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Extraction of natural dye from *Oroxylumb Indicum* (L.) Kurz by using different solvents for Thai eri silk dyeing

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ABSTRACT

Among various textile types in Thailand, silk is the largest market share. Thus, upgrading or developing either the dying process or the quality of fabric dried for Thai silk is still necessary. Natural dyes have become an interesting resource because they are more environmentally friendly than synthetic dyes. Thus, this work aims to study the effect of different solvents for dye extraction from *Oroxylumb Indicum* (L.) *Kurz* on Eri Thai silk dyeing. Each solvent used for extraction gave a different relative tannic acid and quercetin content. Therefore, this further leads to different properties of dyed fabrics, such as color properties and washing fastness index. From the different solvents studied, the yield of the extracted dyes was on the order of EtOH/water (50: 50) > MeOH/water (50:50) > MeOH ~ EtOH > water. Then, the optimal ratios between EtOH and water in the efficient extraction were varied as follows water, EtOH/water (25: 75), EtOH/water (50: 50), EtOH/water (75:25), and EtOH. The result showed that EtOH/water (50: 50) was still the most effective for dye extraction. In addition, the effect of dose, temperature, and time of *Oroxylumb Indicum* (L.) Kurz was 8.0 g/L, 60 °C, and 60 min, which were considered optimal extraction conditions due to the high relative contents of tannic acid and quercetin. As a result, the fabric of Eri silk dyed has a high quality of fastness rating. Overall, these results could point out the use of extracted dye from *Oroxylumb Indicum* (L.) *Kurz* has the potential as an alternative dye for Eri Thai silk dyeing.

Keywords: Oroxylum Indicum (L.) *Kurz*, Ethanol, Water, Natural dye extraction, Eri silk dyeing

INTRODUCTION

Thailand's textile market was valued at USD 8085.0 Billion in 2020 and is projected to reach USD 11097.9 Billion by 2028, growing at a compound annual growth rate (CAGR) of 4.14 % from 2021 to 2028. Among various material types, silk is the largest share of the market [1]. As well as, Thailand is known for its silk, though silk is mainly used for apparel [2]. Thus, upgrading or developing either the dying process or the quality of fabric dried for Thai silk is still necessary.

In general, the majority of the basic ingredients used to color fabrics are synthetic dyes, which take a very long time to degrade [3,4]. Water has been polluted as a result, and the situation has turned into wastewater. Due to the significant amount of organic debris, the outcome might result in a rise in temperature, a strong stench, a color change, and the demise of aquatic life. A shortage of oxygen may also inhibit the activity of some bacteria [3, 5].

People are becoming increasingly interested in natural textile dyes to reduce the issues above since they degrade faster and are safer than synthetic colors [6]. Additionally, natural dyes offer a variety of lovely hues. As well as, Thailand has a wide variety of plants with great potential for producing natural colors.

One should consider that textiles dyed from natural dyes may have fading problems and low resistance to light and convulsions [6]. Thus, some compounds including potassium dichromate, stannous chloride, stannic chloride, ferrous sulfate, cupric sulfate, and aluminum sulfate are used as dyeing agents to make stronger fibers with the dye [7]. These compounds could generate an affinity (electron acceptors) between the dye and fiber with the coordinate covalent bonds

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[8, 9]. However, metallic ions of mordants make them insoluble in water and copper, cobalt, chromium, and lead is not considered environmentally friendly when leached into the environment [10].

To overcome the drawback and deal with a more green chemistry issue, this work discloses the use of available natural dyes and mordants such as tannic acid and quercetin extracted from *Oroxylum Indicum* (L.) *Kurz* for dying silk [10, 11]. These compounds contain a lot of many oxygen atoms and hydroxyl phenolic groups, which could facilitate the creation of hydrogen bonds and/or electrostatic interactions with the fiber, resulting in a good fixation of the dye on the fabric. This remark is possible because the protein and polyamide fibers generate cationic sites in water under acidic conditions resulting in available acid dye anions combining through hydrogen bonding or electrostatic interactions [11, 12]. Besides, *Oroxylumb Indicum* (L.) *Kurz* has been reported as an abundant compound of tannic and alginic acids extracted by water as a solvent [10]. One should be realized that the extraction efficiency of these compounds depends on the kind of solvent system [13]. The solvents with high polarity such as water, methanol, and ethanol would be a good choice for dye extraction containing abundant phenolic group from *Oroxylumb Indicum* (L.) *Kurz* [14]. To our knowledge, there are no reports on the gathering effect of various solvents on the extraction of natural dyes and mordants from *Oroxylum Indicum* (L.) *Kurz.* Besides, it has no commercial value, plentiful in Thailand. Overall, it is worth investigating as an alternative raw material for natural mordant and dye extraction.

Hence, this work aims to investigate the effect of several solvents, including ethanol, methanol, water, ethanol-water, and methanol-water, for extracting natural dyes from *Oroxylum Indicum* (L.) *Kurz* on the dying process. Moreover, some properties of fabric dyed, including color characteristics and color fastness relating to washing, are also discussed.

MATERIALS AND METHODS

Materials and chemicals

Eri silk yarn purchased from the local market of Koh keao Subdistrict, Selaphum District, Roi-Et, Thailand, was used in the present investigation. *Oroxylum Indicum* (L.) bark was from the local area of Roi-Et Rajabhat University, Koh keao Subdistrict, Selaphum District, Roi-Et, Thailand. In order to obtain natural mordants, *Oroxylum Indicum* (L.) bark was extracted by five solvents such as ethanol, methanol, water, ethanol-water, and methanol-water.

Chemicals used in this work were methanol (Lab grade, 99.80% Min) and ethanol (Lab grade, 99.0% Min) were purchased from Giant Leo Intertrade Co.,Ltd., Thailand. Laboratory reagent (LR) grade sodium carbonate obtained from Sigma-Aldrich, and anionic wetting agent (T R Oil, i.e., sulphonated castor oil), non-ionic detergent of the commercial-grade obtained from the local market was used in this investigation.

Degumming of silk

Degumming of silk yarn was performed in a solution containing sodium carbonate or soda ash (0.5 g/L) and non-ionic detergent (2 g/L) at 50 °C for 30 min keeping the material to liquor ratio at 1:30. The degummed yarn was thoroughly washed with cold water and dried at room temperature before dyeing.

Extraction of natural dyes

Oroxylum Indicum (L.) *bark* was peeled and chopped into small pieces, dried in a dry place for 3 - 5 days, and then thoroughly fine blended with a steel blade. An aqueous solution of *Oroxylum Indicum* (L.) *bark* of 0.2 g was added with 10 mL of water. The mixture was stirred, heated, and kept at 60 °C for 60 min in a round bottle flask (50.00 mL) connected to a condenser. Finally, it was filtered through a Whatman® Grade 1 filter paper. The filtrate was used for dyeing after diluting with 100 mL of water. Other solvents such as methanol, ethanol, methanol: water (1: 1), and ethanol: water (1: 1) were carried out with the same procedure mentioned above. Moreover, the appropriate concentration of extracted powder from *Oroxylum Indicum* (L.) bark was studied in the range of 0.1, 0.2, 0.4, 0.6, 0.8, 1.0 , and 1.2 g in 10 mL of water. The appropriate time for extraction was determined by varying in the range of 20, 40, 60, 80, and 100 minutes, as well as the optimum result of extraction temperatures was performed at 30, 40, 50, 60, 70, and 80 °C.

To determine the relative content of natural dyes extracted from *Oroxylum Indicum* (L.), all samples were measured by UV-Visible spectrometer, Hitachi modeled UH5300, at wavelengths of 200 - 800 nm. Before the measurement, the extracted dyes were diluted the volume 100 times with DI water.

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Moreover, the functional group of extracted dye was determined by Fourier transform infrared spectroscopy (FTIR) a PerkinElmer Spectrum™ 10 with ATR mode in the range from 3500 to 550 cm^{-1} with a resolution of 2 cm^{-1}.

Eri silk dyeing

The pre-mordanting of Eri silk thread was adapted from the method in the literature [10]. A sorbent (aqueous solution of soap or wetting agent) of 1.0 g was diluted in 500 ml of water. The solution was stirred homogeneously to wash the thread of Eri silk. The sample was soaked in the solution for 30 min at 100 °C. Finally, the thread of Eri silk was dried at room temperature for 24 h. For dyeing processes, dye baths were prepared according to the method in the literature [11] and the dyeing was performed without the addition of metal salts. Each treatment sample (1.0000 g of Eri silk) was soaked in various concentrations of prepared natural mordants with a mass ratio of 1:10 based on the Eri silk weight to a natural mordant solution at 60 °C for 1 h (flipped the treatment every 10 min for the whole performance). The pH of the dyeing solution was around 5. Dyed Eri silk was washed with 1.0 g/L of neutral detergent in distilled water for 10 min at 30 °C to eliminate excusably natural mordants. After this, the dyed Eri silk was washed in running water and dried at 40 °C for 30 min. Twice repetitions were performed for each dyed Eri silk using various extracted solvents at the optimum condition of dyed extraction.

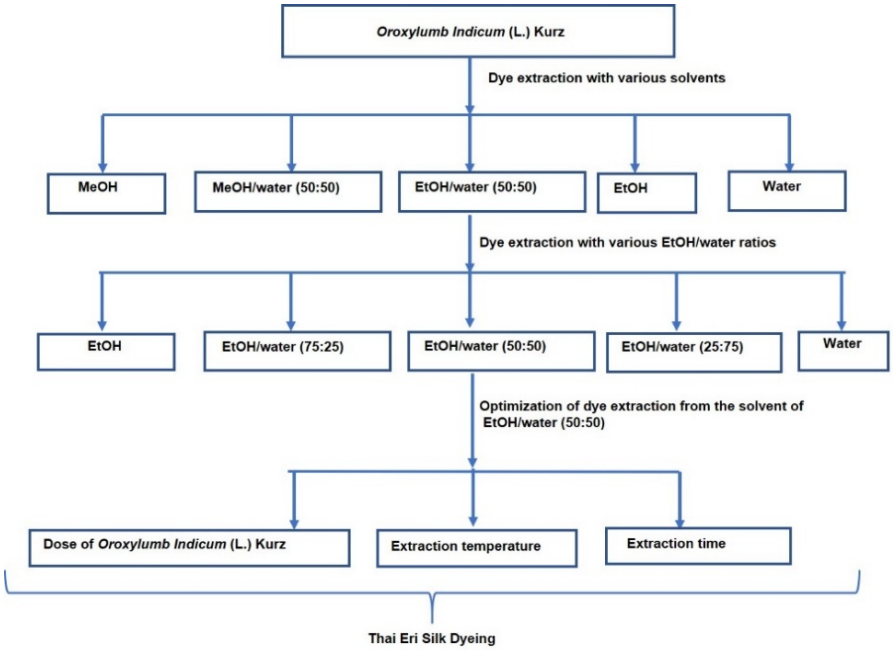
Evaluation of color characteristics

X-Rite Colorimetric analysis was used to measure the CIE L*a*b* values. The operating conditions of the equipment were scanning from 200 to 800 nm, Hunter Lab, modeled MiniScan EZ, and observer angle of 10°. CIE L*a*b* values were analyzed by Analysis of Variance (ANOVA) with a 95% confidence interval and multiple comparisons among means (Tukey's tests). L* is a measure of the lightness/darkness of the dyed Eri silk, which ranges from 100 (white) to 0 (black); a* is a measure of the redness/greenness of the dyed Eri silk, with positive (+) values indicating red and negative (-) values green; and b* is a measure of the yellowness/blueness of the dyed Eri silk, with positive (+) values yellow indicating and negative (-) values blue. The greater the magnitude of the* and b* values, the deeper the colors.

Wash fastness characteristics

The detergent (5.0 g) was dissolved in DI water (1000 mL). Each dyed silk piece was treated in the prepared detergent solution (the weight ratio of dyed silk to the detergent solution, 1:50) at 40 °C for 30 min. The sample was washed with DI water three times and dried at room temperature for 24 h. Then, the wash fastness of the dyed silk samples was measured in Launder-o-meter as per the ISO 105-C06:1994 specifications following the literature [15].

Moreover, to ease understanding, the flowchart of the dye extraction process and optimization is summarized in Scheme 1.



Scheme 1. Diagram of the dye extraction process and optimization in this work.

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RESULTS AND DISCUSSIONS

Physicochemical properties of extracted dye

Figure 1 demonstrates the UV-Vis spectra of natural mordants extracted from *Oroxylum Indicum* (L.) by various solvents. Three crucial bands at 216, 274, and 330 nm corresponded to the characteristic absorptions of castalagin, tannic acid, and quercetin, respectively [11, 16, 17]. This work focuses on tannic acid and quercetin compounds at the wavelengths of 274 and 330 nm because they behave as both natural dyes and mordants for textile dyeing [11]. The chemical structures of both tannic acid and quercetin, containing mainly a type of polyphenol, are demonstrated in Figure 2. The water solubility of both tannic acid and quercetin is 250 g/L and 0.0215 g/L, but the ethanol solubility of both compounds is 100 g/L and 17 g/L, respectively [18].

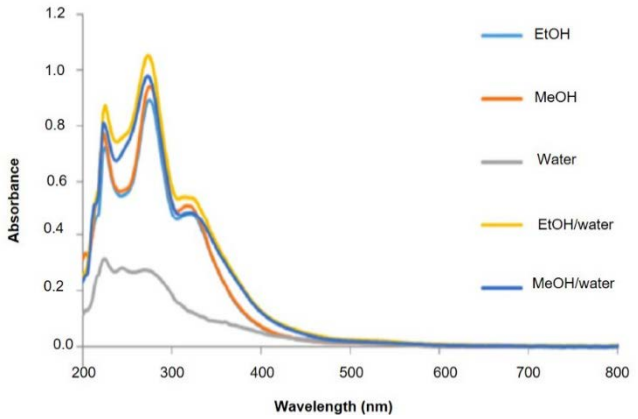

Figure 1 The relationship between absorbance and wavelength of the natural mordants extracted from *Oroxylum Indicum* (L.) bark in different solvents extracted at 60 °C for 60 min.

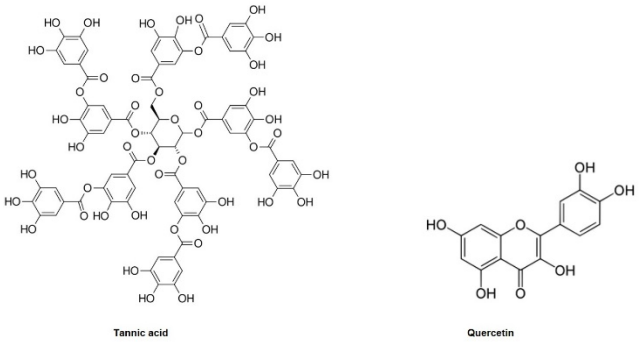

Figure 2 Chemical structures of tannic acid and quercetin.

Moreover, it was noticed that the different solvents used for the extraction provided distinct maxima absorption intensities in the following order EtOH/water (50:50) > MeOH/water (50:50) > MeOH ~ EtOH > water. This result indicates that the co-solvent of EtOH and the water seems the most suitable system for natural mordant extraction.

Figure 3 exhibits FTIR spectra of dye powder extracted using a co-solvent of EtOH/water (50:50). It was observed that there were eleven crucial peaks corresponding to the characteristic functional groups of both tannic acid and quercetin compounds [19, 20]. The bands at 687, 778, and 864 cm^{-1} attributed to =C-H vibrations of arene conjugated to the olefinic group. While the bands at 1048, 1251, 1378, and 1612 cm^{-1} contributed to the vibrations of C-O stretching, C-O-C aryl ketone stretching, C-OH stretching of phenolic hydroxyl group, and C-C stretching of an aromatic ring. Moreover, the bands at 1726, 2172, and 2926 cm^{-1} corresponded to the vibrations of C=O stretching of carboxylate, C-O stretching and C-H stretching, respectively. The broad band at 3219 cm^{-1} was attributed to the vibration of the O-H phenolic hydroxyl group [19, 20]. These character vibrations confirm the presence of both tannic acid and quercetin compounds in the extracted dye from *Oroxylum Indicum* (L.) bark, consistent with UV-Vis results.

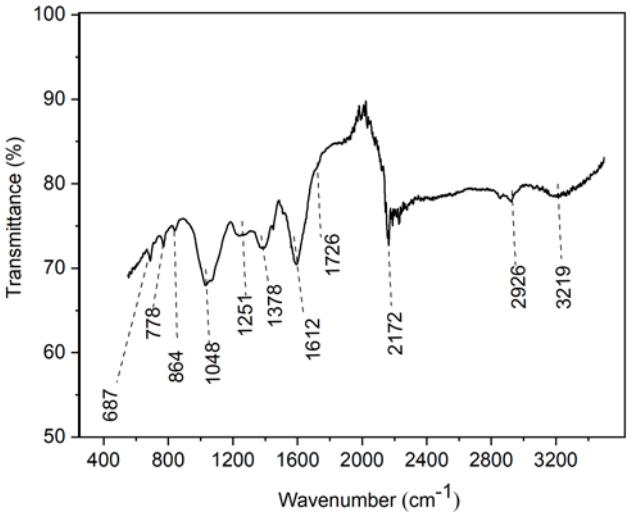

Figure 3 FTIR spectra of dye powder extracted by using a co-solvent of EtOH/water (50:50) at 60 °C for 60 min.

Thus, the co-solvent of water and EtOH system was further selected to optimize extraction capacity with various volume (%) ratios, as illustrated in Figure 4. It was remarked that the extraction efficiency improved with the increase of EtOH content and reached the maximum value at the ratio of 50:50. Then, the decrease in the extraction efficiency was observed with the ratios of 75: 25 and 99% ethanol, respectively. This result suggests that the co-solvent

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with a ratio of 50:50 is the most suitable condition. The consequence might cause by tuning of suitable polarity promoting the extraction efficiency. One should note that tannic acid content is more significant than quercetin in *Oroxylum Indicum* (L.) bark extraction. This is not much surprising because tannic acid is one of the main components in *Oroxylum Indicum* (L.) bark [21]. Moreover, the solubility in water and ethanol of tannic acid is more excellent than that of quercetin resulting in better extraction efficiency.

Besides, It was noticed that maxima absorption intensities of EtOH/water (50:50) than other solvents was probably due to water content in the solvent promoting the swelling of *Oroxylum Indicum* (L.) *bark* pieces. The consequence could increase the contact area between the plant matrix and the solvent, thereby enhancing the extraction yield. Moreover, this result is consistent with the report by Wettasinghe and Shahidi [22] that the co-solvents of water-ethanol (50:50) provide the highest extraction efficiency of flavonoid compounds from *Borago officinalis L.* seeds.

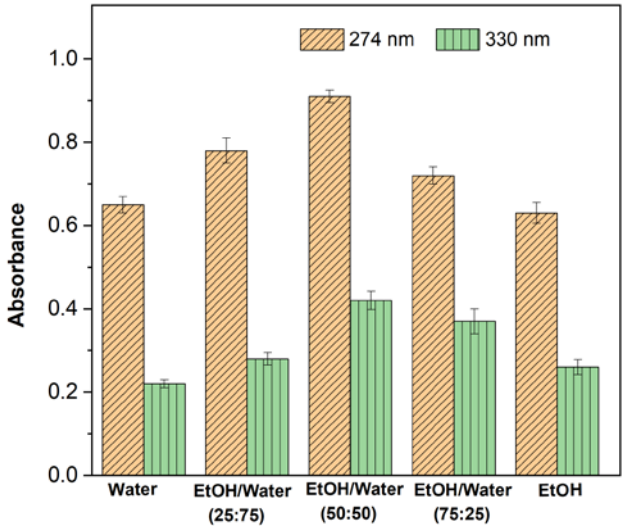

Figure 4 Absorbance of tannic acid and ellagic acid by various water/ethanol ratios at λ (nm) of 274 and 330 nm extracted at 60 °C for 60 min.

Effects of dose, temperature, and time on color intensity for Oroxylum Indicum (L.) *bark extraction.*

Figure 5 reveals the effect of *Oroxylum Indicum* (L.) *bark* dose on the obtained color intensity. The color intensity is significantly enhanced with an increase in *Oroxylum Indicum* (L.) bark powder from 2.0 to 8.0 g/L but almost unchanged when the concentration became 10 g/L and 12 g/L. Thus, the optimum concentration for tannic acid and quercetin extraction from *Oroxylum Indicum* (L.) bark was considered for 8 g/L with absorbance values of 0.83 and 0.31 due to the economic aspect.

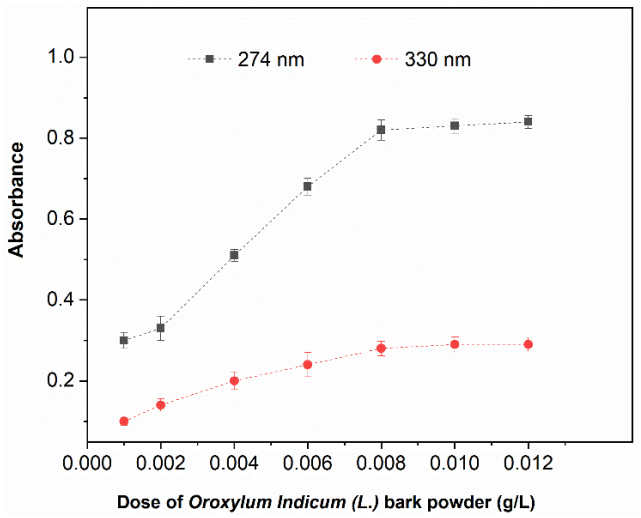

Figure 5 The effect of *Oroxylum Indicum* (L.) bark dose on the color intensity: Extraction condition, a co-solvent of EtOH/water (50:50) at 60 °C for 60 min.

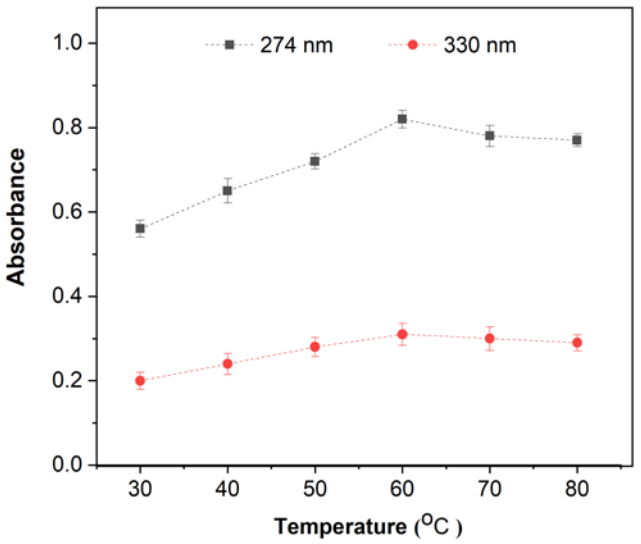

Figure 6 Effects of temperature on color intensity for *Oroxylum Indicum* (L.) bark extraction: Extracted by a co-solvent of EtOH/water (50:50) at various temperatures for 60 min.

Figure 6 displays the effect of extraction temperature on color intensity. The color intensity was dramatically improved with the temperature, from 30 °C to 60 °C. Then, the tendency slightly dropped when the temperature reached 70 °C and 80 °C, respectively. Such a decrease in color intensity of these temperatures could result from the slightly lower ethanol concentration in the liquid phase due to the vaporization effect [23]. The assumption is

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possible because high temperatures, namely, 70 °C and 80 °C, are close and reach the boiling point of ethanol. Thus, the vapor phase of the ethanol becomes dominant in the empty space of the round bottle flask and condenser. The consequence could slightly decrease the ethanol concentration in the liquid phase, consistent with the reported in the literature [23].

Therefore, 60 °C was considered the optimum temperature for further study.

Figure 7 exhibits the effect of extraction time on color intensity. The color intensity was coherently increased with the prolonged extraction time from 20 min to 60 min and became almost constant with times of 70 min and 100 min, respectively. This result suggests that an extraction time of 60 min is the most suitable for optimum conditions. This observation also agrees with the report by Soares and coworkers [23] that the prolonged extraction time could promote the extracted pigments capacity due to greater contact between pigments and solvent, easing the extraction process. Meanwhile, the color intensity decreased after the extraction time. Longer than 60 min may cause by pigment degradation.

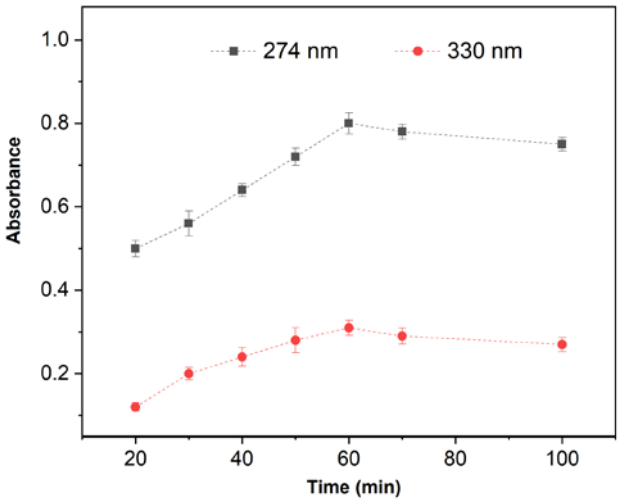

Figure 7 Effects of time on color intensity for *Oroxylum Indicum* (L.) bark extraction: Extracted by a co-solvent of EtOH/water (50:50) with the dose of 8.0 g/L at 60 °C for various times.

Color characteristics of dyed silk

To fairly compare the extracted efficiency based on the solvent effect, the optimum condition for dye extraction in each solvent was a dose concentration of 0.008 g/L, a temperature of 60 °C, and a time of 60 min. Then, the Eri silk was dyed in each solvent with the method adapted from the literature [11].

Figure 8 demonstrates the color appearance of dyed Eri silk with various solvents. The Non-dyed Eri silk had a white color, but the colors of the fabrics dyed became stronger; light yellow (water), yellowish green (EtOH/water, 25:75), dark green (EtOH/water, 50:50), Dark green (EtOH/water, 75: 25), and yellowish dark green (EtOH). The color strength of dyed Eri silk seemed consistent with the increase in the relative content of tannic acid and quercetin compounds [11]. Moreover, different extracted solvents could result in distinct dyed adsorption efficiencies (%) of the fabrics, as illustrated in Table 1. The result showed that the extracted dye with EtOH /water (50: 50) gave the highest adsorption efficiency. Meanwhile, a drastic drop in adsorption was observed in the solvents of EtOH/water (75: 25) and EtOH. This phenomenon is probably due to the more favorable dissolution of tannic acid and quercetin compounds in the ethanol layer rather than the interaction with silk.

Figure 8 Color appearance of dyed Eri silk fabrics in various solvents; (1) non-dyed Eri silk fabrics (2) water, (3) EtOH/water (25: 75), (4) EtOH /water (50: 50), (5) EtOH /water (75: 25), and (6) EtOH.

Table 1 Absorption efficiency (%) comparing pre-dyeing and post-dyeing with various kinds of solvents.

λ_{max} (nm)	Solvent	[a]Absorption efficiency (%)
274	Water	42.1
	EtOH/Water (25:75)	45.9
	EtOH/Water (50:50)	56.7
	EtOH/Water (75:25)	14.6
	EtOH	14.9
330	Water	40.1
	EtOH/Water (25:75)	46.8
	EtOH/Water (50:50)	57.5
	EtOH/Water (75:25)	15.4
	EtOH	16.3

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[a]Calculated by the following equation = (absorption intensity of pre-dyeing - absorption intensity of post-dyeing / absorption intensity of pre-dyeing) × 100

Furthermore, the color data values have altered in the presence of several solvents used in the extraction, as displayed in Table 2. It was found that water-dyed-silk provided the highest L* value of 76.9, indicating a brighter color than ethanol and a mixture of ethanol/water-dyed-silks [11]. All solvents-dyed-silks gave relatively low values of a* ranging from -1.69 to 1.93, suggesting a dominant greenness. While b* values in the range of 26.49 - 17.90 were observed from the silks dyed with water, a mixture of ethanol/water, and ethanol, convincing yellowness [24]. Overall, these results correlate well with the appearance of colors observed by the naked eye.

Colorfastness to washing dyed silks (at 40 °C following ISO 105-C06:1994) from various extraction solvents is demonstrated in Table 3. The fastness rating of the samples dyed with several extracted dye solvents provided slightly different fastness qualities. Silk fabrics dyed with the extracted solvents of water, EtOH/Water (25:75), and EtOH/Water (50:50) were very good fastness ratings (4-5). In terms of silk fabrics dyed with the extracted solvents of EtOH/Water (50:50) and EtOH gave fair to good (3-4) and fair (3), respectively. The greater fastness ratings of silk fabrics dyed with the extracted solvents containing high water content might cause by better absorption efficiency (Table 1). The assumption is rational because flavonoid-containing dyes (quercetin and tannic acid) could facilitate the creation of hydrogen bonds and/or electrostatic interactions with the fiber, resulting in a good fixation of the dye on the fabric [12]. As a result, after dyeing, the flavonoids become insoluble in water during washing, enhancing washing fastness [15].

Table 2 The imparted values of dyed Eri silk fabrics dyed with water, EtOH/water, EtOH.

Solvent	Color Value			Color imparted
	L*	a*	b*	
water	76.99	1.93	26.49	Light yellow
Ethanol/water (25:75)	57.16	-0.45	20.58	Yellowish green
Ethanol/water (50:50)	44.61	-2.75	13.47	Dark green
Ethanol/water (75:25)	40.87	-1.38	17.90	Dark green
Ethanol	49.50	-1.69	17.90	Yellowish dark green

Table 3 Color fastness to washing at 40 °C ISO (105-C06 A1S: 1994).

Solvent	Color staining
Water	4-5
EtOH/Water (25:75)	4-5
EtOH/Water (50:50)	4-5
EtOH/Water (75:25)	3-4
EtOH	3

A comparison of color fastness rating after washing this work to the relevant literature is disclosed in Table 4. It was noticeable that the fastness rating of the present work gave high color staining of 4-5, suggesting a good quality of dyeing fabric, similar to that of the value reported in the literature. This result induces that the extracted dye from *Oroxylum Indicum* (L.) *bark* using the co-solvent of EtOH/Water (50:50) for Thai Eri silk dyeing has a promising strategy for the dyeing process.

Table 4 Color fastness to washing of dyeing silk in the present work compared with the reported value in the literature.

Dye source	Extraction solvent	Fabric	Color staining	Ref.
eucalyptus leaf	water	silk	4-5	[15]
Eucalyptus wood	water	Nylon	4-5	[11]
Calotropis gigantea leaves	water	silk	4-5	[24]
Oroxylumb Indicum (L.) Kurz	EtOH/Water (50:50)	silk	4-5	This work

CONCLUSION

Effect of different solvents for the dye extraction from *Oroxylumb Indicum* (L.) Kurz on the

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Eri Thai silk dyeing was intensively investigated. Each solvent used for the extraction gave different relative contents of tannic acid and quercetin. Consequently, it further resulted in different properties of fabric dyed, namely, color characteristics and washing fastness rating. From various solvents studied, the extracted dye efficiency was in the order of EtOH/water (50:50) > MeOH/water (50:50) > MeOH ~ EtOH > water. Among various ratios of EtOH/water, EtOH/water (50:50) was still the most efficient for dye extraction. Moreover, the effects of a dose of *Oroxylumb Indicum* (L.) Kurz, temperature, and time were 8.0g/L, 60 °C, and 60 min, considered the optimum extraction condition due to providing the high relative content of tannic acid and quercetin. The consequence led to good quality of fabric Eri silk dyed such as an excellent fastness rating.

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