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The type and composition of the main elements in the mud that influence the color shades of purple silk

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Abstract

This research aimed to study the types and compositions of main elements in mud that influence the shades of purple silk. The population was purple silk threads, and the sample consisted of purple silk threads dyed with sappan heartwood and subsequently overdyed with mud. All samples were obtained from the Ban ta long silk weaving group, located in tung wang subdistrict, sa tuek district, buriram province. The analysis of the element composition in the mud was conducted using the x-ray fluorescence energy dispersive spectrometer (EDXRF) technique. The results revealed the presence of iron oxide ions in the mud solution at concentrations equivalent to ferrous sulfate ion solutions exceeding 300 ppm. When silk threads are dyed with an extract from sappan heartwood using alum as a mordant, they attain a red hue characterized by CIELAB color values of L* 60.81, a* 30.90, and b* 13.14. Subsequent overdyeing with a mud solution alters the silk's color to purple, with corresponding CIELAB values of L* 43.49, a* 7.90, and b* 0.17. This shift in color is attributed to the presence of iron ions in the mud solution, which interact with the dye, leading to a perceptible change in hue. Furthermore, the extract from sappan heartwood (*Caesalpinia sappan*) contains a red pigment known as brazilein, which can form an insoluble complex compound with iron ions. This compound exhibits a distinctive purple hue that binds effectively to silk fibers or protein fibers, resulting in uniquely colored natural-dyed silk fabric that reflects a distinctive cultural identity. The findings contribute to community-based knowledge that can be passed on to future generations or individuals interested in studying this valuable traditional wisdom. Moreover, this knowledge has the potential to generate sustainable household income, reduce urban migration, alleviate poverty, and promote environmentally friendly practices.

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1. Introduction

At present, there is a high global demand for natural dyes due to the fact that synthetic dyes are non-biodegradable and carcinogenic, whereas natural dyes are non-toxic, do not cause allergic reactions, and are not carcinogenic. Sources of natural dyes are derived from animals, plants, and elements in the mud, without undergoing chemical processing [1]. In general, natural fibers are categorized into two types: (1) cellulose fibers or plant-based fibers such as cotton, linen, or jute, and (2) protein fibers or animal-based fibers such as wool or silk. Protein fibers tend to absorb natural dyes more effectively than cellulose fibers because their molecular structure contains a large number of functional groups, particularly amino groups (–NH_2) and hydroxyl groups (–OH), which can form strong chemical bonds with natural dye molecules [2], as illustrated in Fig.1 [3].

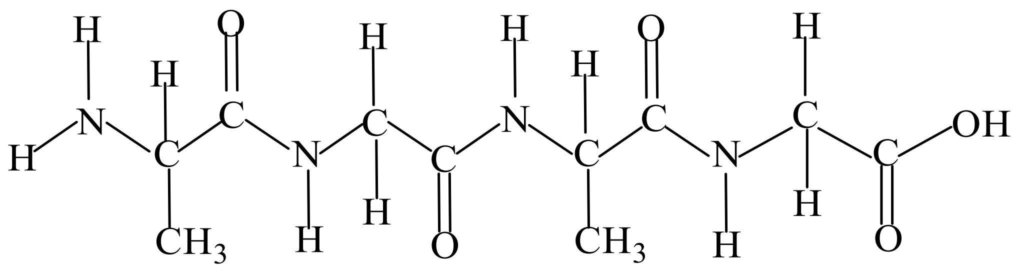


Fig. 1 The structure of silk fiber

Silk is composed of two types of proteins sericin and fibroin, which make up about 25% and 75% of its weight, respectively. Sericin is a gummy protein that surrounds the fibroin fibers. It is a hydrophilic chain that is removed from the inner fibroin using hot water in a process called degumming, which is done prior to dyeing [3]. Natural dyes derived from plants contain color-producing substances extracted from various parts of the plant, such as the fruit, flowers, leaves, stems, and roots. These dyes are classified as organic dyes, and their categorization depends on their chemical structure. According to the research report by Bishal *et al.* [4], plant-based colorants can be classified into the following groups: (1) **Indigoids**: Indigo is an important natural dye that has been used by human civilizations for the longest time and is considered the "king" of natural dyes, providing blue hues. Examples of sources include *Perisicaria tinctoria*, (2) **Pyridine-based dyes**: These produce bright yellow colors. Examples of sources include *Berberis aristata*, *Rhizoma coptidis*, (3) **Carotenoids**: These are tetraterpene pigments that produce yellow, red, orange, and purple colors. Examples of sources include *Crocus sativus* and *Cedrela toona*. (4) **Quinonoids**: These dyes produce yellow to red colors. Examples of sources include *Juglans regia* (Walnut), and *Tabebuia avellanedae* (Taigu/Lapachol). (5) **Flavonoids**: These produce orange, red, yellow, and blue colors. Examples of sources include *Allium cepa* (Onion), and *Datisca cannabina* (Hemp). (6) **Dihydropyran-based dyes**: These produce purple and red colors, with haematoxylin as the main component. Examples of sources include logwood (*Haematoxylon campechianum*) and brazilin from brazilwood (*Caesalpinia sappan*). (7) **Betalains**: These produce purple and yellow colors. Examples of sources include *Opuntia lasiacantha* and *Beta vulgaris*. (8) **Tannins**: These are found in various parts of plants, such as fruits, leaves, gall, bark, roots, wood, etc. Examples of sources include *Punica granatum* and *Quercus infectoria*.

Brazil Wood or Sappan heartwood of Brazilwood (*Caesalpinia sappan)* [4] Generally, the dye extracted from sappan heartwood yields a red color. Textiles can be dyed with or without the use of alum mordant and still result in red shades. However, when sappan heartwood extract is mixed with turmeric, it produces an orange color, and when mixed with *Terminalia chebula* (Manjakani), it gives a deep reddish-brown hue. In Thailand, sappan heartwood is primarily used in silk dyeing and traditional herbal medicine. Additionally, it is also utilized in other important industries, particularly in the cosmetics industry. The color producing compounds in sappan heartwood are brazilin and brazilein. However, brazilin can be oxidized into brazilein when exposed to light, air, and heat, resulting in a red coloration [5], as shown in Fig. 2

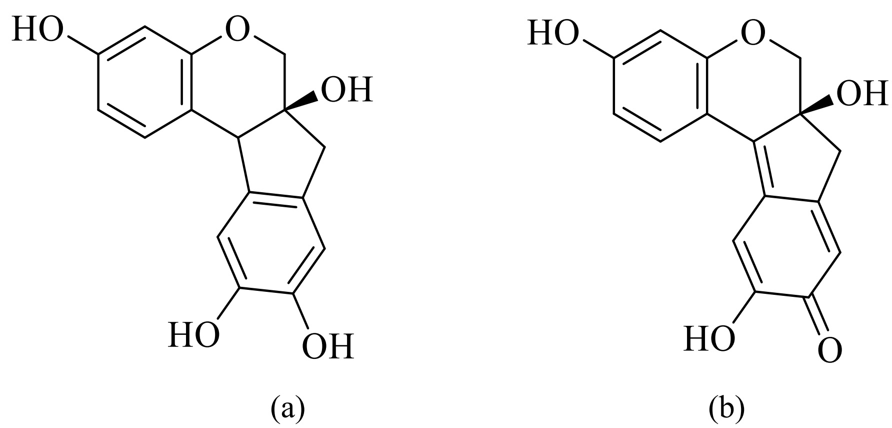


Fig. 2 The structural characteristics of sappan heartwood extracts brazilin (a) and brazilein (b)

Additionally, there is a group of dyes derived from elements in the mud, which are categorized as natural inorganic pigments. These originate from elements and include various colors obtained from inorganic metal salts and metal oxides, such as mercury sulphide (HgS), iron oxide ($Fe_2O_3 \cdot nH_2O$), and arsenic sulphide (As_2S_3), among others [4].

Mordants are elemental substances that help dyes bind more effectively to fibers enabling the dye to bond with the fabric [5]. Some types of fabric, such as silk and wool, can be dyed easily by simply immersing them in natural dye, while other types, like cotton, require the use of mordants because most natural dyes do not readily adhere to fibers. The main types of mordants are metal salts or tannin-based compounds, which create chemical bonds between pigment and fiber - a process known as mordanting. Metal salts act as mordants by forming coordination complexes with both the dye and the fiber, resulting in large molecules (e.g., aluminum sulfate or other metallic mordants fixed to fibers). The chemical reactions involved depend on the functional groups present in the mordant that bond with the dye via covalent bonds, hydrogen bonds, or other intermolecular forces. Mordants can be divided into two categories: (1) Metal-based mordants - including aluminum (Al), chromium (Cr), copper (Cu), iron (Fe), and tin (Sn); and (2) Tannin-based mordants-derived from natural plant sources. Common techniques used to analyze the structural characteristics of silk and cotton fibers include scanning electron microscopy (SEM) and Fourier-transform infrared spectroscopy (FTIR) [3]. Furthermore, to identify the types of element components found in mud muds used in natural dyeing, techniques such as SEM, X-ray fluorescence (XRF), and Energy Dispersive X-ray Fluorescence (EDXRF) are commonly employed [4]. Buriram Province is renowned both nationally and internationally for its silk weaving. Many famous silk types originate from this region, such as Na pho district silk, Ban sanuan nok silk, Wat pho yoi silk, Barai 1,000 years old mud-dyed silk [6], and Ban ta long silk.

Ban ta long, located in Tung wang subdistrict, Satuek district, Buriram province, is an OTOP Na-wat-wi-thi tourism village rich in diverse cultural heritage. One of its most celebrated cultural treasures is the “Royal Purple Silk” with the following historical background. The “Royal Purple Silk” or “Silk Offered to the Princess” is a type of double-weft squirrel-tail silk dyed with 100% natural dyes. It was invented by Mrs.

Muang Yoyram, who discovered that the red dye from sappan heartwood (*Caesalpinia sappan)* would turn purple when dropped onto the mud. This led her to the hypothesis "If silk threads dyed with sappan heartwood are then fermented in mud, the result may be a purple color." She conducted experiments to test this theory and found it to be true. As a result, the naturally purple-dyed silk was successfully presented to Her Royal Highness Princess Maha Chakri Sirindhorn on March 4, 2019 [6].

Therefore, from the discussions and interviews with the leaders and members of the ban ta long silk weaving group, the researcher found that the community is interested in studying the cause of the color change in silk threads from reddish orange to purple-after being fermented with mud. This research was thus initiated to generate knowledge for the community and local area, serving as a guideline for future study and passed down within the community in the future.

2. Materials and Methods

Preparation of solutions

Prepare a 1,000-ppm solution of iron sulfate ($FeSO_4{\cdot}7H_2O$) (AR grade, Ajax Finechem, Australia) with a total volume of 1 L, and a 1,000 ppm solution of alum ($KAl(SO_4)_2{\cdot}12H_2O$) with a total volume of 1 L. [7].

The weight of ferrous sulfate ($FeSO_4{\cdot}7H_2O$, AR grade, Ajax Finechem, Australia) about 0.5 g. was dissolved in 0.5% nitric acid, adjusted the volume with distilled water in a 100 mL volumetric flask. The final concentration was 1,000 ppm. Poured into the bottle for further use.

The weight of alum ($KAl(SO_4)_2{\cdot}12H_2O$) about 10 g was added 100 mL distilled water. Then poured the solution into the container and boiled it at 80 °C. Let it cool at room temperature, and then poured the solution into a 1,000 mL volumetric flask (adjusted the volume with distilled water). Poured into the bottle for further use.

The weigh of 5 grams of sappan heartwood (*Caesalpinia sappan*) and boil in 1 L of water for 1 hour. After boiling, filter the extract using a muslin cloth, then pour the filtered extract into a 1 L beaker and allow it to cool to room temperature for further use. Weigh 10 grams of dried mud and dissolve it in 1 L of deionized water, resulting in a mud solution with a concentration of 10,000 ppm.

Preparation of silk fibers

The silk fibers intended for dyeing must first be cleaned to remove dust, grease, or other impurities. Begin by boiling water at a temperature of 70-90°C, then add 180 grams of silk soap and 50 grams of soda ash per 30-40 L of clean water. This solution is used to clean 1-2 kg of raw silk [7]. Soak the raw silk in the solution for approximately 1 hour and 30 minutes, until the yellow coloring substances are completely removed and the silk fibers appear cream-colored, like eggshells. Then rinse the silk thoroughly with warm water and gently wring it out. Next, soak the silk in salt water for 10 minutes in preparation for the dyeing process. For the experiment, the silk must be divided into 5 groups, each with 3 replications. Each sample should weigh 1 gram and measure 10 centimeters in length.

Preparation of dye solutions for silk fibers

In this step, iron solutions are prepared at final concentrations of 100, 300, 500, and 1,000 ppm in a total volume of 100 mL, to be mixed with sappan heartwood extract. Additionally, dye solutions are prepared by combining sappan heartwood extract with mud solution and with alum solution. The procedures are as follows:

Pipette 90 mL of sappan heartwood extract into a 250 mL beaker, then pipette 10 mL of iron solution from the 1,000 ppm stock and mix thoroughly. This results in a dye solution with a final iron concentration of 100 ppm (repeat 3 times).

Pipette 70 mL of sappan heartwood extract into a 250 mL beaker, then add 30 mL of the 1,000 ppm stock iron solution and mix. This gives a dye solution with a final iron concentration of 300 ppm (repeat 3 times).

Pipette 50 mL of sappan heartwood extract into a 250 mL beaker, then add 50 mL of the 1,000 ppm stock iron solution and mix. This gives a dye solution with a final iron concentration of 500 ppm (repeat 3 times).

Pipette 90 mL of sappan heartwood extract into a 250 mL beaker, then add 10 mL of mud solution from the 10,000 ppm stock, mixing thoroughly. This gives a dye solution with a final mud concentration of 1,000 ppm (repeat 3 times).

Pipette 90 mL of sappan heartwood extract into a 250 mL beaker, then add 10 mL of alum solution from the 1,000 ppm stock, mixing well. This results in a dye solution with a final alum concentration of 100 ppm (repeat 3 times).

Analysis of element in ban ta long mud

The analysis of the major element components in the mud used for silk dyeing was conducted using an X-ray fluorescence energy dispersive spectrometer (XGT-5200) by HORIBA.

Analysis of structural of sappan heartwood extract

In this step, the structural characteristics of the active compounds in silk fibers and sappan heart wood extract were studied using a fourier transform infrared microscope spectrophoto meter (FT-IR), model tensor 27, by Bruker. Condition: Check Signal (Amplitude) − 1753 Resolution 4 Sample scan time 64 Background scan time 64.

Analysis of color values

In this step, the color values of the silk fibers that were pre-washed before dyeing and the silk fibers dyed with iron ion solutions at concentrations of 100, 300, 500, and 1,000 ppm will be analyzed using the amazon colour Thailand-andrew reay color measurement instrument. The color values will be assessed using the L* a* b* scale. The L* axis indicates lightness, ranging from 0 to 100, where 0 is black and 100 is white. The a* axis describes the color spectrum from green (-a*) to red (+a*), and the b* axis describes the color spectrum from blue (-b*) to yellow (+b*).

Statistical Analysis

In this research, two methods of dyeing silk fibers were compared dyeing over pre-dyed silk fibers with sappan heartwood using iron ion solutions compared to a mud solution. The average color values of the silk fibers dyed with both methods were then compared using the L* a* b* values through independent sample t-test at a significance level of 0.05.

3. Results and Discussion

Types and composition of major elements in the mud of ban ta long

In the study of the type and composition of major elements in ban ta long mud, the EDXRF technique will be used with the primary objective of identifying the cause of the change in the color of the silk fibers from red (brazilein) to purple. The test results are shown in Table 1.

Table 1 The types and composition of major elements in the mud

No.	Elements	Results (%)
1	$Al_2 O_3$	10.19
2	SiO_2	85.40
3	SO_3	0.61
4	K_2O	0.13
5	CaO	0.23
6	TiO_2	0.83
7	Cr_2O_3	0.02
8	MnO_2	0.02
9	Fe_2O_3	2.53
10	ZrO_2	0.04

From Table 1, it can be seen that the most abundant major element is SiO_2 or silica, followed by Al_2O_3 in second place. These elements are known as Kaolinite, which has properties that make fabric soft and form stable, water-insoluble complexes. Additional Kaolinite is resistant to sunlight and heat. Moreover, it was found that the mud contains the heavy metal element Fe_2O_3, which contains Fe^{3+} ions. This is the main cause of the color change of the red silk to purple and the formation of stable, water-insoluble complexes as well [8].

Type and structure of active compounds in sappan heartwood

In this step, the uniqueness of the compounds from sappan heartwood was validated to study the types of important compounds or functional groups with the ability to form complex compounds with dye fixatives. According to a report by Ngamwonglumlert *et al.* [9], the active compound in sappan heartwood is brazilin, which is a colorless compound. The structure of brazilin consists of two aromatic rings, one pyrone ring, and one five-membered ring, and it is classified under the group of compounds known as neoflavonoids. However, due to the structure of the compound, it contains a large number of −OH groups. This group is easily oxidized, leading to the formation of C=O, resulting in a new compound called brazilein, which appears red as shown in Fig. 2. To confirm that the sappan heartwood used in this study contains brazilein, the compound was analyzed using the FTIR technique, as shown in Fig. 3. The findings revealed that at the wave number 3400, there was the−OH group, at 3000 C−H aliphatic, at 1600 C=C, at 1300 C=O and C−H band in the aromatic ring within the wavenumber range of 860–650 cm[9], which also described the structure of brazilein from sappan heartwood.

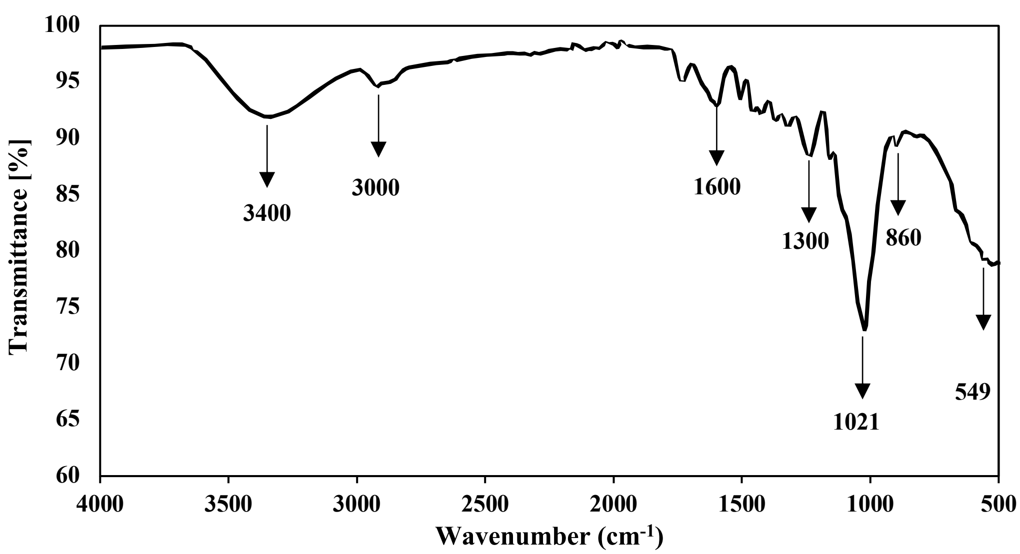
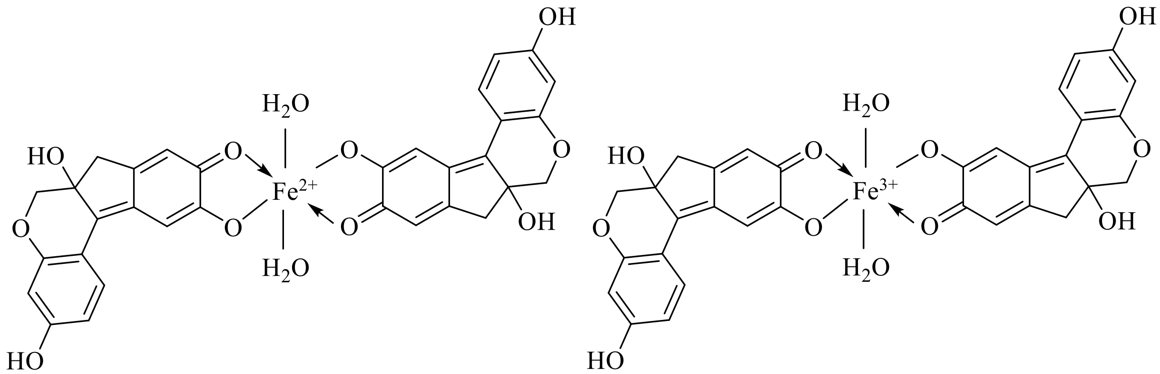


Fig. 3 The types of important functional groups in the structure of the extract from sappan heartwood (brazilein)

According to the research of Petdum *et al.* [10], the study of the complex formation of brazilein with various heavy metal ions, such as Fe^{2+}, Fe^{3+}, Cu^{2+}, Al^{3+}, Ag^{+}, Hg^{2+}, Pb^{2+}, Zn^{2+}, Cr^{3+}, K^{+}, Na^{+}, Li^{+}, Ba^{2+}, Ca^{2+}, Mg^{2+}, Ni^{2+}, Co^{2+}, Mn^{2+}, and Cd^{2+}, revealed that brazilein is most specific to the Fe^{2+} ion and forms a blue-violet complex. In contrast, the Fe^{3+} ion forms a purple-pink complex. The structure of the complex formed between brazilein and the heavy metal ions Fe^{2+} and Fe^{3+} is shown in Fig. 4.



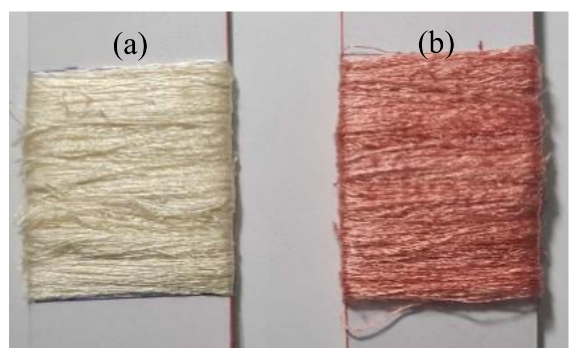
(a) Brazilein–Fe^{2+} (b) Brazilein–Fe^{3+}

Fig. 4 The structural characteristics of the complex compound formed between brazilein with Fe^{2+} (a) and Fe^{3+} (b)

The resulting complex compounds are the cause of the natural color adhering to the silk fiber structure through chemical bonds that are poorly soluble in water [2]. From Figure 5, the mechanism is that iron(III) ions coordinate with the brazilein molecules via oxygen groups from both –OH and C=O functional groups. When this coordination occurs, the electron system within the molecule changes, resulting in light absorption via ligand-to-metal charge transfer (LMCT), which shifts the visible color to purple[11] This coordination also stabilizes the excited state(s) of the molecule, leading to a pronounced change in absorption characteristics, hence the purple–blue coloration observed in the complex. In nature, Fe^{3+} is more prevalent than Fe^{2+} in mineral forms found in soils[12] Research by Jie Chen et al.[13] has shown that silk dyed with iron mordants whether Fe^{2+} or Fe^{3+} exhibits very similar shades when viewed by the naked eye. For this reason, the present study compared silk dyed using Fe^{2+} as a mordant and silk dyed with Fe^{3+}, Fe^{2+} is a commonly used, safer chemical mordant compared to $FeCl_3$ (Fe^{3+})[14]- The findings confirmed that Fe^{3+} in mud solutions clearlyproduces shades aligned with those from Fe^{2+} solutions. [15]

The color value analysis of silk threads dyed with sappan heartwood extract with alum (Al^+) mordant

The results of color analysis for the silk dyed with Brazilwood and alum showed that when compared with the bleached standard silk, the lightness value L* was 60.81, indicating a decrease in lightness. The a* value was a positive 30.90, meaning the presence of red color, and finally, the b* value was the lowest at 13.14, indicating a very slight yellow tone. Therefore, the overall color mixture resulted in red-colored silk, as shown in Fig. 5 and Table 2.



(a) Bleached silk
(b) Sappan heartwood with alum

Fig. 5 The bleached silk (a) and silk fibers dyed with sappan heartwood with alum mordant (b)

Table 2 The color values of silk fibers dyed with sappan heartwood with alum mordant

Types of silk fibers	L*	a*	b*
 Bleached silk	88.11	1.82	20.22
 Sappan heartwood with alum mordant	60.81	30.90	13.14

The color analysis of silk threads dyed with sappan heartwood extract with iron ion solution at various concentration

The results of the color analysis for the silk dyed with sappan heartwood extract with iron ion solution at various concentrations (100, 300, 500, and 1,000 ppm) are shown in Table 3. It was found that the L* value, or lightness of the silk fibers, decreased as the concentration of the iron ion solution increased. The a* value, which indicates the presence of red, decreased, and finally, the b* value, which had a negative value, indicated the development of a darker blue color. All these color shades were observable to the naked eye and are represented by the color values as shown in Table 3.

The color analysis of silk dyed with sappan heartwood with mud solution

The results of the color analysis for the silk dyed with brazilwood with mud solution showed that the L* value was 43.49, a* was 7.90, and b* was 0.17. These values were closest to the silk dyed with iron ion solution at a concentration of 300 ppm, and the color shade was between the iron ion solution concentrations of 300 and 500 ppm, as shown in Table 4. From Table 4, it was found that when calculating the CIELAB values L*, a*, and b*) for the three groups of silk threads dyed with iron ion solutions at concentrations of 300 ppm, 500 ppm, and with a mud solution at 10,000 ppm, the statistical analysis performed using computer software showed significant differences at the 0.05 level.

Table 3 The color values of silk fibers mordanted with iron ion solution

Characteristics of silk fibers	Concentration of iron ion solution	L*	a*	b*
 Bleached silk	-	88.11	1.82	20.22
 Sappan heartwood with alum mordant	100 ppm	53.10	8.12	0.93

Table 3 (Continued)

Characteristics of silk fibers	**Concentration of iron ion solution**	**L***	**a***	**b***
 Iron ion	300 ppm	47.37	9.56	1.79
 Iron ion	500 ppm	32.30	7.04	-3.07
 Iron ion	1,000 ppm	19.33	6.43	-2.24

Table 4 The compared silk dyed with sappan heartwood, mud solution and iron ion solution

Characteristics of silk fibers	**Concentration of iron ion solution**	**L***	**a***	**b***
 Bleached silk	-	88.11	1.82	20.22
 Sappan heartwood with alum mordant	100 ppm	53.10	8.12	0.93
 Iron ion	300 ppm	47.37	9.56	1.79
 Iron ion	10,000 ppm	43.49	7.90	0.17
 Iron ion	500 ppm	32.30	7.04	-3.07

The color value analysis using statistical methods with computer software

The results of the statistical color analysis using the SPSS software revealed that, when observed with the naked eye, the color shade of the silk dyed with mud was most similar to that of the silk dyed with an iron ion solution at a concentration of 300 ppm, showing L*, a*, and b* values that were close to each other. To verify and confirm the accuracy of these experimental results, the hypothesis was tested using computer-based statistical analysis software to compare the L*, a*, and b* values statistically. It was found that the silk dyed with mud showed a significantly different color shade at the 0.05 level when compared to the silk dyed with the iron ion solution at a concentration of 300 ppm. Furthermore, it could be predicted that the mud solution at 10,000 ppm is rich in iron ion elements, with concentrations higher than 300 – 400 ppm.

4. Conclusion

The purple silk fabric offered to the Ban ta long which was dyed with a mud solution, underwent a color change from red to purple due to the influence of iron ions present in the mud. When the mud solution was prepared at a concentration of 10,000 ppm, the amount of iron ions was equivalent to that of a ferrous sulfate solution at concentrations greater than 300 ppm. Which states that iron ion solutions darken the color, turning it towards black or dark gray, in contrast to alum solutions that produce bright colors. Therefore, it can be concluded that the color change of the silk from red to purple resulted from the combination of the red color of brazilein with the iron ions, which contribute a dark or gray tone, thus creating the purple color of the silk. In the future, it will be necessary to study the color shades produced by the iron solution and the mud solution at equal concentrations to re-examine the resulting hues.

5. Suggestion

1. In studies related to silk as local wisdom, consideration should be given to developing practical applications that can genuinely increase or generate income.
2. This research involved an academic synthesis aimed at creating knowledge for community members or interested individuals. It resulted in the production of various textile-based products that add value, generate income, and contribute to long-term sustainable development. Examples include transforming the silk into office shirts, traditional work skirts, and a variety of bags.

6. Acknowledgement

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