ENHANCEMENT OF DYE-ABILITY OF VISCOSE FABRIC VIA MODIFICATION WITH FIBROIN REGENERATED FROM WASTE SILK COCOONS

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Abstract: *This work demonstrated the possibility of enhancing the dye-ability of viscose fabric via surface modification with silk fibroin. Herein, silk fibroin from waste Bombyx mori cocoons was degummed and then dissolved in a lithium bromide/ethanol/water solution. The silk fibroin solution was purified using the QuixStand Benchtop system equipped with the microfiltration and ultrafiltration of hollow-fiber cartridges to gather the fibroin segments with molecular weight over 10 kDa. The obtained silk fibroin was coated onto a viscose woven fabric via the padding method. The fibroin treated and untreated viscose fabrics were stained with C.I acid blue 203 (0.5% on the weight of fabric (wof) and dyed with C.I reactive yellow 176 (0.1, 0.5 and 1.0 %wof). The presence of silk fibroin on the viscose fabric was confirmed by scanning electron microscopy (SEM), Fourier transform infrared (FT-IR) analysis and color measurement. The dyed treated viscose fabrics revealed a higher color strength (K/S) than that of the dyed untreated ones at the same initial dye concentration. The color fastness to washing of the fibroin treated and untreated viscose fabrics after dyeing with the reactive dye were good to very good. The physical analysis results indicated a decrease in the air permeability and the horizontal wicking values of the silk fibroin treated fabrics compared to the untreated fabrics.*

Keywords: *Bombyx mori cocoons, regenerated fibroin, LiBr, viscose, dyeing.*

1 INTRODUCTION

Viscose is the most of the world's man-made cellulose fibers because it can be engineered chemically and structurally in many ways [1]. Viscose fibers have inherent properties of cellulosic fibers including breathability, high moisture regime, softness, drapability and biodegradation [1]. Due to these desirable properties, the demand of viscose fibers for the textile industry is ever-growing in the global market. However, the disadvantages of viscose fabrics include easy wrinkle, low dimensional stability and poor protection against UV radiation [1]. To overcome and enhance these properties of viscose fabrics, some special finishes have been applied on such materials to achieve specific end uses such as wrinkle free effect, UV-protection and antibacterial activity [2, 3]. However, conventional finishes had been used for textiles which generally employed synthetic polymers for certain outcomes. Toward the trend of sustainable development, the finishing of textile materials using natural polymers has gained rapid progress during the last few decades [4-6]. One of the natural polymers recently attracted the attention of researchers all over the world is the regenerated silk fibroin [7-9].

Fibroin is the main component of silk fiber containing up to 90% of the amino acids (glycine, alanine and serine) leading to formation of microcrystalline β-sheet in the fibers [10]. Silk fibroin could be dissolved in various highly concentrated salt solutions including LiBr, NaSCN, N-methyl morpholine N-oxide and $CaCl_2$/water/ethanol (CWE) [7, 8, 10-13]. Among them, LiBr/ethanol and N-methyl morpholine N-oxide are the best solutions to dissolve silk fibroin. Depending on application purpose, regenerated silk fibroin could be shaped into many different structures: fibers, membrane, sponge scaffolds, hydrogels, microspheres, coating on the materials [7, 9, 10, 14].

Finishing textile materials using fibroin solution has been reported in numerous works [7-9, 15-20]. Surface modification of textile material through deposition of regenerated silk fibroin indicated that the potential of such fibroin films to be developed further as antistatic finishing for synthetic textiles [7]. The use of fibroin as additive with citric acid for the crease resistant finishing of cotton fabrics not only improved the wrinkle property but also avoided the fabric yellowing problem [8, 17, 19]. The fibroin formed a film onto the wool surface was responsible for the anti-felting, thus grafting such a natural biopolymer onto wool would enhance this feature [15].

Finishing textile by silk fibroin would bring new perspectives in the application of regenerated silk

fibroin on textile materials. Silk fibroin solution applied to the cellulose fabric surface can significantly improve the wrinkle recovery angle as well as other properties while slightly effect to the color of the treated fabrics [9, 16]. This process could make such the material of high interest for wound dressing and clothing required in therapy of skin diseases. To expand the applications of the fibroin treated viscose fabric in the apparel industry, it needs to diversify in color. In textile processes, dyeing is one of the most important procedures to enhance aesthetics value of the textile materials.

In this paper, the processes of degumming *Bombyx mori* silkworm cocoons, dissolving silk fibroin in lithium bromide salt solution and finishing woven viscose fabric were studied. To evaluate the impact of the fibroin on the dyeing behavior of the fibroin treated viscose fabric, the untreated and treated samples were dyed using C.I. acid blue 203 and C.I. reactive yellow 176. The efficiency of the process implementation was determined by measuring the color coordinates and analyzing the physical properties of dyed samples. The results provided the understanding of deposition and dyeing properties of the viscose fabric treated silk fibroin. This is a new approach to finishing textiles in a sustainable development trend.

2 MATERIALS AND METHODS

2.1 Materials

Waste *Bombyx mori* silkworm cocoons were collected from Vong Nguyet village, Bac Ninh province, Vietnam. Plain woven viscose fabric (staple viscose, Ne 30/1) was scoured and supplied by Nam Dinh Textile Garment Co. Ltd., Vietnam. C.I. acid blue 203 (Telon Blue M-BLW) and C.I. reactive yellow 176 (Remazol Yellow 3RS) dyestuffs were obtained by DyStar Pte. Ltd., Singapore. Other chemicals (Na_2CO_3, C_2H_5OH, CH_3COOH, LiBr, $Al_2(SO_4)_3.18H_2O$) were purchased from Aladdin Shanghai Biochemical Technology Co. Ltd, China. Double distilled water from an EYELA Still Ace SA-2100E was used as the solvent in all experiments.

2.2 Experimental methods

Degumming

Bombyx mori silkworm cocoons were degummed in a solution of 5 g/L Na_2CO_3 at 98°C for 30 min at a liquor ratio of 1:20 (mass in gram per volume in mL) [7, 9]. The silk fibroin was rinsed five times by warm and cold distilled water, then dried at 40°C and stored at 65% relative humidity and 20°C.

Dissolution of silk fibroin

The dissolution of silk fibroin procedure has been reported in our previous work [16]. In a typical experiment, 2.8 g degummed silk fibroin was dissolved in a triangular flask containing 10 mL solution of Lithium bromide/Ethanol/Water (LiEtW) with a mass ratio of 45:44:11, at 80°C for 60 min. The obtained fibroin solution was diluted 15 times with double distilled water to reduce the viscosity. The fibroin solution was then removed excess LiBr and ethanol through microfiltration and ultrafiltration systems with hollow-fiber cartridges in the QuixStand Benchtop system (Watson-Marlow 323 peristaltic pump, UK). In the first stage of the filtration, 0.2 µm hollow-fiber cartridge was used to remove impurities and high molecular weight fibroin segments. Next stage, the fibroin solution was subsequently filtered through a 10,000 NMWC (nominal molecular weight cutoff) hollow fiber ultrafiltration cartridge to get the fibroin segments with molecular weight over 10 kDa retaining inside the filter tube. The solution passed through the ultrafiltration system contained low molecular weight fibroin segments, excess LiBr, ethanol and water. The fibroin content in the obtained solution was measured using an infrared moisture analyzer (MA35, Sartorius). The scheme of degumming, dissolving and filter silk fibroin was illustrated in Figure 1.

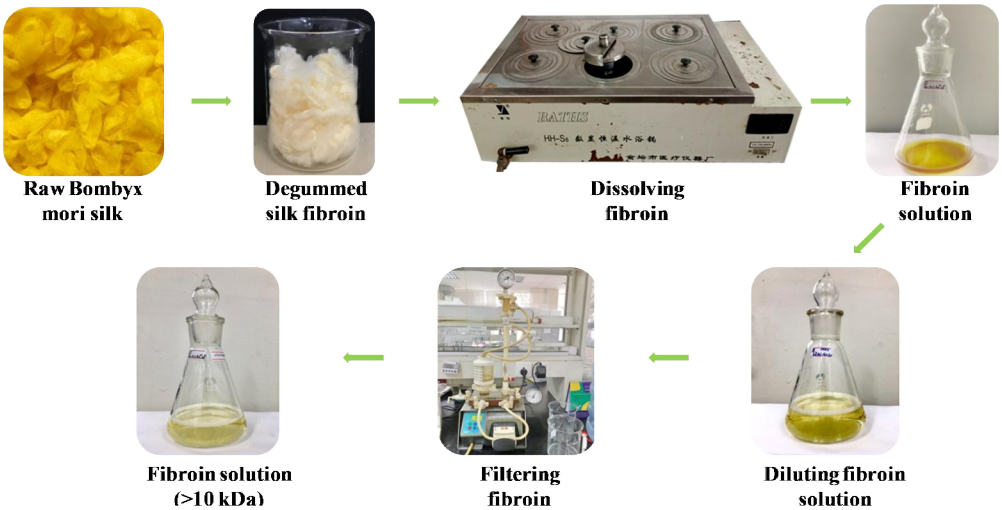


Figure 1 Scheme of degumming, dissolving and filtering silk fibroin

Treatment of viscose fabric with silk fibroin solution

Viscose fabric samples with size of 35×35 cm were impregnated in 100 mL fibroin solutions (>10 kDa) with different concentrations of 1.0, 2.5 and 5.0%. After that, the fabric samples were padded using Atlas D394A laboratory padder, and the padding pressure was adjusted at 3 kg/cm^2 to allow a pickup of 80%. The padded fabrics were dried for 2 min at 110±3°C using SDL mini-drier 398 laboratory thermo-fixation. The dipping–padding–drying processes of viscose fabrics were repeated 2 times. In the following step, the dried samples were soaked in a 10 g/L aluminum sulfate solution and then padded at 80% wet pickup to regenerate and fix silk fibroin onto viscose fabrics. The treated fabrics were dried at 60°C in an electric heated oven. The process of viscose fabric treated with silk fibroin solution was presented in Figure 2.

Dyeing of viscose fabric with the acid dye

The untreated and treated viscose fabrics were dyed in an aqueous solution with 0.5 %wof acid dye C.I. Acid Blue 203 under acidic conditions (pH = 4 with acetic acid) at a fabric to liquor ratio of 1:20, for 45 min at 80°C in an infrared dyeing machine (Figure 3a). The dyed samples were thoroughly rinsed with warm and cold water then air-dried.

Dyeing of viscose fabric with reactive dye

The untreated and treated viscose fabrics were dyed with a reactive dye C.I. Reactive Yellow 176. To investigate the effect of dye concentration, the fabrics were dyed in aqueous solutions containing different dye concentration (0.1, 0.5 and 1.0 %wof), Na_2SO_4 20 g/L, Na_2CO_3 15 g/L, at a liquor ratio of 1:20. The dyeing process was started by raising the temperature to 80°C and dyed for 45 min in an infrared dyeing machine (Ti-Color dyeing machine, ICL, Prato, Italy), (Figure 3b). After dyeing, the dyed samples were rinsed with warm and cold water then air-dried.

Figure 2 Scheme of viscose fabric treated with silk fibroin solution

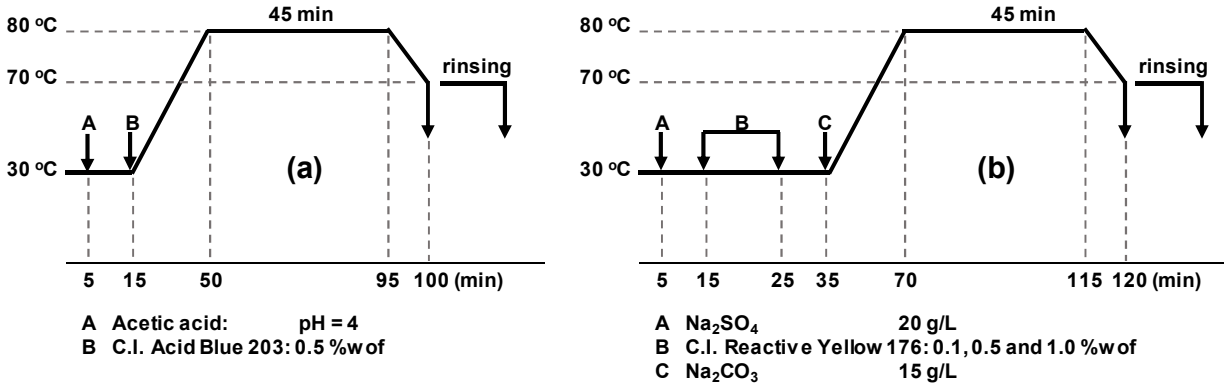


Figure 3 Exhaust dyeing line diagrams of the untreated and treated viscose fabrics with (a) acid dye and (b) reactive dye

2.3 Analytical methods

Morphological analysis: The morphologies of the untreated and treated viscose fabrics with silk fibroin were characterized via a scanning electron microscope (SEM) SM-6510LV Jeol, Japan.

FT-IR analysis: Fourier transform infrared spectrophotometry (Thermo Nicolet 6700 FT-IR spectrometer, USA) was used to confirm the regeneration of silk fibroin onto viscose fabric.

Color analysis: To evaluate dyeing performance, the color strength (*K/S*) and CIELAB of the dyed samples were determined using a reflectance spectrophotometer (X-rite, Ci4200) with D65 illumination, 10° observer. The *K/S* was calculated by the Kubelka–Munk equation (1).

$$K/S = (1-R)^2 / 2R \quad (1)$$

where *K* is the absorption coefficient, *S* is the scattering coefficient and *R* is the fractional reflectance.

The color difference was expressed as ΔE^* and was calculated by the following equation (2):

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{0.5} \quad (2)$$

where ΔE^* is the CIELAB color difference between batch and standard. ΔL^* denotes the difference between lightness (where $L^* = 100$) and darkness (where $L^* = 0$), Δa^* is the difference between green ($-a^*$) and red ($+a^*$) and Δb^* is the difference between yellow ($+b^*$) and blue ($-b^*$).

Color fastness test: Color fastness to washing of the dyed samples was conducted following standard test method ISO 105-C10:2006 using a color fastness to washing tester (Changzhou, China).

Physical property measurements: The horizontal wicking of the fabrics was evaluated according to AATCC 198-2013. The air permeability of the samples was performed according to ASTM D737-04 using a MOZIA Air Permeability Tester (USA).

3 RESULTS AND DISCUSSION

3.1 Evidence of silk fibroin adhered on viscose fabric

To demonstrate the deposition of silk fibroin on viscose fabric, the samples were conducted by SEM analysis and the results were shown in Figure 4. At the magnification of over ×1000, SEM images of the 2.5 wt% fibroin treated viscose fabric revealed clearly the fibroin films covered viscose fibers, while the fiber surface of untreated viscose fabric was smooth. The SEM images confirmed the existence of the silk fibroin adhered onto viscose fibers after padding and aluminum salt treating processes.

In order to determine the change of functional groups of silk fibroin during its dissolution and regeneration, the FT-IR measurements of degummed silk fibroin (DeSilk), regenerated silk fibroin in $Al_2(SO_4)_3$ aqueous solution (ReFib), viscose fabric (Vis) and fibroin treated viscose fabric (VisFib) samples were carried out, and the spectra were given in Figure 5. The characteristic peaks of the DeSilk at 3280, 1621, 1513, 1228 and 1065 cm^{-1} were assigned to N-H (amino), C=O (amide I), C-N and N-H (amide II), C-N and N-H (amide III) and O-C-N groups, respectively [7-16]. Compare to the degummed silk fibroin, the regenerated silk fibroin in $Al_2(SO_4)_3$ aqueous solution revealed a slight shift in the characteristic peaks. The peaks of the functional groups in the ReFib shifted toward higher wavenumbers at 1622, 1515, 1231 and 1068 cm^{-1}, respectively, while the peak of N-H in amino group shifted to lower wavenumber at 3273 cm^{-1}. Moreover, the intensity of the absorption peaks in the ReFib spectrum was significantly decreased. These imply that the interaction of Li^+ and Al^{3+} ions with amino and amide groups of fibroin molecules to form the fibroin-Li^+ and fibroin-Al^{3+} complexes took place.

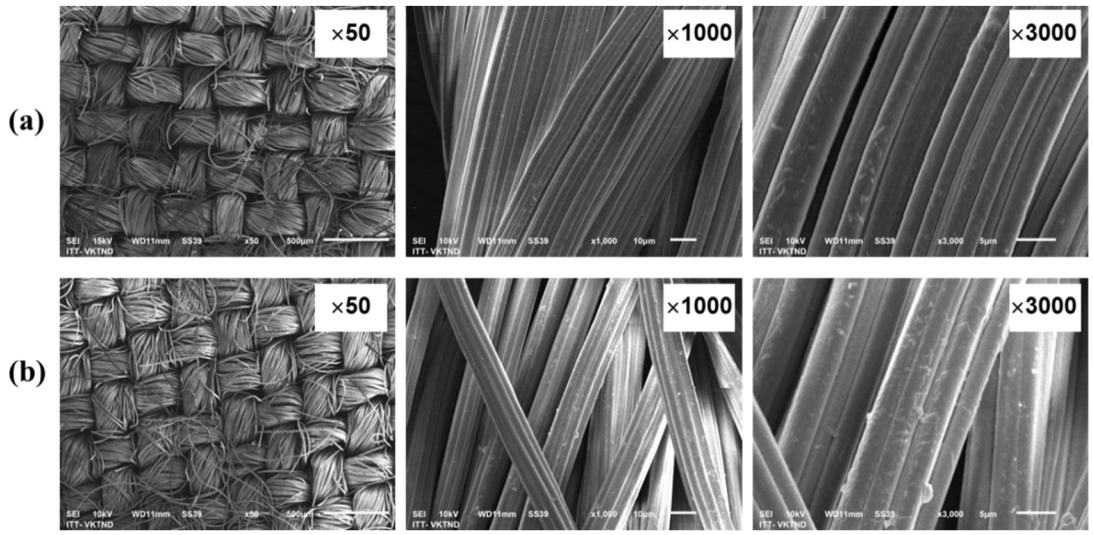


Figure 4 The SEM images of (a) untreated viscose fabric and (b) treated viscose fabric with 2.5 wt% fibroin at magnification of ×50, ×1000 and ×3000

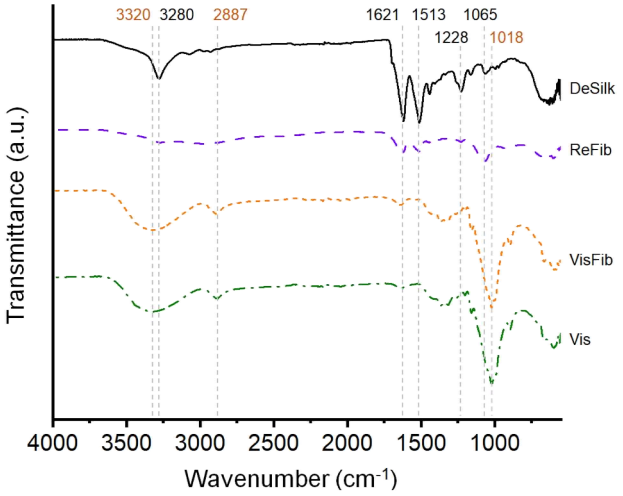


Figure 5 FT-IR spectra of the samples: (DeSilk) degummed silk fibroin; (ReFib) regenerated silk fibroin in $Al_2(SO_4)_3$ aqueous solution; (Vis) viscose fabric and (VisFib) fibroin treated viscose fabric

The characteristic peaks corresponding to viscose appeared at 3332 cm^{-1} (O-H stretching), 2887 cm^{-1} (C-H stretching), 1639 cm^{-1} (C=O bending), 1364 cm^{-1} (C-H bending) and 1018 cm^{-1} (C-O-H stretching) [1-3, 7-9]. These peaks were also seen in the VisFib spectrum, suggesting the chemical structure of the VisFib was mostly unchanged. Despite the spectral similarity, the differences in the intensity between the higher Vis peaks and the lower VisFib peaks at 3332, 1639 and 1018 cm^{-1} were noticeable, indicating that the interactions might be occurred via formation of hydrogen bonding between fibroin's amide groups and viscose's hydroxyl groups, and/or via complexation of Al^{3+} ions with appropriate functional groups of fibroin and viscose. On the basis of the FT-IR observations, the mechanism of fibroin regeneration and deposition onto viscose fabric was elucidated in light of Figure 6.

3.2 *Dyeing behavior of fibroin treated viscose fabric*

Dyeing with the acid dye

In order to evaluate the effect of the silk fibroin deposited onto viscose fabric, the samples were dyed by C.I. Acid Blue 203. The color difference (ΔE^*) of the viscose fabrics before and after dyeing were evaluated using X-rite, Ci4200 spectrophotometer. The values of *L*, a*, b** and *K/S* of the neat viscose fabric (Vis), the fibroin treated fabric (VisFib), the dyed untreated fabric (VisA) and the dyed treated fabric (VisFibA) were presented in Table 1.

The obtained values show that it is difficult to distinguish the color difference of the untreated and treated fabric samples with silk fibroin by naked eyes (ΔE^* = 0.48).

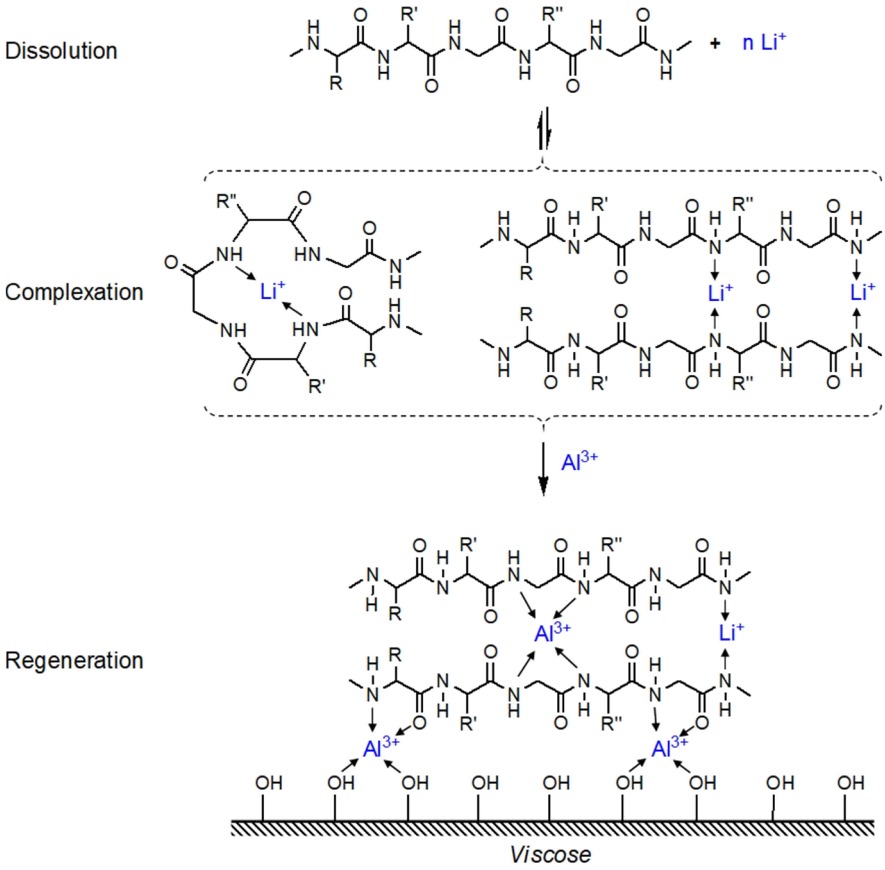
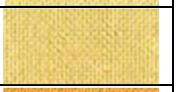


Figure 6 The proposal mechanism of fibroin regeneration and deposition onto viscose fabric

Table 1 *L*, a*, b** and *K/S* values of the fabric samples dyed with acid dye

Sample	L*	a*	b*	C*	H*	ΔE*	K/S	Scan sample
Vis	93.69	-0.58	4.78	4.82	96.93	0	0.06	
VisFib	93.00	-0.84	5.03	5.11	97.15	0.48	0.09	
VisA	85.82	-4.63	-7.21	8.56	109.02	16.54	0.15	
VisFibA	69.76	-0.32	-22.55	22.55	117.73	34.26	0.72	

Table 2 *L*, a*, b** and *K/S* values of the fabric samples dyed with reactive dye

Sample	L*	a*	b*	C*	H*	ΔE*	K/S	Scan sample
VisR1	87.81	2.91	37.79	37.91	94.26	35.91	0.75	
VisFibR1	87.09	4.30	39.10	39.34	93.76	37.52	0.84	
VisR2	77.77	16.36	58.35	60.61	90.86	61.05	3.52	
VisFibR2	79.30	16.83	62.18	64.42	90.18	65.07	3.69	
VisR3	75.58	22.47	68.86	72.44	88.84	73.95	6.33	
VisFibR3	75.55	22.54	69.25	72.83	88.83	74.37	6.44	

However, the *K/S* and *ΔE** values of the VisFibA fabric were 4.8 and 2.1 times higher than those of the VisA fabric, respectively. Thus, it could be concluded that the silk fibroin has been regenerated onto the viscose fabric, and it strongly effected on the dye uptake due to the ionic interaction between acid dye molecules and protonated amines of fibroin. The neat viscose fabric is lack of sufficient functional groups for binding to the acid dye molecules, thus the acid dyes was only stained on the surface of fabric VisA resulting in its very poor washing fastness.

Dyeing with the reactive dye

To investigate the dye-ability of the fibroin treated viscose fabrics, the untreated and treated samples were dyed with a reactive dye C.I. Reactive Yellow 176 at various concentrations (0.1, 0.5 and 1.0 %wof). The obtained values (Table 2) show that increasing reactive dye concentrations increased the color strength (*K/S* values) of both the dyed untreated fabric samples (VisR1, VisR2, VisR3) and the dyed treated fabric samples (VisFibR1, VisFibR2, VisFibR3). Furthermore, the color strength of the dyed treated samples was higher than that of the dyed untreated samples.

This observation was similar to the color difference (*ΔE**) values of the samples. It could be explained by the formation of covalent bonding of amine groups in fibroin adhered on viscose fabric with reactive dye molecules which supplemented to the linkage of hydroxyl groups of cellulose in viscose fabric with the dye molecules. Therefore, the fibroin treated viscose fabric could improve the reactive dye uptake to compare with the untreated fabric.

3.3 Evaluation of color fastness to washing

The washing fastness property of the various viscose fabrics dyed with the reactive dye was evaluated and given in Table 3. The washing fastness of the fabrics was evaluated in terms of the degree of color change and color staining. As shown in Table 3, the color fastness to washing of both the untreated and treated fabric samples were good to very good (4 to 4-5). Additionally, the color fastness to washing of the dyed treated viscose fabrics was the same as it of the dyed untreated samples at a given reactive dye concentration.

Table 3 The washing fastness of the samples

Sample	Color fastness to washing	
	Changing	Staining
VisR1	4-5	4-5
VisFibR1	4-5	4-5
VisR2	4	4
VisFibR2	4	4
VisR3	4	4
VisFibR3	4	4

3.4 Physical properties of fibroin treated viscose fabric dyed with a reactive dye

We have found that the formation of silk fibroin layer on viscose fiber was responsible to the change of physical properties of the treated fabric including air permeability, wrinkle recovery angle and breaking strength [16]. In this research, the impact of reactive dye concentration (0.1, 0.5 and 1.0 %wof) in the dyebath on the air permeability and horizontal wicking of the dyed viscose fabrics untreated and treated with silk fibroin. It is clearly observed from Figure 7 that the air permeability of the neat viscose fabric decreased slightly and gradually after dyeing by the reactive dye with an increase in initial dye concentration. However, after dyeing, the air permeability of the fibroin treated viscose fabric decreased obviously with increasing the initial dye concentration.

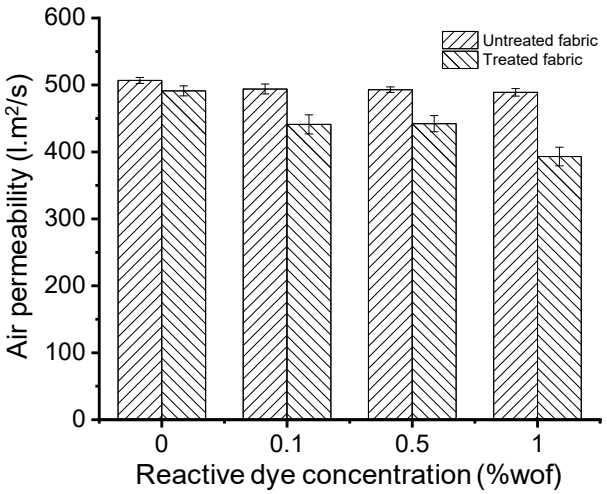


Figure 7 The air permeability of the fibroin treated and untreated viscose fabric dyed with C.I. reactive yellow 176 at 0.1, 0.5 and 1 %wof

The decrease in the air permeability of the dyed fabric with the increase in the initial dye concentration could be due to adsorption and permanent interaction of dye molecules onto the fabric. For the fibroin treated fabric, the dye uptake was not only by the interaction of dye molecules with hydroxyl groups of celluloses but also by the covalent bonding between dye molecules and amines of silk fibroin. The higher dye uptake of the dyed fabric could lead to the lower its air permeability. These findings were correlated with the observations of SEM analysis and color measurement.

To determine the influence of the silk fibroin treatment and the initial reactive dye concentration on the water-transporting property of viscose fabric, a horizontal wicking test was conducted as shown in Figure 8.

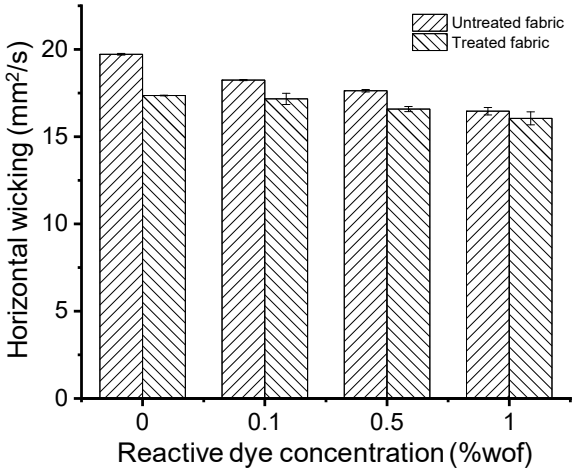


Figure 8 The horizontal wicking of the fibroin treated and untreated viscose fabric dyed with C.I. reactive yellow 176 at 0.1, 0.5 and 1.0 %wof

The results show that the neat viscose fabric revealed the highest horizontal wicking among other samples, while the dyed fabrics showed gradually a decrease in the wicking ability together with increasing the dye concentration. The horizontal wicking of the fibroin treated fabric declined about 12% in comparison with the untreated fabric, indicating the possibility of the silk fibroin deposited on the surface of fibers to hinder capillary flow of water through inter-fiber or inter-yarn spaces in the fabric. Comparing to the dyed untreated fabrics, the horizontal wicking of the dyed treated fabrics with C.I. reactive yellow 176 at different concentrations (0.1, 0.5 and 1.0 %wof) decreased 5.91, 5.95 and 2.48%, respectively. The decrease in the wicking ability of fabric after dyeing could be attributed to the adsorption of dye molecules into the micro capillaries' surface and the formation of covalent bonding with hydroxyl groups of cellulose, subsequently resulting in the decrease of water absorbency and wicking ability of the dyed fabrics.

4 CONCLUSION

In this study, the dissolution of silk fibroin obtained from the waste *Bombyx mori* cocoons in LiBr/ethanol/water solution (45:44:11) and the regeneration of purified fibroin onto viscose fabric have been investigated. The QuixStand Benchtop system equipped with the microfiltration and ultrafiltration of hollow-fiber cartridges was used

to remove excess LiBr and ethanol and to obtain the fibroin segments with molecular weight over 10 kDa. The deposition of fibroin on the treated fabric was evidenced via SEM analysis and color measurement using an acid dye labeled fibroin on the fabric. The dye-ability of the fibroin treated viscose fabric with a reactive dye was enhanced in comparison with the untreated fabric through the increasing color strength. The color fastness to washing for both the untreated and treated fabrics colored with the reactive dye were good to very good. The air permeability and horizontal wicking of the fabrics were decreased after finishing fabrics with silk fibroin and/or dyeing them with the reactive dye. In view of the attractive finishing technique, dye-ability improvement and sustainable development, the regenerated silk fibroin could be utilized as a potential finishing agent for the textile industry.

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