Development of All-In Dyeing and Lemon Oil Microcapsule Finishing Process for Silk

Phussadee Lim and Jantip Setthayanond

ABSTRACT--- In this research, the all-in acid dyeing/lemon oil microcapsule finishing process was studied with the aim to develop an efficient energy- and time-saving process for silk fabrics. Telon Red M-R and Supralan Red C-WN dyes were acid dyes used in this study. In the 2-step process, silk fabrics were dyed using pad-dry-bake process and then the finishing agent, lemon oil microcapsules, was treated onto the dyed silk fabrics using pad-dry-cure process in the presence of 20 g/l acrylic binder. In the developed all-in process, silk fabrics were treated in the mixed bath of acid dye and lemon oil microcapsules in the presence of acrylic binder. The results showed that silk fabric from the all-in process could attain comparable color strength and shade as compared with those from the 2-step process. In addition, the all-in process could also render a similar finishing degree to the 2-step process on silk, especially at pH 4, comfirming with the amount of extracted lemon oil from the treated fabrics. This study indicates the feasibility of the all-in acid dyeing/essential oil microcapsule finishing process for eco-friendly, energy- and time-saving silk processing.

Keywords - Silk, finishing, lemon oil microcapsule, acid dyeing, all-in process

INTRODUCTION

The chemical processes used in textile production are known to cause tremendous problems to environment and human well-being. Owing to a large consumption of chemicals and energy for textile processing, there is an attempt to improve the processing of textiles with the cleaner technology. Reducing chemical and energy usages as well as process time saving are parts of cleaner production criteria that can be applied to develop a cleaner textile processing.

Silk is a lustrous textile fiber used for high value textile production for both apparel and non-apparel applications. Silk is normally dyed with acid dyes and it can also be dyed with reactive dyes for those products with wet fastness concern. After dyeing, silk is normally subjected to finishing process to enhance a desirable properties that add more value to the final silk products. Perfumes and essential oil from herbal plants are widely studied and used for aroma finishing on silk and other textile fibers (Jiayong 1998: Li et al. 2008: Hipparagi et al., 2016: Srivastava and Srivastava, 2017). The essential oils for examples lemongrass, lemon, lavender etc. are typically in microencapsulated form (Bezerra, Carmona et al. 2016, Branta Lopes, Speranza et al. 2016). This type of silk finishing is typically done separately from dyeing process. It would be of a great interest if the dyeing and finishing process for silk can be conducted at the same time in order to save energy, time and also chemicals to fulfill cleaner silk production. One bath, all-in process has been reported on pretreatment and dyeing for cotton (Preša and Tavčer, 2008: Eren et al., 2009), however, up to date, there is no such or related research studied on silk yet.

PROBLEM STATEMENT

Dyeing and finishing processes are on the list of those processes heavily consume chemicals and energy as well as bringing about an environmental problems. Owing to these concerns, the production of textiles including silk, has been being upgraded to be more cleaner. In stead of using typical dyeing followed by finishing, it is possible to do both processing simultaneously on silk by all-in process. In this case, the dyeing and finishing efficiencies need investigation in order to achieve a satisfactory dyeing level and desirable degree of finished properties on silk.

THE AIM OF RESEARCH

In this paper, the all-in dyeing and lemon oil microcapsule finishing process for silk was developed in order to create an energy- and time-saving process with satisfactory dyeing and finishing results on silk fabric.

METHOD OF RESEARCH

Materials

Plain woven silk fabric with the weight of 61.76 g/m^2 was degummed before use. Microencapsulated lemon oil (with melamine-formaldehyde shell) and acrylic binder were purchased from Ratchada Chemicals, Co. Ltd., Thailand. The lemon oil microcapsules were characterized for their chemical compositon in this research by using Shimadzu gas chromatography - mass spectrophometer (GC-MS) and the results are showed in Table 1. Acid dyes viz., Telon Red M-R and Supralan Red C-WN were kindly supplied by DyStar, Thailand. Wetting agent used were Starpon 4488. All chemicals used were in analytical grade.

Table 1. GC-MS results of major chemical components found in the lemon oil microcapsules

Component	Extent (%)
D-limonene	45.59
1-Butanol, 3-methyl-, acetate	14.33
Butanoic acid, ethyl ester	7.12
7-Octen-2-ol, 2,6-dimethyl-	6.34
Hexanoic acid, ethyl ester	2.20

Revised Manuscript Received on February 14, 2019.

Phussadee Lim, Department of Textile Science, Faculty of Agro-Industry, Kasetsart University, Bangkok, Thailand.

Jantip Setthayanond, Department of Textile Science, Faculty of Agro-Industry, Kasetsart University, Bangkok, Thailand.

Methods

1. Acid dyeing

The degummed silk fabrics were dyed with 2 acid dyes viz. Telon Red M-R and Supralan Red C-WN at the concentration 1%owf using pad-dry-bake method. The fabrics were firstly immersed in the dye liquor for 15 minutes and then were squeezed through the pad mangle to attain 80% wet pick up. The padded fabrics were then dried at 120°C for 0.5 minutes and subsequently baked at 160°C for 2 minutes. The obtained dyed fabrics were rinsed with running water and after that, they were boiled in a 1 g/l wetting agent solution at 70°C 15 minutes, rinsed and air dried.

2. Finishing of lemon oil microcapsules

The silk fabrics dyed with Telon and Supralan dyes were taken to the finishing processing. In this process, lemon oil microcapsules were used as a finishing agent providing an aroma properties to the fabrics. The process was conducted by pad-dry-cure process by immersing the dyed fabrics in the solutions containing lemon oil microcapsules at various concentrations (10, 20 and 30 g/l) and 20 g/l acrylic binder for 15 minutes. After that, the fabrics were squeezed through the pad mangle at 80% wet pick up and then dried and subsequently cured at120°C for 0.5 minutes and 160°C for 2 minutes, respectively.

3. All-in dyeing/finishing process

In this process, the acid dyes and the lemon oil microcapsules were simultaneously applied onto silk fabrics by immersing the fabrics in a bath containing 1%owf dyes with lemon oil microcapsules at various concentration (10, 20 and 30 g/l) and 20 g/l acrylic binder for 15 minutes. The drying and curing steps and also the aftertreatment process were carried out as mentioned in the previous sention (1.). The effect of pH was also investigated by conducting the all-in bath at pHs 4 and 6.

4. Property measurement of the dyed and finished silk fabrics

The silk fabrics that passed through dyeing, 2-step dyeing/finishing and all-in dyeing/finishing processes were taken to examine their color strength (K/S values) and shade with a Macbeth ColorEye 7000 spectrophotometer. The surface morphology of silk was monitored with Jeol JSM-6480LV scanning electron microscope (SEM) at 10 kV.

The amount of lemon oil on the fabrics were examined by extracting 5cm x 10cm fabrics (0.30 g) with 25 ml ethanol in the stainless steel pots containing 25 steel balls (6-mm diameter size) and the extraction was conducted in a Starlet Daelim infrared dyeing machine at 35^{o}C for 2 hours. The absorbance values of the extracted liquor was measured with a Specord UV/vis spectrophotometer at the wavelength of 315 nm. The amount of lemon oil was calculated from the standard curve plotted between the concentrations and absorbance values of lemon oil solutions in ethanol.

RESULTS AND DISCUSSION

Table 2. shows the color strength (K/S) and colorimetric properties of the silk fabrics dyed with Telon Red M-R and Supralan Red C-WN dyes at 1%owf. Telon Red M-R is the milling acid dye while Supralan is a metal complex dye. At 1%owf applied depth, these two dyes exhibited nearly the same color strength level. Telon dye displayed bluish red with higher lightness as compared with Surpalan dye that showed yellowish red shade on silk.

When the dyed silk fabrics were finished with the lemon oil microcapsules at various concentrations, their colorimetric properties changed for the case of Telon dye (Table 3). For both 2-step and all-in processes, the K/S values of the dyed fabrics slightly increased as compared with the unfinished, dyed fabric. The 2-step process rendered a slightly better color yield on silk than the all-in process. For Supralan dye, the K/S values of the fabrics from two processes were not significantly different and they did not show any obvious difference from the unfinished, dyed fabric. Considering the 2-step finishing with the all-in dyeing/finishing processes, the results on color strength indicates that the all-in process could provide a comparable acid dyeing efficiency on silk to the typical 2- step process.

Table 2. Color properties of silk fabrics dyed with Telon Red M-R and Supralan Red C-WN dyes at 1%owf

Dye	Color properties			
	L*	a*	b*	K/S
Telon	84.19	15.28	-4.67	0.60±0.015
Supralan	76.45	25.04	1.02	0.77±0.040

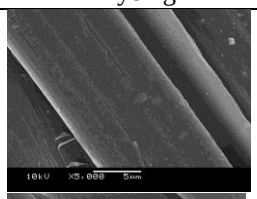
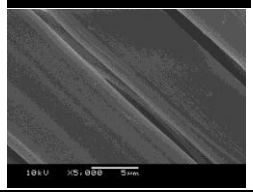
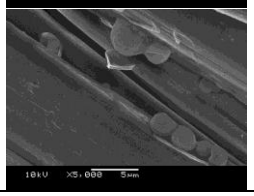
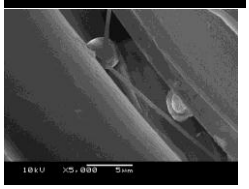
Table 3. Color strengths of the finished dyed silk fabrics

Dye	Lemon oil microcapsules (g/l)	K/S values		
		unfinished	2-step process	All-in process
Telon	0	0.60±0.015	-	-
	10		0.60±0.005	0.69±0.006
	20		0.83±0.017	0.70±0.001
	30		0.87±0.124	0.69±0.006
Supralan	0	0.77±0.040	-	-
	10		0.73±0.025	0.75±0.010
	20		0.72±0.015	0.77±0.012
	30		0.72±0.012	0.75±0.021

Table 4. illustrates the SEM images of the dyed silk fabrics in the absence and presence of lemon oil microcapsule finishing at 30 g/l. The unfinished silk

fibers showed a smooth surface. The existence of lemon oil microcapsules were observed being attached on the silk fiber surface that passed through the finishing process both the 2-step and the all-in processes. From the images, the presence of lemon oil microcapsules on the all-in processed silk fibers seemed slightly fewer than those of the 2-step process. However, the distribution of the lemon oil microcapsules may be varied from place to place on the fibers. Therefore, the quantitative measurement of lemon oil microcapsules on silk needs to be conducted.

Table 4. SEM images of the dyed silk fabrics with and without finishing with microencapsulated lemon oil at 30 g/l

Dye	Treatment		
	Dyeing	2-step,dyeing/finishing	All-in dyeing/finishing
Telon			
Supralan			

Apart from observing the fiber surface from SEM, the existence of lemon oil microcapsules on silk fabrics was also quantitatively confirmed by extraction technique. The finished silk fabrics were extracted with ethanol and the amount of extracted lemon oil was analyzed. Table 5. shows the amount of lemon oil extracted from silk fabrics finished with 30 g/l lemon oil microcapsules by all-in process as compared with the silk fabric exposed only to the finishing process. The pH of the finishing and all-in baths observed was in the vicinity of 6. The finished fabric contained 0.394 g lemon oil/g fabric, while those of the all-in process had marginally less amount of lemon oil (0.350 and 0.364 g/g fabric for Telon and Supralan dyes, respectively). The effect of pH on finishing efficiency was also conducted at pH 4. Only the acidic pH was chosen for this study in order not to cause an adverse effect to the dyeing ability of acid dyes on silk if the alkaline pH was employed. In addition, alkaline pH is not recommended for protein fiber processing as it can causes fiber damage (Gulrajani, 1992). When the pH of the all-in bath was reduced to 4, the amount of lemon oil extracted from the fabrics increased from those at pH 6 for both types of dyes used. Telon-dyed fabrics had 0.388 g lemon oil/g fabric and those of Supralan dye contained 0.408 g lemon oil/g fabric. This result points out that the bath pH influences on the finishing efficiency of the all-in process. By reducing pH to 4, the lemon oil microcapsules could well be treated onto the silk fabrics to a similar level obtained on the finished ones.

In order to assure that the pH 4 all-in bath condition would not impair the dyeing ability of the acid dyes on silk, the color strength of the fabrics exposed to all-in process at pH 4 was examined and the results was shown in Figure 1. The color strength of the dyed fabrics were compared between those from 2-step process and those from all-in process at pHs 4 and 6. It was found that the optimum condition providing highest color strength on silk for both Telon and Supralan dyes was the all-in process at pH 4. This may be explained by the more protonated amino groups on silk fiber. These greater positively-charged amino groups on silk attract more acid dyes to uptake into the fiber, hence the color strength was increased. Such condition also gave the best finishing efficiency of lemon oil microcapsules on silk among the three condition considered.

Therefore, from this research, it is suggested that the all-in dyeing and finishing process developed is practically feasible for silk processing and the condition recommended is that at pH 4.

Table 5. Amount of extracted lemon oil from silk fabrics finished with lemon oil microcapsules at 30 g/l

Bath pH	Process used	Amount of lemon oil (g/g fabric)
	Finishing process	0.394 ± 0.002
6	All-in process	
	- Telon dye	0.350 ± 0.042
	- Supralan dye	0.364 ± 0.045
4	All-in process	
	- Telon dye	0.388 ± 0.075
	- Supralan dye	0.408 ± 0.032

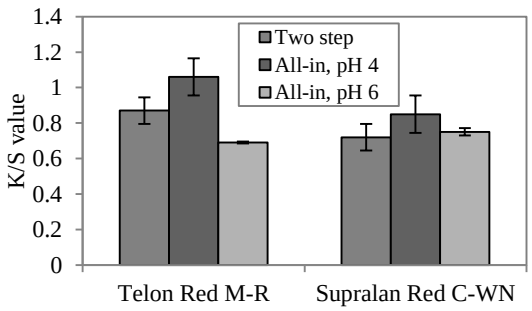


Figure 1. K/S values of the dyed sik fabrics underwent 2-step and all-in dyeing/finishing processes

CONCLUSION

The all-in process of acid dyeing and lemon oil microcapsule finishing was developed for silk fabric. By comparing with the 2-step process (acid dyeing followed by lemon oil microcapsule finishing), the all-in process could provide a comparable color strength of Telon Red M-R and Supralan C-WN dyes obtained on silk fabrics and even better color strength level could attain when the all-in bath pH was adjusted from pH 6 to pH 4. The all-in process conducted at pH 4 also rendered a satisfactory finishing level of lemon oil microcapsules on silk, comfirming with SEM and extraction results. Therefore, it can be said that the all-in process acid dyeing and lemon oil finishing is practically viable for cleaner silk processing.

ACKNOWLEDGEMENTS

The authors are indebted to Kasetsart University Research and Development Institute, Kasetsart University for financially supporting this research.

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