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Optimizing the Dyeing of Silk Fabric with Gemstone Powder using Exhaustion Process

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The silk fabrics were modified by a cationic reagent, *N*-(3-chloro-2-hydroxypropyl)trimethylammonium chloride in order to prepare the fiber to be dyed with gemstone powder (*i.e*. malachite, lapis lazuli and jasper) by an exhaustion process. The effect of dyeing conditions such as temperature, dyeing time, pH and gemstone powder concentration on colour strength were investigated. Optimum results were achieved when dyeing at a liquor ratio of 1:100 at pH 3 (for malachite and jasper) or pH 5 (for lapis lazuli), at 90 ºC for 60 min. The color fastness to crocking of cationic treated silk fabric dyed with gemstone powder was fair to good, whereas the color fastness to washing was poor to fair. However, the colour fastness to light was very good. This study demonstrated that cationic treated silk fabric can dyed with gemstone powder by exhaustion process.

Keywords: Gemstone powder, Exhaustion, Dyeing, Silk, Dyeing property.

INTRODUCTION

Gemstones have been mined and cherished since pre-history [1,2]. Gemstones are natural inorganic minerals that are used as precious stones in jewelry or accessories. The annual value of worldwide trade in rough stones (excluding jade) is estimated at US $ 2 to 3 billion [3]. Thailand is recognized as one of the world's major gem and jewelry centers. In recent years, Thailand has developed into one of the five leading gem cutting centers along with Belgium, India, Israel and the United States. The cutting skills of Thai craftsmen have improved greatly over time, developing the world renowned "Bangkok cut" [4,5]. However, a large amount of residue gemstone powder is created during jewelry production from settled sediments from machines used to cut and polish gemstones. The utilization of residue gemstone powder, therefore, offers attractive opportunities to add economic value through use of this gemstone residue.

Silk fabric is a well-known natural clothing material, often called "queen of textiles" because of its luster, luxury appeal, comfort, elegance, glamour and sensuousness [6-9]. Silk′s natural beauty, softness, superior wear comfort and retention of warmth during winters have made it a desirable fiber for high-fashion. Silk consists of a large number of amino acids of the general formula $NH_2CHRCOOH$, which has end amino and end carboxylic groups in its polymer molecule, and, thus, forms salt linkages with dye anions or dye cations [10]. Silk is also dyeable with cationic dyes such as acid, direct, metal complex, acid mordant and reactive dyes. Traditionally, silk is also dyed with natural dyes [11-16]. However, it has been reported that silk fabric dyeing with pigments by exhaustion process is possible by imparting substantivity with cationic auxiliary to induce the necessary affinity between pigments and fiber [17-19].

The present research investigated silk fabric dyeing with gemstone powder by use of exhaustion method. Before the dyeing process, silk fabrics were pretreated with cationic reagent. These cationic pretreated silk fabrics were then dyed with three anionic gemstone powders, *i.e*. malachite, lapis lazuli and jasper. Different factors affecting dyeing ability were examined.

EXPERIMENTAL

Silk fabrics (150 g/m^2) purchased from a local fabric store were degummed with a sodium carbonate solution. *N*-(3-chloro-2-hydroxypropyl)trimethylammonium chloride (CHTAC), cationic reagent, were purchased from Sigma-Aldrich. Other chemicals in this study *viz*. sulphuric acid and sodium hydroxide were purchased from Sigma-Aldrich. Three gemstone powders *i.e.* lapis lazuli ($Na_6Ca_2(Al_6Si_6O_{24})(SO_4,S,S_2,S_3,Cl,OH)_2$), jasper ($SiO_2$) and malachite ($Cu_2CO_3(OH)_2$) with a particle size of 63 microns were used as the pigments for the silk fabric dyeing.

Cationic pretreatment of silk fabric: Cationic pretreatment of silk fabrics was carried out using the exhaustion method. The cationic reagent (*N*-(3-chloro-2-hydroxypropyl)trimethylammonium chloride, CHTAC) was used at concentration of 2 g/L with a liquor ratio of 1:100. The pH value was then adjusted to 8. The silk fabrics were treated at 80 ºC for 30 min. After treatment, treated samples were rinsed with cold water and then dried at room temperature.

Dyeing of silk fabric: Four different dyeing conditions were varied (temperature, dyeing time, pH, and gemstone powder concentration) to study the dye uptake behaviour of gemstone powder on silk fabrics.

To investigate the effect of dyeing temperature, silk fabrics were dyed at six different temperatures, *i.e*. 40, 50, 60, 70, 80 and 90 ºC using gemstone powder concentration of 1 g/L at a liquor ratio of 1:100 and at the original pH (=7) for 60 min.

Silk fabrics were dyed in four set of the 1 g/L of gemstone powder concentration at 90 ºC, at a liquor ratio of 1:100 and pH 7, for different time intervals (30, 40, 50, and 60 min).

Silk fabrics were dyed in 1 g/L of the gemstone powder concentration in dyebaths at different pH values 3, 4, 5, 6 and 7, at a liquor ratio of 1:100, at 90 ºC for 60 min. The pH of dyebaths was adjusted to the desired values with sulphuric acid and sodium hydroxide solution.

The gemstone powder concentration was varied at 1, 3, and 5 g/L. Silk fabrics were dyed at a liquor ratio of 1:100 and pH 3 (for japer and malachite) and pH 5 (for lapis lazuli), at 90 ºC for 60 min. After dyeing, dyed samples were rinsed with cold water, soaped with 1 g/L of non-ionic detergent at a liquor ratio of 1:40, at 90 ºC for 10 min and finally rinsed with cold water and dried at room temperature.

Testing and measurement: To evaluate dyeing performance, the colour strengths (K/S) of the dyed samples were measured using a spectrophotometer (UltraScan VIS) with illuminant D65 and 10º observer. The K/S values of the dyed samples were calculated using the Kubelka-Munk equation [20]:

$$\frac{K}{S}=\frac{(1-R_{\lambda_{max}})^2}{2R_{\lambda_{max}}}$$

where K = absorption coefficient, S = scattering coefficient and $R_{\lambda max}$ = reflectance value of the dyed samples at the wavelength of maximum absorbance. K/S values were taken at λ_{max} 530, 480, 700 nm for malachite, lapis lazuli, and jasper, respectively.

The wash fastness properties of dyed samples were evaluated according to AATCC test method 61-2010 Test no. 1A. To evaluate light fastness, the dyed fabrics were exposed to a Xenon-Arc lamp according to ISO-B02: 2013. Dry and wet crock fastness of dyed fabrics were tested according to the AATCC test method 8-2007.

RESULTS AND DISCUSSION

Effect of dyeing conditions: In alkaline condition, the chlorohydroxypropyl group of CHTAC converts to an epoxy form 2,3-epoxypropyltrimethylammonium chloride (ETAC), which reacts with the nucleophlilic amine group in silk [17]. Therefore, cationic pretreated silk fabric can reacts with the anion of gemstone powder by bond.

Dyeing silk fabrics in the gemstone powder dye solution using exhaustion process showed that increasing the temperature increased the colour strength ((K/S value) for all of the different gemstone powders. Based on Fig. 1, maximum colour strength was obtained at 90 ºC. The colours of dyed silk fabrics were light green, blue and reddish-brown for malachite, lapis lazuli, and jasper, respectively.

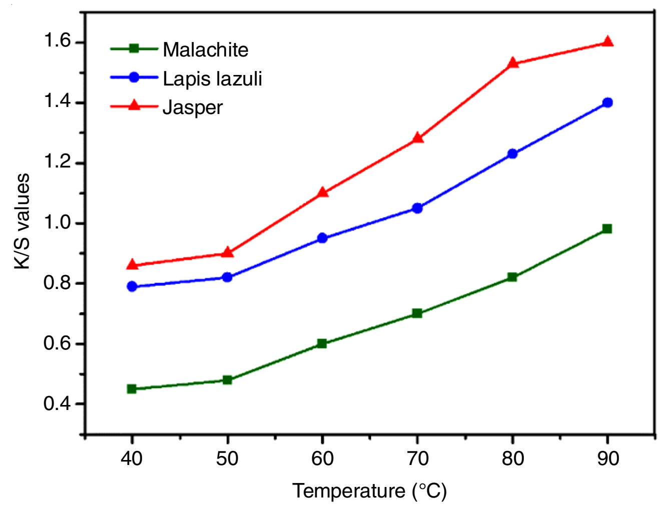

Fig. 1. K/S values with varying temperature from 40 to 90°C at gemstone powder concentration of 1 g/L, liquor ratio of 1:100, and pH 7 for 60 min

The effect of dyeing time on K/S values is illustrated in Fig. 2. A longer dyeing time results in higher colour strength (K/S value) until dye exhaustion attains equilibrium, and there

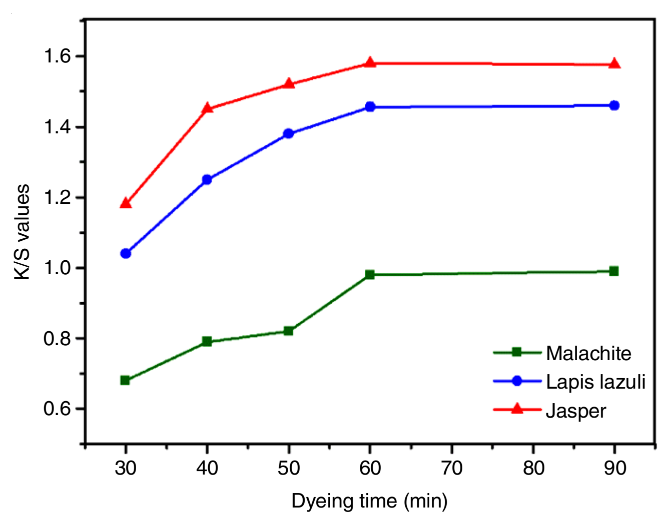

Fig. 2. K/S values with varying dyeing time from 30 to 90 min at 90 °C, gemstone powder concentration of 1 g/L, liquor ratio of 1:100, and pH 7

is no significant increase associated with further increases in dyeing time. The optimum time for dyeing the silk fabrics was obtained at 60 min.

The effect of pH values on K/S values is shown in Fig. 3. The maximum dye uptake was observed at pH 3 for malachite and jasper, while for lapis lazuli, dye uptake increased from the initial pH 3 until pH 5, and then decreased at higher pH values. In acid solution, the H^+ ions protonate the silk fiber′s amino groups, making them cationic. Anionic gemstone powder will dye fibers with cationic sites and are usually substituted for ammonium ions (NH_3^+) in silk. Dyeing involves exchange of the anion associated with an ammonium ion in the fiber, with a dye in the bath [21,22]. The reaction of silk fiber with acid dye is shown in Fig. 4. In alkaline solution, reaction with hydroxyl ions (OH^-) converts the ammonium ion (NH_3^+) to amino (NH_2) groups and the silk fiber contains more carboxylate ion (COO^-) [23,24]. Therefore, electrostatic repulsion between the anionic gemstone powder and the silk fiber occurs, which leads to a decrease in the dye uptake.

Fig. 5 demostrates the K/S values of silk fabrics dyed with gemstone powder dye solution at different gemstone powder concentrations. The K/S values increased with an increase of gemstone powder concentration. However, higher gemstone powder concentrations in the dye solution (content of gemstone powder more than 3 g/L) appeared to induce the formation of larger aggregates of gemstone powder on the surface of silk fabric, which restrict to the penetration property and substantivity of gemstone powder. The silk fabrics dyed with gemstone powder dye solution are shown in Fig. 6.

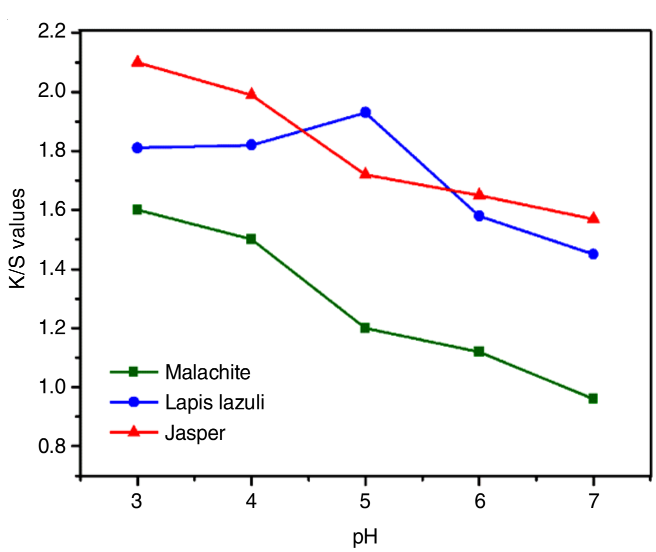

Fig. 3. Values with varying pH from 3 to 7 at gemstone powder concentration of 1 g/L, liquor ratio of 1:100, pH 7, and 90°C for 60 min

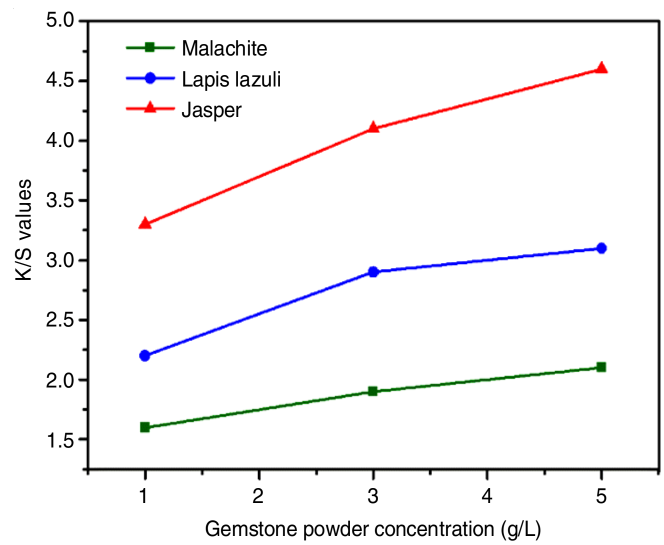

Fig. 5. K/S values with varying gemstone powder concentration from 1 to 3 g/L at a liquor ratio of 1:100, pH 3 (for malachite and jasper) and pH 5 (for lapis lazuli), and 90 °C for 60 min

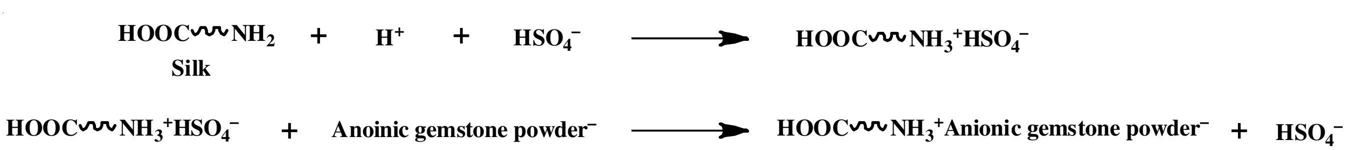

Fig. 4. Reaction of anion gemstone powder with silk fiber

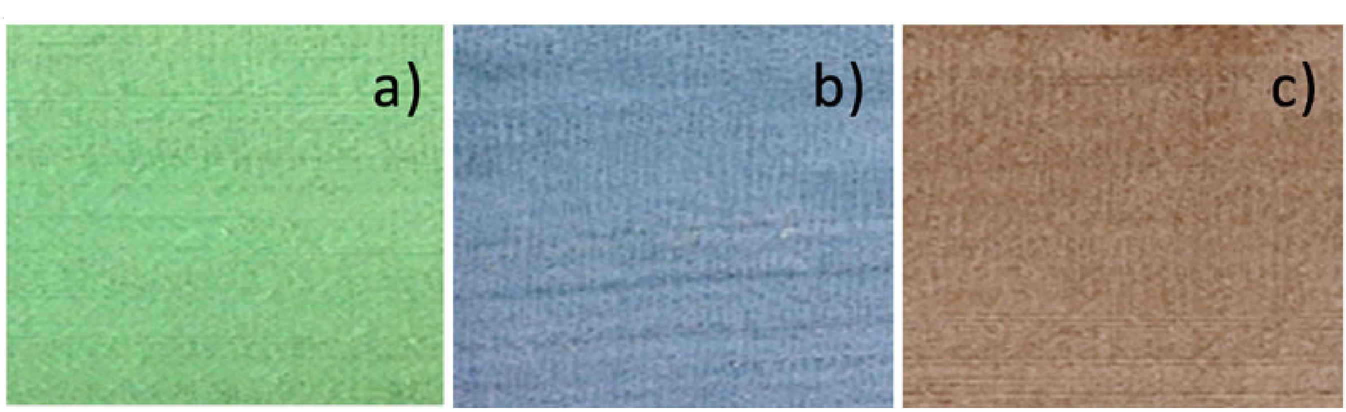

Fig. 6. Silk fabrics dyed with gemstone powder by the exhaustion method at a liquor ratio of 1:100, gemstone powder concentration 3 g/L, pH 3 (for malachite and jasper) or pH 5 (for lapis lazuli), and 90°C for 60 min

TABLE-1
COLOUR FASTNESS VALUES OF THE SILK FABRICS DYED WITH GEMSTONE POWDER BY THE EXHAUSTION METHOD[a]

Gemstone powder	Cationic pretreatment	Light fastness	Crock fastness		Wash fastness		
			Dry	Wet	Color change	Color staining	
						Cotton	Silk
Malachite	Untreated	5	2-3	2	1-2	4	4
	Treated	5	4	4	2-3	4	4
Lapis lazuli	Untreated	5	2	1-2	2	4-5	4
	Treated	5	3-4	4	3	4	4-5
Jasper	Untreated	5	2	1-2	1-2	4-5	4
	Treated	5	4	3-4	3	4-5	4

[a]Silks were dyed at a liquor ratio of 1:100, gemstone powder concentration 3 g/L, pH 3 (for malachite and jasper) or pH 5 (for lapis lazuli), and 90 °C for 60 min.

Colour fastness: Table-1 summarizes the colour fastness properties of silk fabrics dyed with gemstone powder using the exhaustion method. All samples showed very good colour fastness to light. Colour fastness to crocking of cationic treated silk fabrics was fair to good, which is better than that of untreated silk fabrics. The wash stain fastness of cationic treated silk fabric was comparable to the results found with untreated silk fabric because the gemstone powder has no affinity with the silk. Compared with untreated silk fabric, the wash fastness of cationic treated silk fabric was greatly increased. This may be attributed to the strong ionic bond between gemstone powder and silk fabric.

Conclusion

The dyeing properties such as dyeability and colour fastness of gemstone powder, *i.e.* malachite, lapis lazuli and jasper exhaustion used for dyeing cationic treated silk fabrics were investigated. For the range of conditions used in the experiments, the optimum dyeing effect was achieved at a liquor ratio of 1:100, temperature of 90 ºC, 60 min dyeing time and pH 3 (for malachite and jasper) and pH 5 (for lapis lazuli). Further improvement in colour yield was observed with increase in the gemstone powder concentration. The colour fastness data obtained in this study reveals that colour fastness can be improved significantly for cationic treated silk fabrics.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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