RESEARCH ARTICLE

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Studies on Dyeing of Silk Yarn with Lac Dye: Effects of Mordants and Dyeing Conditions

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ABSTRACT The effects of mordanting method, dyeing temperature and time, as well as the type of mordants used in lac-dyeing process on the properties of silk yarn were studied. Three different mordanting methods, including pre-mordanting, simultaneous mordanting, and post-mordanting, were employed and the amount of adsorbed dye, the quality, and the breaking strength values of silk yarn were compared. Various natural products and chemicals were also used as mordants and they provided finished silk products of different color quality and breaking strength.

KEYWORDS: natural lac dye, silk, mordant.

INTRODUCTION

Lac dye (CI Natural Red 25; CI 75450) is obtained from an insect *Coccus laccae* (*Laccifer lacca* Kerr), found on the twigs of certain tree native to Southeast Asia. The insect produces a resin known as stick lac.[1] Lac dye or laccaic acid has two major compositions: laccaic acid A and B whose structures are shown in Figure 1 and 2, respectively.[2-4] Laccaic acid represented approximately 0.5-0.75% by weight of stick lac.[5]

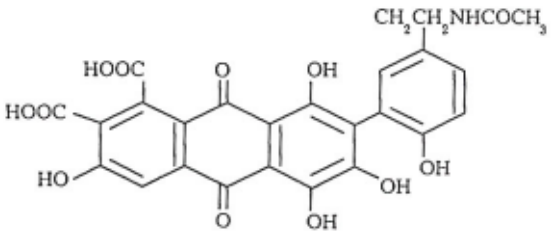

Fig 1. Structure of laccaic acid A.

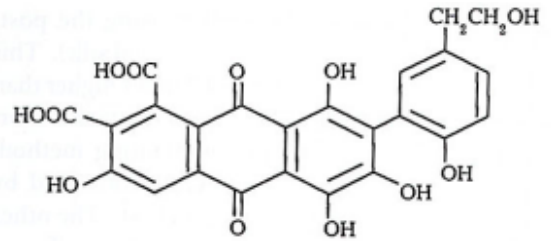

Fig 2. Structure of laccaic acid B

Silk fiber is protein fiber that is produced from silk worms. It is composed of different alpha amino acids orienting to form long chain polymer by condensation and polymerization. Silk fiber consists of 97% protein and the others are wax, carbohydrate, pigments, and inorganic compounds. The proteins in silk fiber are 75% fibroin and 25% sericin by weight, approximately.[6] The sericin makes silk fiber to be strong and lackluster, therefore, it must be degummed before dyeing.

Most of natural red-color dyes have high solubility in water, therefore, the color fastness to washing of the dyed fabric is quite low. In order to improve its color fastness quality most of dyeing processes were conducted using metal salts (eg, potassium dichromate, stannous chloride, ferrous sulfate and copper sulfate) as mordants.[7-16] The metal ions can act as acceptors to electron donors to form co-ordinate bonds with dye molecule, which is insoluble in water.[17] The reported color fastness to washing are in the range of 3 to 5 for color change and 3-4 to 5 for staining, depending on type of dye and mordant used. However, the wastewater containing heavy metal ions from these mordants may affect the environment and public health. Offering an alternative way of natural dyeing and avoiding the hazardous problems, this work chose some natural products and mild chemicals instead of metal salts to be used as mordants for lac dyeing on silk yarn. Some of them, especially memecylon (*Memecylon Scitellatum* (Lour) Naud), have been used since

ancient times in the northeastern part of Thailand. In addition, the effects of mordanting methods, type of mordants, as well as dyeing temperature and time on the quality of dyed silk yarns were investigated and reported.

Materials and Methods

General procedure

The silk yarn was dyed using the dyeing machine SP110 (UGOLINI srl, Italy) with programs to control temperature, time, and speed of circulation of solution. The dyed silk samples were tested and certified by the Textile Industry Division, Ministry of Industry for color fastness to washing (Laundry-O-meter), fastness to light (Xenon Weather Meter), and breaking strength (Constant-Rate-of-Extension) followed the Standard Test Method for Textiles, Thai Industrial Standards Institute (TISI): TIS 121 Part 3-2518 (1975), TIS 121 Part 2-2518 (1975), and TIS 121 Part 8-2518 (1975) with practical limits of error of ±3% at confidence level of 95%, respectively.

Silk and dye

The used silk yarn is a hybrid-race silk (Chul 5) produced by Chul Thai Silk Co, Ltd and stick lac used was obtained from Banquaw district, Chaiyaphum province, Thailand. The silk yarn was degummed by treating in a chip-soap solution. After washing and drying, the silk yarn (which weighed about 25% less) was ready for dyeing. One kilogram of stick lac was ground into coarse powder. Ten liters of water were added and the solution was stirred and left standing for 24 hours. After filtration, a red solution of lac dye was obtained. The lac dye solution was kept cold for further use.

Mordants

Both natural products and chemicals were employed as mordants in silk dyeing with natural lac dye. The mordants are used as follows: memecylon, tamarind, 0.1 M formic acid, 0.5 M acetic acid, 0.5 M tartaric acid, and 0.1 M alum. To prepare the mordant solutions from memecylon, 500 g of memecylon was boiled in 2 liters of water for 90 minutes, the mixture was then filtered and a yellow-green solution was obtained. The mordant solution from tamarind was prepared in a similar manner except only 300 g of tamarind was used, and a yellow-brown solution was obtained. These solutions were kept cold for further use.

Effects of mordanting methods

Silk yarn (20 g) was dyed in a 2-liter solution of lac dye and memecylon (3:1 v/v) at 100 °C and motor speed of 750 rpm for 60 minutes. Three different methods of mordanting were used: (1) pre-mordanting method: the sample was first mordanted and then dyed; (2) simultaneous mordanting method: the sample was treated with the dye and a mordant at the same time; and (3) post-mordanting method: the sample was first dyed and then mordanted. The concentration of the remaining dye solution and the amount of dye adsorbed on the silk fiber were calculated from the measured absorbance of dye solution at 520 nm (UV-Vis spectrophotometer, UV-160A, Shimadzu) before and after dyeing. The dyed silk samples were tested for color fastness to light, fastness to washing, and breaking strength.

Effects of dyeing temperature and time

Silk yarn (20 g) was dyed in a 2-liter solution of lac dye and memecylon as previously described. The dyeing temperature was at 60, 80, and 100 °C and the time was varied from 30 to 240 minutes. The dyed samples were then tested for color fastness to light, fastness to washing, and breaking strength.

Effects of type of mordants

Different mordants including memecylon, tamarind, a mixture of memecylon and tamarind (1:1 v/v), formic acid, acetic acid, tartaric acid, and alum were used in lac dyeing of silk fiber using the post-mordanting method. The silk fiber was first dyed with a solution of lac dye at 100 °C with a motor speed of 750 rpm for 60 minutes, then with a solution of mordant for 30-50 minutes. The dyed samples were next tested for color fastness to light, fastness to washing, and breaking strength. The finished colors of dyed samples were also compared using a spectrophotometer (Spectraflash 500).

Results and Discussion

The effects of mordanting methods on silk dyeing with a solution of lac dye and memecylon are shown in Table 1. It is evident that the amount of lac dye adsorbed on silk is highest when using the post-mordanting method (22.950 mg dye/g of silk). This amount is approximately 1.5 and 2 times higher than values obtained when using the simultaneous mordanting method and the pre-mordanting method, respectively. The breaking strength obtained by simultaneous mordanting is the highest. The other properties of the silk samples seem to be unaffected by mordanting methods.

Dyeing temperature and time are also important parameters influencing the quality of finished dyed samples. It is known that dyeing silk at high temperature for a long period of time tends to decrease the strength of silk fiber. Therefore, silk samples were dyed with a solution of lac dye and memecylon using simultaneous mordanting at the temperature ranging from 60 to 100 °C for 30 to 240 minutes and the quality of finished dyed samples are compared in Table 2. Although post-mordanting gives the best result, simultaneous mordanting is the most commonly used method of mordanting in the northeastern part of Thailand. Therefore, the effects of dyeing temperature and time with simultaneous mordanting were conducted.

Table 1. Effects of mordanting methods on the amount of dye adsorbed on silk, the quality, and the breaking strength values of silk yarns dyed with a solution of lac dye and memecylon.

method	dye on silk (mg/g silk)	quality level				breaking strength (g_f)
		fastness to washing			fastness to light	
		staining on standard cloth		color change		
		silk	cotton			
pre-mordanting	11.096	4-5	5	3	4-5	1178.6
simultaneous mordanting	15.532	4-5	5	3	5	1286.5
post-mordanting	22.950	4-5	5	3	5	1186.3

note: Quality level from 1 to 5 (5 means excellent).

Table 2. Effects of dyeing temperature and time on the quality of silk dyed with a solution of lac dye and memecylon.

dyeing condition		fastness to washing			fastness to light
		staining on standard cloth		color change	
temperature (°C)	time (minutes)	silk	cotton		
60	30	5	5	1-2	3-4
	40	5	5	3	4
	50	5	5	2-3	3
	80	5	5	1-2	3-4
	110	5	5	2	3-4
	140	4-5	5	2-3	3-4
	170	5	5	2-3	3-4
	200	5	5	2-3	3-4
	240	5	5	2-3	3-4
80	30	5	5	2-3	3
	40	5	5	3	3-4
	50	5	5	3-4	3-4
	70	5	5	1-2	3-4
	90	5	5	2	3-4
	110	5	5	2	3-4
	130	5	5	2	3-4
	150	5	5	2-3	3-4
100	30	5	5	2-3	3-4
	40	5	5	3	4
	50	5	5	3-4	3
	70	5	5	2	3-4
	90	5	5	2-3	3-4

note: Quality level from 1 to 5 (5 means excellent).

It can be seen that almost all silk samples have excellent quality in staining on standard cloth (level 5). The results show that neither dyeing temperature nor dyeing time has an effect on quality in staining; however, the dyeing time seems to affect the color change and fastness to light to some extent. Dyeing silk at 60-100 °C for longer than 40-50 minutes gives no further improvement in color change and fastness to light, as the quality level is slightly decreased after 40-50 minutes of dyeing.

Further experiments were performed to study the effects of dyeing temperature and time on breaking strength of dyed silk. It is found that dyeing temperature (60-100 °C) and dyeing time (30-90 minutes) have slight effects on breaking strength as shown in Tables 3 and 4, respectively. It should be noted that variation of the obtained breaking strength might be caused by the inconsistent diameter of the yarn, which is the general characteristic of Thai silk.

Different mordants, including both natural products and chemicals, were employed in the lac dyeing of silk using the post-mordanting method. It is clear that type of mordants has some influence on the quality level and the breaking strength of the dyed silk samples as well as the color of the finished samples (Tables 5 and 6). Comparing to the silk sample dyed without any mordant, samples dyed with 0.1 M acetic acid as a mordant show the highest overall breaking strength values while samples dyed with 0.5 M tartaric acid show the lowest overall breaking strength values. Moreover, silk samples dyed with tamarind and with a mixture of memecylon and tamarind (1:1 v/v) show the highest overall quality of finished silk samples in terms of color change and fastness to light. Even though the samples dyed with a mixture of memecylon and tamarind show slightly better quality level, they have lower breaking strength values than those dyed with only memecylon or tamarind as a mordant.

Table 3. Effect of dyeing temperature on breaking strength of silk dyed with a solution of lac dye and memecylon for 40 minutes.

temperature (°C)	breaking strength (g_f)
60	1210.2
80	1237.3
100	1189.5

Table 4. Effect of dyeing time on breaking strength of silk dyed with a solution of lac dye and memecylon at 100 °C.

time (minutes)	breaking strength (g_f)
30	1256.0
40	1189.5
50	1277.5
70	1286.7
90	1216.8

The type of mordants also has an effect on the finished color of the silk samples as shown in Table 6. The use of mordants for lac dyeing of silk decreases the color brightness of the finished products. The redness of color (positive a* value) and the clarity of color (positive C* value) also increase with the use of all mordants. The use of memecylon as a mordant gives the highest redness value of 35.06 and the use of tartaric acid solution gives the highest clarity of color of 37.39. Additionally, different shade of color can be obtained by changing the type of mordants, eg, memecylon or the mixture of memecylon and tamarind gives a red color; tamarind, formic acid, acetic acid, or tartaric acid gives a red-orange color, and alum gives a purple color.

Conclusions

This research demonstrated that the mordanting method used in silk-dyeing process with natural lac dye and memecylon as a mordant influences the amount of adsorbed dye on silk yarn, with the post-mordanting method showing the highest amount of adsorbed dye. However, the mordanting method has no significant effect on other physical properties such as color quality or breaking strength. The effect of dyeing temperature or dyeing time is found to be insignificant on the dyeing process being studied. The type of mordants, on the other hand, shows major influences on the properties of finished silk samples (such as fastness to washing, fastness to light, shade of color, and breaking strength of the silk yarn). Different shades of color can be obtained on the silk yarn by changing the mordant used.

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Table 5. Effects of type of mordants on the quality and the breaking strength values of silk dyed at 100 °C.

mordant	dyeing time (minutes)	quality level				breaking strength (g_f)
		fastness to washing			fastness to light	
		staining on standard cloth		color change		
		silk	cotton			
none	40	4-5	5	4	4-5	1149.8
memecylon	30	5	5	2-3	3-4	1256.0
	40	5	5	3	4	1189.5
	50	5	5	3-4	3	1277.5
tamarind	30	3-4	4-5	4	4-5	1195.0
	40	4	4	3	4-5	1056.9
	50	4-5	4	3	4-5	1091.1
memecylon + tamarind	30	4-5	5	4-5	4-5	976.0
(1:1 v/v)	40	5	4-5	3	4-5	1134.0
	50	4-5	4-5	3-4	4-5	946.8
0.1 M formic acid	30	4-5	4	1-2	4	972.4
	40	4-5	3-4	3-4	4-5	1093.1
	50	4-5	4-5	3	4	1520.2
0.5 M acetic acid	30	4	4	3	4	1609.1
	40	3-4	4	3	4	1225.8
	50	4-5	4	3	4-5	1365.9
0.5 M tartaric acid	30	4	4-5	2-3	4-5	833.8
	40	4-5	4	1-2	4-5	1089.5
	50	4-5	4	1-2	4-5	1165.6
0.1 M alum	30	4-5	5	3-4	2	1155.9
	40	4-5	4-5	1-2	2	1138.3
	50	5	4-5	1-2	2	1361.1

note: - Quality level from 1 to 5 (5 means excellent).

Table 6. The finished colors of silk samples dyed at 100 °C for 40 minutes with different mordants, measured with a spectrophotometer (Spectraflash 500).

mordant	color values				
	L*	a*	b*	h*	C*
none	78.62	2.40	4.64	62.23	5.24
memecylon	40.15	35.06	11.56	19.26	35.84
tamarind	44.31	28.43	19.56	34.56	34.53
memecylon + tamarind	41.58	33.76	11.27	18.46	35.59
0.1 M formic acid	38.34	24.04	14.88	31.76	28.28
0.5 M acetic acid	42.20	23.24	13.93	30.94	27.09
0.5 M tartaric acid	47.10	30.63	21.44	34.98	37.39
0.1 M alum	53.24	21.14	1.78	4.81	21.22

note: The CIE L* a* b* (CIELAB) system was used for identifying the color of silk samples.

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