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Research Article

Dyeing Studies with *Eucalyptus*, Quercetin, Rutin, and Tannin: A Research on Effect of Ferrous Sulfate Mordant

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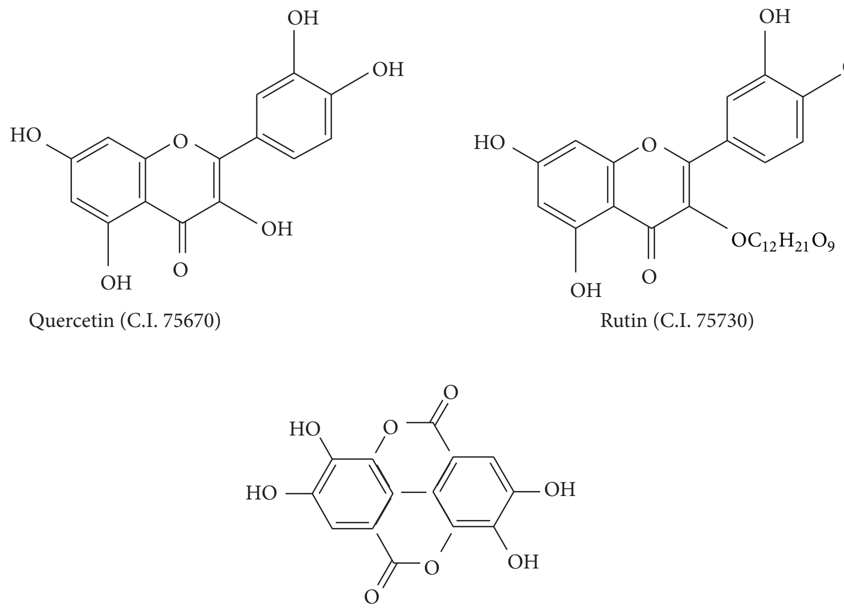
Natural dyes from *Eucalyptus* leaf extract, quercetin, rutin, and tannin were applied to silk fabric by pad-batch and pad-dry techniques under different conditions. Ferrous sulfate was used as a mordant. The dyeing properties were evaluated by measuring *K/S* and *CIELAB* values. In addition, the different fastness properties were evaluated. The effect of dyes at different concentration levels with respect to their colour strength was also studied.

1. Introduction

Natural dyes are comprised of dyes and pigments that are obtained from various parts of plants including roots, bark, leaves, flowers, and fruit [1]. The major applications are coloring of food, leather, wood, and natural fibers like wool, silk, cotton, and flax. Natural dyes may have a wide range of shades. Dyeing with natural dyes, however, normally requires the use of mordants, which are metallic salts of aluminum, iron, chromium, and copper, among others, to ensure a reasonable color fastness to sunlight and washing [2]. The metal ions of these mordants can act as electron acceptors for electron donors to form coordination bonds with the dye molecules, making them insoluble in water [3]. Lately, there has been increasing interest in natural dyes, as the public is becoming more aware of the ecological and environmental problems related to the use of synthetic dyes. The use of natural dyes cuts down significantly on the amount of toxic effluent resulting from synthetic dye processes. Natural dyes have also been used for printing and dye-sensitized solar cells [4–7]. Alongside their aesthetic qualities, natural dyes may offer other benefits, being antibacterial, deodorizing, and UV protective [8–14]. The use of natural dyes in textile applications is growing rapidly, reflecting the strict environmental standards being established in many countries, and the concern about the health hazards associated with synthetic dyes, for example, the recent ban on the use of azo dyes by the European Union, has also increased the scope for the use of natural dyes [15].

Eucalyptus is one of the most important sources of natural dye, yielding several yellowish-brown colorant. *Eucalyptus* has ample natural tannins and polyphenols ranging from 10% to 12% [16]. The major coloring component of *Eucalyptus* bark is quercetin, which is also an antioxidant. It has been used as a food dye with strong antioxidant properties [17]. *Eucalyptus* leaves contain up to 11% of the major components of tannin



Ellagic acid (C.I. 75270)

Figure 1: Color composition of *Eucalyptus* leaf extract dye.

(gallic acid and ellagic acid) with flavonoids (quercetin, rutin, etc.) as minor substances [18–20]. Tannins and flavonoids are considered to be very useful substances during the dyeing process because of their ability to fix dyes within fabrics. The structures of the important coloring components of *Eucalyptus* leaves are given in Figure 1.

Vegetable tannin extracts contain a variety of amorphous materials including polyphenolic tannins of large relative molecular mass (M_r), such as hydrolysable gallotannin, and tannic acid, as well as non-tannins, such as flavones and gums [21]. Because tannins have a large M_r and are water-soluble phenolic compounds, they undergo typical phenolic reactions, notably the chelation of metal ions. Tannins have been used in textiles for several thousands of years, as exemplified by the dyeing of cotton and silk with dyewoods, in which the tannin is "fixed" by a metal salt (e.g., $CuSO_4$) employed as a mordant [22]. Burkinshow [23] used tannins as mordants to increase the uptake of cationic dyes (e.g., mauvein) onto cotton by firstly applying tannin to the cotton, followed by "fixing" of tannin by the application of metal (Fe, Al, Cu, Pb, or Sn) salts.

Flavonoids (polyphenolic pigments) are widely present in plants. Rutin (3,3′,4′,5,7-pentahydroxyflavone-3-rham-no-glucoside) and quercetin (3,3′,4′,5,7- pentahydroxyl-flavone) are phenolic compounds derived from hydroxyl substitutions on a flavone chromophore. Flavone-based compounds are known to form stable complexes with metal cations (Fe, Cu, Al, and Cr) [24]. Flavonoids and tannins are two of the most interesting natural phenolic compounds.

Pure quercetin, rutin, and tannin were also used as colorants in this study in order to compare their dyeing characteristics using *CIELAB* and *K/S* values as well as their fastness properties with those of *Eucalyptus* leaves. $FeSO_4{\cdot}7H_2O$ mordant was used in this work. Our interest lies in the color that these compounds are imparted when added to silk fabric by pad-dry and pad-batch dyeing techniques.

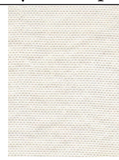
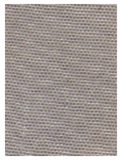
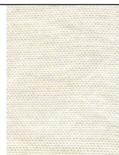
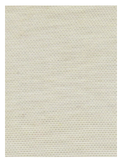
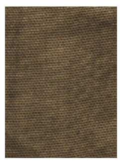
2. Experimental

2.1. Materials and Chemicals. The *Eucalyptus* leaves (*Eucalyptus camaldulensis*) used in this study were collected in Thailand. Quercetin dihydrate, 98% purity ($C_{15}H_{10}O_7{\cdot}2H_2O$, $M_W = 338.80$), rutin hydrate, 95% purity ($C_{27}H_{30}O_{16}{\cdot}xH_2O$, $M_W = 610.52$), and tannin Ph. Eur. 5 ($C_{76}H_{52}O_{46}$, $M_W = 1701.20$) were purchased from Sigma, Fluka and Lachner, respectively. Plain weave scoured and bleached silk fabric was used. The mordant used was ferrous(II) sulfate heptahydrate ($FeSO_4{\cdot}7H_2O$). The anionic wetting agent, Altaran S8 (sodium alkylsulfate), and anionic soaping agent, Syntapon ABA, were supplied by Chemotex Děčin, Czech Republic.

2.2. Instruments. The mordanting and dyeing were carried out in a two-bowl padding mangle machine (Mathis, type number HVF.69805). A spectrophotometer (Datacolor 3890) was used to measure the color strength. The color strength, in terms of K/S values, was calculated using the Kubelka-Munk equation, $K/S = (1 - R)^2/2R$, where R is reflectance of the dyed fabric, K is the sorption coefficient, and S is the scattering coefficient.

2.3. Dye Extraction from Eucalyptus Leaves. Fresh *Eucalyptus* leaves were dried in sunlight for one month and crumbled using a blender. They were then used as the raw material for dye extraction, which was achieved by the reflux technique (70 g of crumbled *Eucalyptus* leaves was mixed with 1 L of distilled water and refluxed for 1 h). The suspension was then filtered and the dye solution was separated into two parts: one

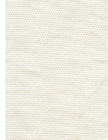
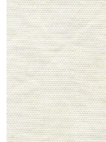
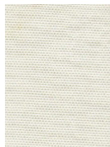
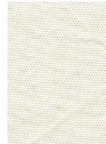
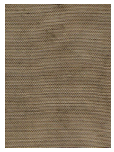
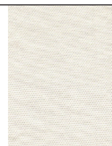
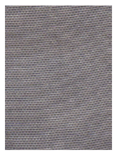
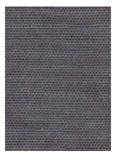
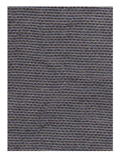
Table 1: Color value of silk fabric dyed with eucalyptus leaf extract and quercetin dyes.

Type of dye	Concentration	Pad-batch					Pad-dry				
		K/S	L^*	a^*	b^*	Dyed sample	K/S	L^*	a^*	b^*	Dyed sample
Eucalyptus	1 g/L dye (without mordant)	0.59	86.5	2.0	−3.6		0.58	86.4	2.1	−3.8	
	5 g/L dye (without mordant)	0.71	85.8	1.4	−1.4		0.66	86.2	1.5	−1.2	
	1 g/L dye + 5 g/L $FeSO_4$	1.54	56.9	2.1	−1.8		1.48	60.9	2.3	−1.6	
	5 g/L dye + 5 g/L $FeSO_4$	1.75	69.1	2.4	−1.2		1.63	58.2	1.9	−1.1	
Quercetin	1 g/L dye (without mordant)	1.17	85.4	−2.2	6.0		1.09	86.3	−1.6	5.1	
	5 g/L dye (without mordant)	1.58	86.3	−3.4	10.2		1.38	87.0	−3.0	8.2	
	1 g/L dye + 5 g/L $FeSO_4$	4.55	46.9	3.5	20.1		4.30	51.9	1.7	15.2	
	5 g/L dye + 5 g/L $FeSO_4$	8.03	36.7	1.4	14.9		7.61	39.8	2.9	16.8	

for evaporating with a rotary evaporator and one for dyeing. The crude dye extract was used for obtaining the standard calibration curve. The dilution of the *Eucalyptus* leaf extract gives a relatively clear solution with a linear dependence on the concentration absorbance and an absorption peak (λ_{max}) at 262 nm [25]. The concentration of 20 g/L was calculated from a standard curve of concentrations versus absorbance at the 262 nm wavelength.

2.4. Mordanting and Pad Dyeing. A simultaneous padding process was used in this research. To study the effect of dye concentration, two concentrations of the *Eucalyptus* leaf extract, quercetin, rutin, and tannin dyes were chosen: 1 and 5 g/L. Ferrous sulfate mordant was used at concentration of 5 g/L for each dye concentration, and 1 g/L of an anionic wetting agent was added to the dye solution. The pH of the dyeing solution was adjusted to 4 using an acetic acid. This pH was adopted from the previous studies [12, 26, 27]. The fabric was then immersed in the dye solution at room temperature and padded on a two-bowl padding mangle at 80% pick up. For the pad-dry method, the samples were dried at 90°C for 3 min. For the cold pad-batch method, the padded fabric was

Table 2: Color value of silk fabric dyed with rutin and tannin dyes.

Type of dye	Concentration	Pad-batch					Pad-dry				
		K/S	L^*	a^*	b^*	Dyed sample	K/S	L^*	a^*	b^*	Dyed sample
Rutin	1 g/L dye (without mordant)	0.71	87.5	0.05	0.1		0.65	87.4	0.8	−1.6	
	5 g/L dye (without mordant)	0.83	86.1	−0.4	2.0		0.76	87.0	−0.1	1.1	
	1 g/L dye + 5 g/L $FeSO_4$	4.10	60.1	1.5	20.0		2.30	69.4	3.3	19.7	
	5 g/L dye + 5 g/L $FeSO_4$	4.73	56.4	1.2	19.2		3.00	63.1	2.3	19.0	
Tannin	1 g/L dye (without mordant)	0.58	86.2	2.0	−3.4		0.56	86.7	2.4	−4.1	
	5 g/L dye (without mordant)	0.78	84.7	0.9	−1.0		0.67	86.8	1.3	−1.0	
	1 g/L dye + 5 g/L $FeSO_4$	2.04	53.0	2.3	−1.8		1.77	54.5	2.1	−2.1	
	5 g/L dye + 5 g/L $FeSO_4$	2.96	40.1	2.9	−4.4		2.81	41.6	4.1	−6.4	

rolled on a glass rod and a plastic sheet wrapped around the rolled fabric. Fabric was then kept at room temperature for 24 hours. The samples were washed in 1 g/L of the soaping agent at 80°C for 15 min and air-dried at room temperature.

2.5. Evaluation of Color Strength and Fastness Properties. The color strength (K/S) and *CIELAB* of the dyed samples were evaluated using a spectrophotometer (Datacolor 3890). All samples showed a λ_{max} value of 400 nm. The color fastness to washing, light, and rubbing of the dyed samples was determined according to ISO 105-C06 A1S: 1994, ISO 105-B02: 1994, and ISO 105-X12: 2001, respectively.

3. Results and Discussion

3.1. Effect of Dyeing on CIELAB and K/S Values. The color value results are presented in Tables 1 and 2. Silk fabrics dyed with *Eucalyptus* leaf extract and tannin dye showed a pale yellowish-grey shade, while those dyed with ferrous sulfate showed a dark greyish-brown color. Silk fabric dyed with quercetin without a mordant had a yellowish-green color, while those mordanted with ferrous sulfate produced a dark yellowish-brown shade. Silk substrates dyed with rutin gave a pale yellowish-green color, while those dyed with ferrous sulfate had a yellowish-brown color.

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

Dyeing and mordanting conditions	Color change	Color staining of multifibers					
		Acetate	Cotton	Nylon	Polyester	Acrylic	Wool
Pad-batch							
Eucalyptus	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Eucalyptus + Fe	4	4-5	4	4-5	4-5	4-5	4
Quercetin	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Quercetin + Fe	4	4-5	4	4	4-5	4-5	4
Rutin	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Rutin + Fe	4	4-5	4-5	4-5	4-5	4-5	4-5
Tannin	4-5	4-5	4-5	4-5	4-5	4-5	4
Tannin + Fe	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Pad-dry							
Eucalyptus	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Eucalyptus + Fe	4	4-5	4-5	4-5	4-5	4-5	4
Quercetin	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Quercetin + Fe	4	4-5	4	4-5	4-5	4-5	4-5
Rutin	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Rutin + Fe	4	4-5	4	4-5	4-5	4-5	4-5
Tannin	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Tannin + Fe	4	4-5	4	4-5	4-5	4-5	4

Note: Fe: $FeSO_4$.

Table 4: Color fastness to light (ISO 105-B02: 1994).

Dyeing and mordanting conditions	Color change	
	Pad-batch	Pad-dry
Eucalyptus	3-4	3-4
Eucalyptus + $FeSO_4$	3-4	3-4
Quercetin	2	2
Quercetin + $FeSO_4$	3-4	3-4
Rutin	3	3
Rutin + $FeSO_4$	3-4	3-4
Tannin	4	4
Tannin + $FeSO_4$	4-5	4-5

From Tables 1 and 2, it is clear that the color shade of the fabrics dyed with tannin (a major constituent of *Eucalyptus* leaves) is colorimetrically and visually observed to be very similar to that one using *Eucalyptus* leaf extract dye. The colors obtained with the different dyes vary in their tone due to the fact that when the dyes (*Eucalyptus* leaf extract, quercetin, rutin, and tannin) are combined with ferrous sulfate to form dye-ferrous complexes, different shades are attained.

Tables 1 and 2 also show the color strength (K/S) values of silk fabric dyed with *Eucalyptus* leaf extract, quercetin, rutin, and tannin, respectively. It can be observed that the K/S values increase with an increase in dye concentration. Silk fabric dyeing by pad-batch technique showed higher K/S values than pad-dry technique. This is because the pad-batch method exposes the fibers to the dye molecules for a longer time period, promoting greater dyeability.

It can be concluded that the successful dyeing of silk fabrics with *Eucalyptus* leaf extract dye, quercetin, rutin, and tannin is due to the formation of ferrous coordination complexes. Ferrous sulfate readily chelated with the dyes. As the coordination number of ferrous sulfate is 6, some coordination sites remain unoccupied when they interact with the fiber, which allows functional groups, such as amino and carboxylic acids, on the fiber to occupy these unoccupied sites. Thus ferrous sulfate can form a ternary complex on one site with the fiber and on another site with the dye [28].

3.2. Color Fastness Properties. The fastness ratings of silk fabric dyed with and without mordants at a dye concentration of 5 g/L and ferrous sulfate (5 g/L) are presented in Tables 3–5. Table 3 indicates that the washing fastness rating of silk fabric dyed with *Eucalyptus* leaf extract, quercetin, rutin, and tannin is very good (4 to 4-5). A probable explanation for the good fastness property is that tannin and flavonoids (quercetin and rutin) can form metal chelates with the ferrous mordant. Hence, after mordanting, the tannin and flavonoids are insoluble in water, which improves the washing fastness.

As seen in Table 4, a light fastness in the range of 3-4 (fair) can be observed in the silk fabrics, except for that dyed with quercetin without mordant, whose rating was 2 (poor). This is attributed to the fact that the presence of 3-hydroxy groups in quercetin reduces the light fastness due to lower photostability [29]. Silk dyed with tannin with or without a mordant was in the range of 4 to 4-5 (good).

From Table 5, very good (4-5) dry rubbing fastness can be observed in silk fabric dyed with *Eucalyptus* leaf extract, quercetin, rutin, and tannin, except for those mordanted with ferrous sulfate, whose ratings were 3 to 4 (fair to

Table 5: Color fastness to rubbing (ISO 105-X12: 2001).

Dyeing and mordanting conditions	Pad-batch				Pad-dry			
	Warp direction Color staining		Weft direction Color staining		Warp direction Color staining		Weft direction Color staining	
	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
Eucalyptus	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Eucalyptus + Fe	3	2-3	3	2-3	3	2-3	3	2-3
Quercetin	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Quercetin + Fe	3-4	3-4	3-4	3-4	3-4	3-4	4	3-4
Rutin	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Rutin + Fe	4	4	4	4	4	4	4	4
Tannin	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Tannin + Fe	3	2-3	3	2-3	3	2-3	3	2-3

Note: Fe: $FeSO_4$.

good). However, the fabrics dyed with *Eucalyptus* and tannin mordanted with ferrous sulfate show a wet rubbing rating of 2 to 3 (poor to fair). This is attributed to a difference in the extent to which the low aqueous soluble ferrous-tannate complexes were able to diffuse within the dyed fiber. The large molecular size complex that was formed within the dyeing bath could be anticipated to display very low diffusional behavior and, therefore, to deposit mostly on the periphery of the dyed fiber, resulting in a low rubbing fastness [21].

4. Conclusions

Silk fabrics dyed with *Eucalyptus* leaf extract, quercetin, rutin, and tannin using the pad-batch method show higher color strength than those dyed with the pad-dry technique. Tannins are considered as a main colorant in dyeing processes because of the shade similarities of *Eucalyptus* leaves. The color fastness to washing showed very good results, whereas the color fastness to rubbing was fair to good, except for silk fabric dyed with tannin mordanted with *Eucalyptus* with ferrous sulfate, where ratings were poor when subjected to wet rubbing. The light fastness rating of the silk fabric mordanted with ferrous sulfate showed a fair to good result, but in the case of the silk fabric dyed with quercetin without mordant, the fastness rating was poor. The application of natural dyes to silk fabric by the pad-batch technique can be considered to be an effective ecooption; hence this technique could be considered to be the most suitable for small scale industry and cottage dyeing.

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