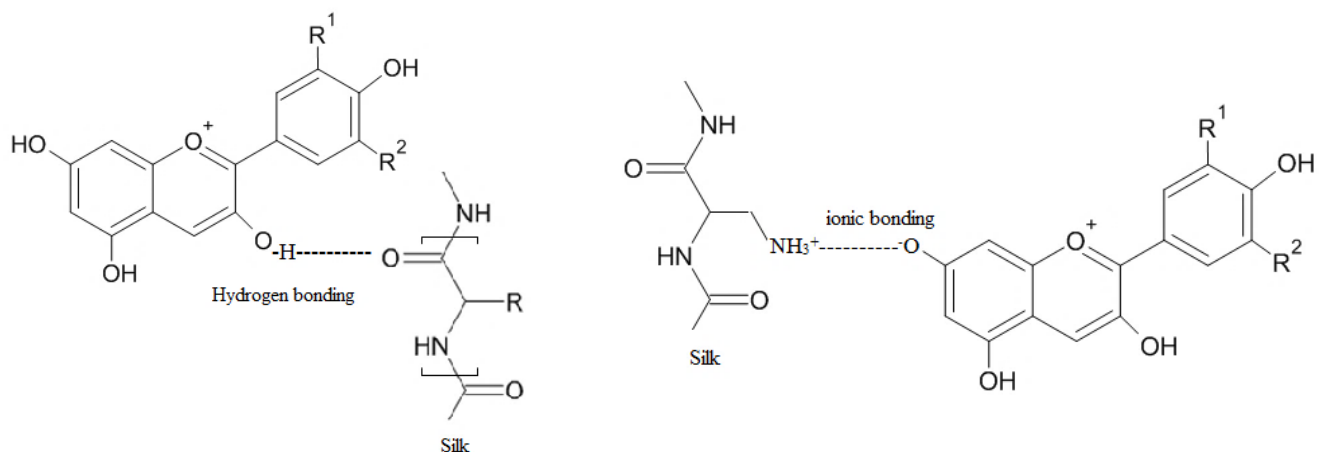








Figure 5. Hydrogen bonding and ionic bonding between red kidney bean dye and silk fiber.

Table 1. Color values at varying time with a temperature of 100°C without mordanting.

Time (min)	K/S	L*	a*	b*	Color samples
0	0.0736	88.96	0.24	9.67	
30	0.5449	72.58	9.16	9.58	
45	0.6749	69.97	10.29	9.60	
60	0.6498	70.51	10.04	9.64	
75	0.5894	71.57	9.96	9.32	
90	0.6281	70.77	10.32	9.27	

3.3 Dyeing and Mordanting

To ensure that the natural dyes are able to achieve the most durable and long lasting colors, sometimes mordants is required [16]. There are several metal salts used as mordants e.g. copper sulfate and ferrous sulfate, which can give different color shades with the same dye.

The dyeing results from mordanting experiments revealed that varying chemical mordants produced different shades and viable ratings of fastness properties on red bean-treated silk yarns. Table 2 shows CIE L*a*b* values for eri silk yarn dyed with three different mordants by three different mordanting methods. The meta-mordanting method showed lower color strength than pre-mordanting and post-mordanting method, except copper sulfate meta-mordanting. This suggests in favor of a dye-mordant interaction in the dye bath solution rather than on the fiber [17]. In addition, using copper sulfate and ferrous sulfate as mordants showed higher color strength than stannous sulfate, which was a similar result to that in the study conducted by Mongkholrattanasit, et al. [15]. An explanation for this observation is that copper sulfate and ferrous sulfate create coordination complexes, which is the coordination number of copper sulfate and ferrous sulfate is 4 and 6, respectively. Some coordination sites remain unoccupied when mordant reacts with fiber so that these metal mordants can create ternary complex on which one site binds with the fiber and the other site binds with dye [18]. Also, stannous sulfate have strong bonds with the dye and not with the fiber resulting in reducing the dye interaction with the fiber [18].

The L* value indicates perceived lightness in CIE color space varying from 0 to 100 with 0 being black and 100 being white. Among different chemical mordants used in the study, lightness of

dyed silk yarns with copper sulfate and ferrous sulfate tended to decrease, which is comparable to that of dyed silk yarn without mordants. Meanwhile, the dyed silk yarn with stannous sulfate became more bright or light in color. The ferrous sulfate produced darker shades than others, which may be associated with the change of ferrous sulfate into a ferric form by air oxidation. Ferrous and ferric forms coexist in the fiber and overlapping of the spectra causes a shift in λ_{max} , resulting in a color change to a darker shade [19].

The a* value indicates red (+a*) and green (-a*) while the coordinate b* represents the difference between blue (-b*) and yellow (+b*). The silk yarn dyed without using any mordant showed the highest redness value compared with the dyed yarn with the mordants, while the lowest a* value was obtained when using stannous sulfate as a mordant in the meta-mordanting stage. In the case of b* value, the copper sulfate and stannous sulfate gave more yellow color than that obtained from dyeing without mordant; however, using ferrous sulfate during the pre- and post-mordanting stage has shown a decreased yellow color. The dyeing mechanisms of eri silk fiber with red kidney bean soaking water by pre-mordanting and post-mordanting methods can be shown in Figure 6 and Figure 7, respectively.

During pre-mordanting, the mordant first bonds with amino and carboxyl groups in silk and then the mordant bonds with the dye. The mordant assists to form a bridge between silk and dye molecules and thus causes fixation of the dye to the silk fiber (Figure 6) [22]. In post-mordanting, the dye molecules initially form bonds with amino and carboxyl groups in silk and then the dyes bond with the mordant. In this case, the mordant does not act as a bridge, instead the mordant blocks the active sites of the dye molecule so that it cannot react with other chemicals (Figure 7).

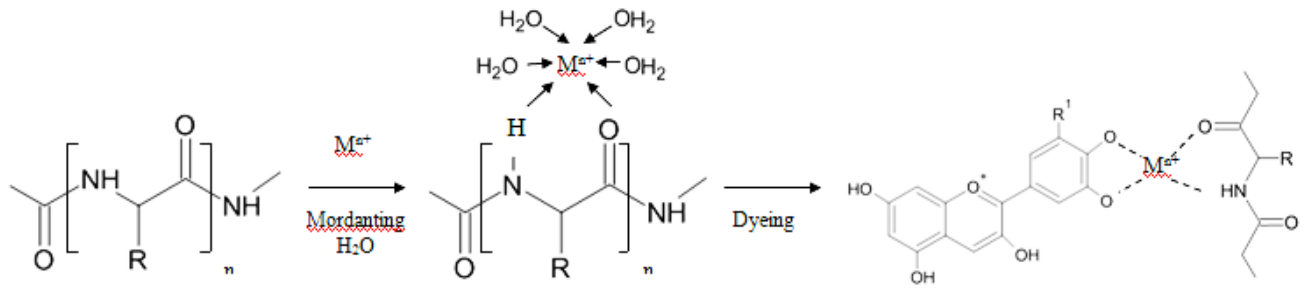












Figure 6. Proposed mordant-dye complex according to pre-mordanting method [20,21].

Table 2. Color values of eri silk yarns dyed with red kidney bean soaking water.

Mordant	Method mordanting	K/S	CIE L*a*b*			Color sample
			L*	a*	b*	
without	-	0.9445	66.53	12.98	12.19	
	Pre-mordanting	2.7463	47.58	4.72	9.81	
Ferrous sulfate	Meta-mordanting	1.4297	59.43	3.42	17.47	
	Post mordanting	2.1519	50.96	2.19	10.53	
Copper sulfate	Pre-mordanting	2.5064	52.44	7.40	20.04	
	Meta-mordanting	1.6651	57.97	2.81	21.12	
Stannous sulfate	Post mordanting	1.4295	58.86	6.24	13.08	
	Pre-mordanting	0.4759	76.42	5.29	19.18	
Stannous sulfate	Meta-mordanting	0.2080	85.87	1.40	20.51	
	Post mordanting	0.7751	69.95	10.93	15.66	

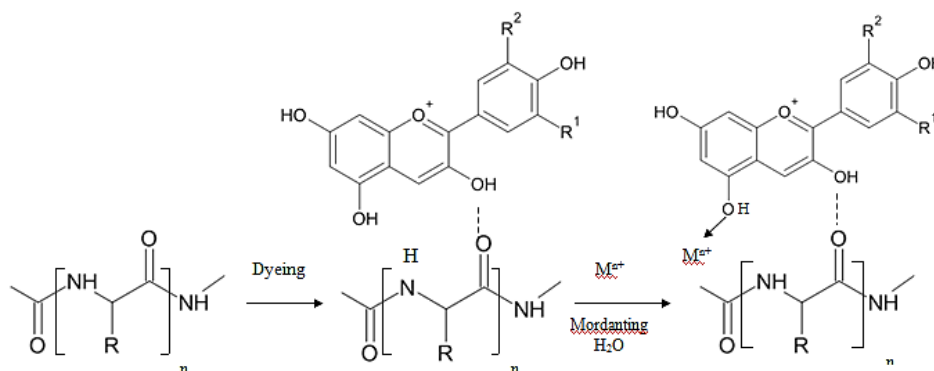


Figure 7. Proposed mordant-dye complex according to post-mordanting method [21].

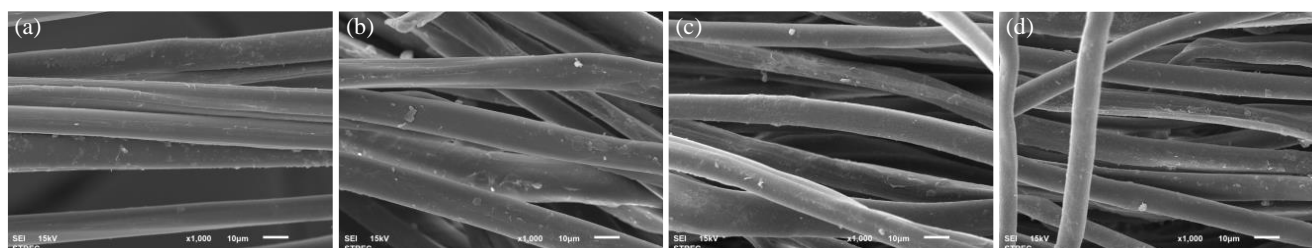


Figure 8. Surface morphology of (a) eri silk yarn and (b-d) three mordanting methods of ferrous sulfate mordanted silk dyed with red kidney bean extract (b) pre-mordanting (c) meta-mordanting and (d) post-mordanting.

3.4 Scanning electron microscopy analysis

The eri silk morphologies before and after dyeing are depicted in Figure 8. It can be seen that the surfaces of eri silk fibers and eri silk fibers dyeing with pre-mordanting, meta-mordanting and post-mordanting method showed a similar morphology which without any physical changes.

3.5 Color fastness

Color fastness is the resistance of a material to change its color or transfer its colorants to adjacent materials, or both. Normally, fastness properties are expressed in ratings of fastness and range from a rating of 5, which means unchanged, to a rating of 1, which means major change [23].

3.5.1 Wash fastness

The wash fastness properties of red kidney bean soaking water dyed eri silk yarn using $5 \text{ g}\cdot\text{L}^{-1}$ mordant concentration are presented in Table 3.

The wash fastness rating was measured using standard the grey scale for loss of shade depth and staining. The wash fastness ratings of unmordanted dyed yarns in both color change and color staining were very good (4) when compared with their respective unwashed control samples. Mordanting with different metal salts has significantly altered wash fastness ratings of dyed silk yarn. Color staining of the dyed yarns on adjacent fibers demonstrated a rating of good to excellent (3 to 5) shown in Table 3. In dyeing with the meta-mordanting method, the colors on the silk yarns with three different mordants were changed more than that of the unmordanted dyed silk yarns. This

result is the same as that reported by Saravanan, P. and Chandramohan, G. [24], whereby the color fastness rating was found to depend on mordant types and methods used. In addition, some yarns tended to become more reddish after the washing test due to the process of ionization of the hydroxyl groups in the molecules of the dyes. Such ionization process occurred either due to the alkaline properties of the detergent solution which was applied [25,26], or due to the dye itself decomposing. This then results in a compound that is colorless or differentially colored [26].

3.5.2 Rub fastness

Generally, the dye particles that do not interact with the fibers deposited on the fiber surface. When the textile materials were worn, they can be rubbed on fiber surfaces which can cause the dye particles to stain the skin. Therefore, the color fastness to rubbing was tested. The tests involve rubbing the sample with a dry rubbing cloth and a wet rubbing cloth. Dry rubbing color fastness refers to the fading and staining of dyed yarn when rubbed with a standard white cloth. Wet rubbing color fastness refers to the fading and staining of dyed yarn when rubbed with a standard white cloth the water content of which is between 95% and 105%. Both color fading of the fabrics and staining of cotton white fabric may appear after rubbing in dry and especially in wet conditions, the results are shown in Table 4.

Table 4 shows that an outstanding dry rubbing fastness rating without any color fading was achieved. The color stain on cotton cloth after wet rubbing showed higher color stain than dry rubbing. During the rubbing test, the unfixed or loose dye particles from the dyed textile material may have been transferred to different surfaces, and were absorbed by the white fabric [27]. In the case of wet rubbing, unfixed dyes will dissolve in water and then be transferred to the test fabric, hence resulting in poor wet rubbing fastness [28].

3.5.3 Perspiration fastness

Perspiration fastness means the ability of a dyed textile material to not fade or stain due to sweat. This method is intended to determine the resistance of color of dyed textile to artificial acidic and alkaline perspiration. The results of perspiration fastness of dyed eri silk yarns are shown in Table 5 and Table 6.

The fastness grades of unmordanted samples showed a very good (4) rating in terms of color change, and a very good to excellent (4-5) rating in terms of color staining when exposed to both acid and alkali perspiration (Table 5 and Table 6). In most of the cases, the color change ratings found for acid perspiration were higher than the ratings for alkali

perspiration, which concurs with the study conducted by Uddin, M.G. [22]. This can be explained by the pH value of perspiration affecting the hydrolytic stability of dye-fiber bonding, which then causes the fading of dyes [29]. Furthermore, the resistance to color change and staining varied according to the mordant type and method used. When ferrous sulfate and stannous sulfate were used as mordants a fairly poor (2) to very good (4) rating of resistance to color change was observed, whereas when copper sulfate were used, a good (3) to excellent (5) rating of resistance to color change was observed. Among the staining results, significant to good to very good staining (rating 3-4) was noticeable when using ferrous sulfate during the meta- and post-mordanting stage. All other staining results were found to be satisfactory

Table 3. Color fastness to washing of eri silk yarn dyed with red kidney bean soaking water.

Mordant	Method mordanting	Grey scale rating						
		Color change	Color staining					
			Acetate	Cotton	Nylon	Polyester	Acrylic	Wool
None	-	4	4	4	4	4-5	4-5	4
Ferrous sulfate	Pre-mordanting	4	4-5	4	4	4-5	5	4
	Meta-mordanting	3	4-5	4	4	4-5	4	4
	Post mordanting	3-4	4	3-4	4	4	4	3-4
Copper sulfate	Pre-mordanting	4	4	4	4-5	4-5	5	4
	Meta-mordanting	3	4-5	4-5	4-5	5	4-5	4-5
	Post mordanting	3-4	4	4	4	4-5	4-5	4
Stannous sulfate	Pre-mordanting	2-3	4	3-4	4	4-5	4-5	4
	Meta-mordanting	3	4	4	4	4-5	4-5	4
	Post mordanting	4	4-5	4-5	4-5	4-5	4-5	4-5

Table 4. Color fastness to rubbing of eri silk yarn dyed with red kidney bean soaking water.

Mordant	Method mordanting	Grey scale rating (color staining)	
		Dry	Wet
		None	-
Ferrous sulfate	Pre-mordanting	4-5	2
	Meta-mordanting	2	2-3
	Post mordanting	2	2
Copper sulfate	Pre-mordanting	4-5	3
	Meta-mordanting	4	4
	Post mordanting	3-4	4
Stannous sulfate	Pre-mordanting	4-5	4
	Meta-mordanting	4-5	4
	Post mordanting	4	3-4

Table 5. Color fastness to acid perspiration of eri silk yarn dyed with soaked red kidney bean.

Mordant	Method mordanting	Grey scale rating						
		Color change	Color staining					
			Acetate	Cotton	Nylon	Polyester	Acrylic	Wool
None	-	4	4-5	4-5	4-5	4-5	4-5	4-5
Ferrous sulfate	Pre-mordanting	3	4	4	4	4	4	4
	Meta-mordanting	3	4	4	4	4	4	4
	Post mordanting	2	4	4	4	4	4	4
Copper sulfate	Pre-mordanting	3-4	4	4	4	4-5	4-5	4
	Meta-mordanting	4-5	4	4	4	4	4	4
	Post mordanting	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Stannous sulfate	Pre-mordanting	3	4-5	4-5	4-5	4-5	4-5	4-5
	Meta-mordanting	4	4	4	4	4-5	4-5	4
	Post mordanting	4	4	4	4	4-5	4-5	4

Table 6. Color fastness to alkali perspiration of eri silk yarn dyed with soaked red kidney bean.

Mordant	Method mordanting	Color change	Grey scale rating					
			Color staining					
			Acetate	Cotton	Nylon	Polyester	Acrylic	Wool
None	-	4	4-5	4-5	4-5	4-5	4-5	4-5
Ferrous sulfate	Pre-mordanting	4	4-5	4-5	4-5	4-5	4-5	4-5
	Meta-mordanting	2	4	3-4	3-4	4	4	3-4
	Post mordanting	2	4	3-4	3-4	4	4	3-4
Copper sulfate	Pre-mordanting	4-5	4	4	4	4	4	4
	Meta-mordanting	4	4-5	4-5	4-5	4-5	4-5	4-5
	Post mordanting	4	4	4	4	4-5	4	4
Stannous sulfate	Pre-mordanting	2	4	4	4	4-5	4-5	4
	Meta-mordanting	4	4-5	4	4	4-5	4-5	4
	Post mordanting	3	4-5	4-5	4-5	4-5	4-5	4-5

4. Conclusions

The study demonstrated that red kidney bean soaking water can be used as a source of natural dyes. The color values with respect to K/S, L*, a*, b* values and fastness properties were found to be influenced by type of mordants and mordanting methods. Ferrous sulfate mordant was found to be the most influence to color values. Mordants, with the exception of stannous sulfate, improved K/S values of the dyed yarns compared to unmordanted dyed yarns. The color change to washing for all the treated samples is found to be fairly poor to very good for all mordant methods. The dry rubbing color fastness of dyed samples was found to be better in comparison with wet rubbing. The acidic perspiration fastness of dyed yarns demonstrated higher resistance to color change than that of alkali perspiration. The resistance to color staining of all mordants was acceptable.

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