

# Application of Silk Sericin to Polyester Fabric

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**ABSTRACT:** Sericin powder was prepared from a boiled solution of silk cocoons through salting-out with ammonium sulfate. The obtained sericin powder was characterized with Fourier transform infrared (FTIR), X-ray, ultraviolet (UV) absorption, and nitrogen content measurements. The aqueous sericin solution was applied onto the polyester fabric (pretreated with NaOH) along with glutaraldehyde as a crosslinking agent with a pad-dry-cure method. The processing conditions of the crosslinking agent were optimized. The presence of sericin was confirmed by the estimation of the nitrogen content and amino groups on the treated fabric. The dyeability of the treated fabric with acid and reactive dyes was studied. The performance properties, such as the moisture content, UV absorption, antistatic, crease recovery, and bending length, of the treated fabric

were tested. The FTIR, X-ray, UV absorption, and nitrogen content results confirmed the purity of the sericin powder obtained with the salting-out method. The amino acid content, nitrogen content, and Kubelka Monk function ( $K/S$ ) values of the sericin-treated fabric increased with an increase in the concentration of sericin in the padding liquor. The  $K/S$  value of the dyed samples was found to be linearly correlated with the number of amino groups present on the samples. There was a noticeable improvement in the moisture content and antistatic and UV absorption properties of the sericin-treated fabrics. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 109: 314–321, 2008

**Key words:** biopolymers; crosslinking; peptides; polyesters; proteins

## INTRODUCTION

Sericin has attracted special attention among researchers because of its diverse physiological properties, such as antioxidant, ultraviolet (UV) protection, moisture absorption, and antibacterial activity properties. Sericin is a macromolecular globular protein found in silkworms that performs the function of gluing the silk fibers together. It contains many hydrophilic amino acids, which give it high hydrophilicity and sensitivity to chemical modification. Sericin can be crosslinked, copolymerized, and blended with other macromolecular materials, especially synthetic polymers, to produce materials with improved properties.<sup>1</sup>

At present, sericin is mostly discarded in silk-processing wastewater. If sericin can be used as a finishing agent for synthetic fabrics, it will represent a natural value-added finishing agent developed from waste. In addition to that, the recovery of sericin reduces the pollution load in wastewater. The recovery of sericin from wastewater by various processes has been reported in the literature.<sup>2,3</sup> Recovered sericin proteins have found effective applications in the areas of cosmetics, food, medical, and textiles.<sup>4</sup>

Polyester fabrics have taken a major position in textiles because of their excellent physical properties, such as the tensile strength, crease recovery angle, and biological resistance. However, they show poor comfort properties because of their low moisture regain, which causes a number of problems, such as a tendency to accumulate static charge and soiling.

The application of sericin to polyester fabric improves its moisture absorption and antistatic properties.<sup>5–7</sup> Attempts have been made by many researchers to induce favorable properties in polyester fabrics with sericin. Jin et al.<sup>5</sup> applied sericin to ethylenediamine-pretreated polyester fabric along with chloromethyloxirane or cynuric chloride as a crosslinking agent. The finished fabric showed improved moisture absorption ability. However, the harshness of the fabric was also increased. Sericin crosslinked with ethylene glycol diglycidyl ether on polyester fabric grafted with *N*-vinylformamide and hydrolyzed with sulfuric acid was studied by Lee et al.<sup>6</sup> The finished fabric showed improved moisture absorption and antistatic properties without changes in the original smooth touch of the fabric. Pan et al.<sup>7</sup> developed a method to improve the wearing characteristics of polyester fabrics by a sericin and glutaraldehyde treatment.

Polyester fibers and fabrics treated with sericin and dyed with natural dyes have been patented by several researchers.<sup>8–10</sup> The treated fibers and fabrics have exhibited silklike colors.

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The aim of this study was to develop various application areas in the field of textiles for sericin, which has potential physiological properties. As mentioned previously, sericin has been used for textile finishing in numerous investigations, but the UV-protective properties have not yet been elucidated. Furthermore, a study on polyester fabric pretreated with sericin and dyed with reactive and acid dyes has not been reported. In this study, sericin was extracted from mulberry cocoons. The extracted sericin was characterized with FTIR, nitrogen content measurements, X-ray diffractograms, and UV absorption spectra. Sericin was crosslinked to the polyester fabric (pretreated with sodium hydroxide) with glutaraldehyde. The presence of sericin on the polyester fabric was confirmed with nitrogen and amino group estimation. The acid and reactive dyeability of the treated fabrics were studied. The performance properties of the sericin-treated fabric, such as the moisture content, UV protection, crease recovery, bending length, and antistatic ability, were also studied.

## EXPERIMENTAL

### Materials

Mulberry silk cocoons were procured from Tamilnadu (Karur, India). Polyester fabric with a plain weave of 80 gsm was used throughout the study. Laboratory-grade ammonium sulfate, magnesium chloride, acetic acid, glutaraldehyde, potassium sulfate, copper sulfate, sulfuric acid, bromocresol green solution, methyl red indicator, sodium propionate, ninhydrin, methyl cellosolve, propionic acid, glycine, and isopropanol were used. Navimill Yellow 56N (acid dye) and Remazol Black B (reactive dye), procured from Dystar, Ltd. (Ahmedabad, India), were used for dyeing.

### Preparation of sericin powder

The cleaned cocoon shells were cut into small pieces for sericin extraction. The aqueous extraction of sericin was carried out through the boiling of the cocoons in distilled water for 90 min with a 1:30 material-to-liquor ratio. Ammonium sulfate (40 g) was added to every 100 mL of the extracted solution to salt out the sericin.<sup>11</sup> The precipitated particles were filtered out and dried to obtain sericin powder.

### Characterization of sericin powder

#### Fourier transform infrared (FTIR)

The FTIR spectra of the sericin samples were recorded ( $400\text{--}4500\text{ cm}^{-1}$ ) on a PerkinElmer Spectrum-BX FTIR system (Waltham, MA) with the KBr pellet technique. The KBr pellets were prepared by

the grinding of 1 part of the sample with 9 parts of spectral-grade KBr and pressing in an evacuated die under suitable pressure to get pellets.

#### UV absorption

Sericin was dissolved in distilled water to get a very dilute solution. The UV absorption spectrum of the solution was recorded with a Pharmacia LKB ultraviolet-visible spectrophotometer (Uppsala, Sweden) with Bio-Chrom 4060 software.

#### X-ray analysis

The X-ray diffraction pattern of sericin powder was recorded in the  $2\theta$  range of  $10\text{--}35^\circ$  on an X-Pert Pro Pananalytical X-ray diffractometer (Almelo, The Netherlands). Cu  $K\alpha$  radiation (wavelength =  $1.54\text{ \AA}$ ) was used for the X-ray diffraction study.

#### Nitrogen content

The nitrogen content of the sericin powder and treated fabrics was estimated by Kjeldahl method.<sup>12</sup>

### Application of sericin to polyester

The polyester fabric was pretreated with 15% NaOH (owf), with the material-to-liquor ratio kept at 1 : 40, at  $60^\circ\text{C}$  for 30 min to get a weight loss of 5%. This weight loss was expected to give a sufficient number of end groups so that further treatment could be carried out.<sup>13</sup> The pretreated fabrics were padded (80% expression) with the sericin solution along with glutaraldehyde, magnesium chloride, and acetic acid in a laboratory padding mangle by a 2-dip/2-nip process. The padded fabric was dried and cured. The cured samples were washed at  $60^\circ\text{C}$  and dried.

### Estimation of amino groups on the finished fabric

The end amino groups of different varieties of silk were determined with the ninhydrin method as described by Chavan and Nallankilli.<sup>14</sup>

### Dyeing of the sericin-treated fabrics

The sericin-treated fabrics were dyed with 4% shade of both acid (Navimill Yellow 56N) and reactive dyes (Remazol Black B). Acid dyeing of the sericin-treated samples was carried out at  $90^\circ\text{C}$  for 60 min, with the material-to-liquor ratio kept at 1:30, at pH 5–6. The dyed samples were washed at  $60^\circ\text{C}$  and dried. Reactive dyeing was carried out in a neutral medium for 1 h at  $90^\circ\text{C}$  in the presence of 60 g/L sodium sulfate.

Kubelka Monk equation ( $K/S$ ) defines a relationship between spectral reflectance of sample and its

absorption ( $K$ ) and scattering ( $S$ ) characteristics.  $K/S$  of the dyed samples was measured with a spectrophotometer (Color Eye 7000A, Gretag Mcbeth, New York). Five readings were taken for each sample, and the average  $K/S$  values at  $\lambda_{\max}$  are reported. The reported  $K/S$  values were calculated with the following equation:

$$(K/S)_{\text{corr}} = (K/S)_{\text{dyed}} - (K/S)_{\text{undyed}}$$

where  $(K/S)_{\text{corr}}$  is the corrected  $K/S$  value,  $(K/S)_{\text{dyed}}$  is the  $K/S$  value for the sample after dyeing, and  $(K/S)_{\text{undyed}}$  is the  $K/S$  value for the sample before dyeing.

### Performance properties of the finished fabrics

The samples were conditioned for 48 h before performance testing.

The moisture content of the finished samples was measured with a Sartorius moisture analyzer (MA 51, 98648-002-76) (Goettingen, Germany). The instrument automatically calculated the moisture content on the basis of the initial and final weights:

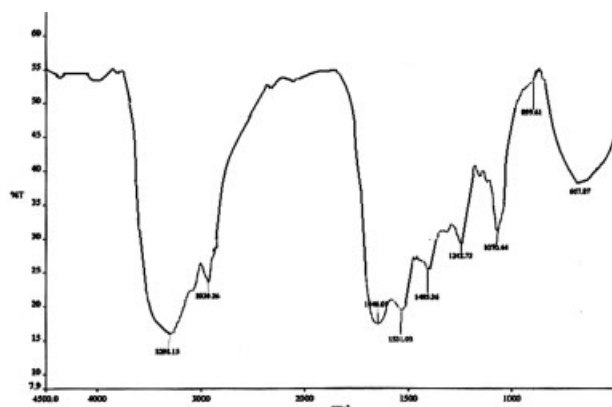
Moisture content (%)

$$= \frac{\text{Initial weight} - \text{Final weight}}{\text{Initial weight}} \times 100$$

The bending length of the finished fabric was measured according to ASTM Standard D 1388-96.

The crease recovery properties were determined in the conditioned state of the treated fabric according to ASTM Standard D 1295-67.

The antistatic property of the fabric was measured with a static honestmeter according to Japanese Standard JIS L 1094 : 1997. Results were interpreted in the form of the half-decay time in seconds. It was half of the time that the fabric needed to discharge the static charges imposed by the test machine.



**Figure 1** FTIR graph of sericin powder obtained by the salting-out process.

**TABLE I**  
FTIR Peaks of Sericin Powder Obtained by the Salting-Out Process

Amide A peak (cm <sup>-1</sup> )	Amide B peak (cm <sup>-1</sup> )	Amide I peak (cm <sup>-1</sup> )	Amide II peak (cm <sup>-1</sup> )
3298	2930	1646	1530

The mean UV protection factor (UPF) and rated UPF values of the samples were measured according to Australia/New Zealand standards in the sun penetration and protection measurement system of SDL (ATLUS, UK).

## RESULTS AND DISCUSSION

### Characterization of sericin

#### FTIR analysis

The IR spectrum of the extracted sericin is presented in Figure 1. Prominent IR transmission bands of this spectrum are listed in Table I. The peptide group of the proteins gives nine characteristic bands named amides A, B, I, II, III, IV, V, VI, and VII.<sup>15</sup> Among these, amide I is the most intense absorption band of proteins. The amide A band intensity is primarily due to the N—H stretching vibration. Amide I is primarily governed by the stretching vibration of the C=O (70–85%) and C—N groups (10–20%). Its frequency is found in the range of 1600–1700 cm<sup>-1</sup>. Amide II is found in the 1510–1580-cm<sup>-1</sup> region, and it is more complex than amide I. Amide II derives mainly from the in-plane N—H bending. The rest of the potential energy arises from the C—N and C—C stretching vibrations.

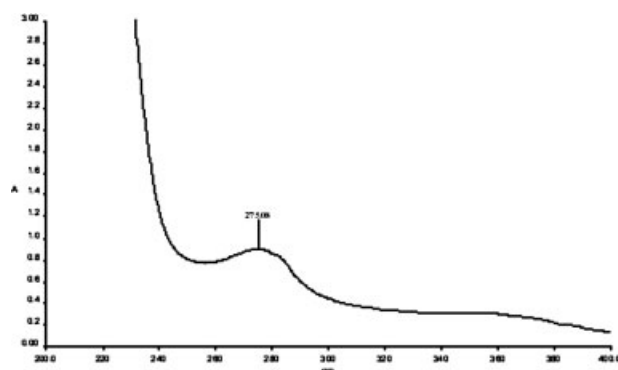
Sericin extracted by the salting-out method shows at least one peak between 1600 and 1700 cm<sup>-1</sup> confirming amide I absorption. Peaks found in the region of 1510–1580 cm<sup>-1</sup> confirm the presence of the amide II absorption band. The peaks in the region of 3000–3500 cm<sup>-1</sup> are amide A and amide B bands associated with N—H stretching vibrations. The other peaks that are found in the region of 1500–400 cm<sup>-1</sup> may be associated with the complex amide III and V bands. The FTIR peaks of the sericin powder prepared in this study are very similar to those of sericin powder obtained by other researchers.<sup>16,17</sup>

#### Nitrogen content

The nitrogen content of the sericin powder was found to be 15.8%. Wu et al.<sup>3</sup> also reported that sericin recovered from wastewater contained 14.65% nitrogen.

#### X-ray diffractogram

It has been reported in the literature that sericin is an amorphous macromolecule.<sup>18</sup> The X-ray diffracto-



**Figure 2** UV absorption spectrum of the sericin solution.

gram of the sericin powder does not show any distinct peak. This indicates that the extracted sericin powder is amorphous in nature.

#### UV absorption spectra

The UV spectrum of the extracted sericin solution is presented in Figure 2. The UV absorption spectrum

shows a characteristic peak of sericin in the region of 275 nm.<sup>19</sup> Because the sericin absorbs UV light, it may act as a UV-protective agent.

#### Optimization of the processing parameters for the crosslinking agent

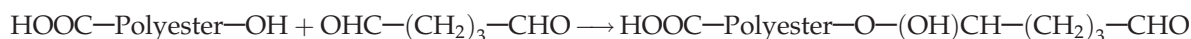
##### Concentration of the crosslinking agent

In this study, glutaraldehyde was selected as the crosslinking agent. Glutaraldehyde, having two aldehyde groups, has the ability to react with two different chemical groups simultaneously. Alcohols are better nucleophiles than water. Therefore, they add across a  $\pi$  bond of an aldehyde to form hemiacetal.<sup>20</sup> The resultant hemiacetal, having another aldehyde group on its other end, can react with the amino groups as well as hydroxyl groups of serine of sericin. The possible chemical reactions are shown in Illustrations 1–5. There is a probability of all these reactions taking place simultaneously in the padding liquor:

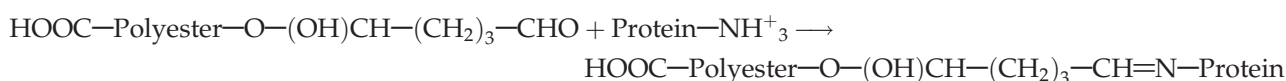
#### Illustration 1

##### Scheme A

##### Step 1



##### Step 2



##### Scheme B

##### Step 1

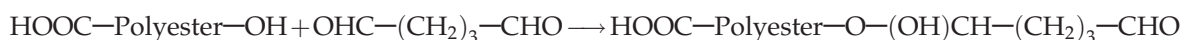


##### Step 2

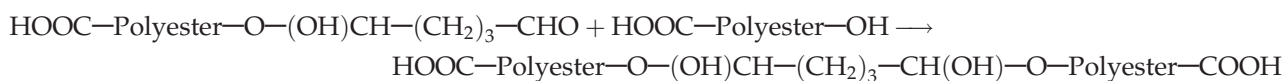


#### Illustration 2

##### Step 1



##### Step 2



#### Illustration 3

##### Step 1



Step 2

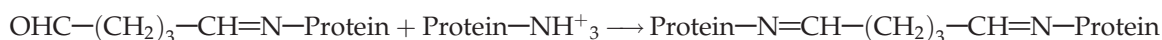
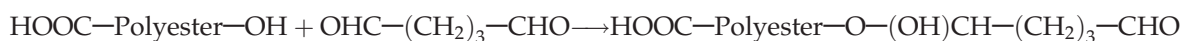


Illustration 4

Structure of serine :  $\text{HO}-(\text{NH}_2)-\text{COOH} \cong \text{Ser}-\text{OH}$

Step 1



Step 2

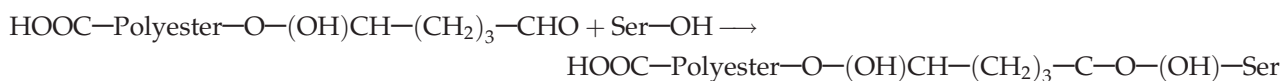
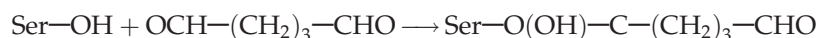


Illustration 5

Step 1



Step 2



To assess the effect of glutaraldehyde on sericin fixation, fabrics were padded with sericin (10 g/L), different concentrations of glutaraldehyde, magnesium chloride (10 g/L), and acetic acid (0.1%), dried, cured at 150°C for 3 min, and washed. The washed fabrics were dyed with the acid dye Navimill Yellow 56N (4% shade) at 90°C for 60 min and washed. Because the acid dye was attached to the amino group of sericin by electrovalent bonds, the *K/S* value was taken as an indirect indication of the sericin content. The *K/S* values of the fabrics dyed after sericin treatment with different concentrations of glutaraldehyde are given in Table II. It has been found that, with an increase in the concentration of glutaraldehyde, the dye uptake increases up to 10 mL/L; beyond this, there is no further improvement in the dye uptake. The increase in dye uptake may be attributed to the fixation of more sericin to the fabric with a higher concentration of glutaraldehyde,

which possibly becomes saturated at a concentration of 10 mL/L.

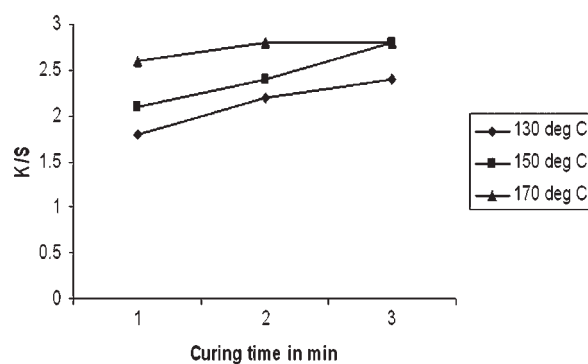
Curing conditions

Curing conditions were optimized with respect to the curing temperature and time. The pretreated polyester fabrics were padded with a solution containing (10 mL/L) glutaraldehyde, sericin (10 g/L), magnesium chloride (10 g/L), and acetic acid (0.1%). The padded fabrics were cured at different temperatures for different time periods. Then, the cured fabrics were washed and dyed with 4% Navimill Yellow 56N at 90°C for 60 min. The dyed fabrics were washed, and their *K/S* values were measured. The *K/S* values of the dyed fabrics are given in Figure 3. The *K/S* values increase with an increase in the curing temperature from 130 to 170°C. This indicates that the fixation of sericin on the fabrics increases

**TABLE II**  
Optimization of the Concentration of Glutaraldehyde

Sample code	Glutaraldehyde (mL/L)	Sericin (g/L)	MgCl <sub>2</sub> (g/L)	Acetic acid (mL/L)	<i>K/S</i> (acid dye)
G 2.5	2.5	20	10	1.0	0.8
G 5.0	5.0	20	10	1.0	1.3
G 10.0	10.0	20	10	1.0	2.5
G 20.0	20.0	20	10	1.0	2.7
G 40.0	40.0	20	10	1.0	2.5





**Figure 3** Effect of the curing temperature and time on  $K/S$ .

with an increase in the temperature of curing. The effect of the curing time on the  $K/S$  values (i.e., fixation of sericin) is more prominent at 130 and 150°C, whereas for the fabric cured at 170°C,  $K/S$  is not substantially affected by the curing time. The dye uptake of the treated fabric is maximum on curing at 150°C for 3 min.

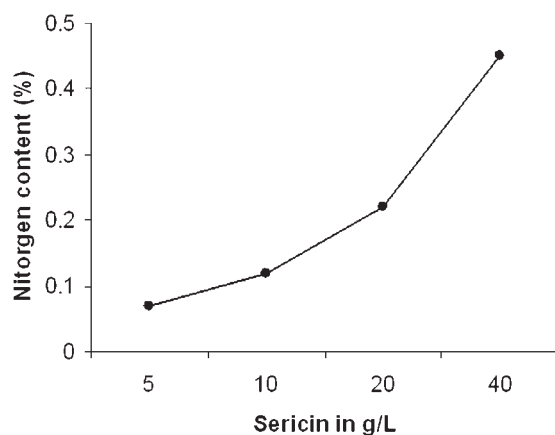
On the basis of these results, the 10 mL/L concentration of glutaraldehyde and curing at 150°C for 3 min emerged as the best conditions for the application of sericin onto polyester fabric. These conditions were employed for further studies.

### Application of sericin to polyester

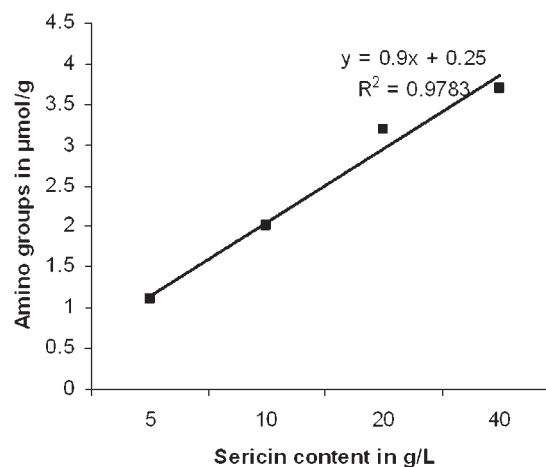
Fabrics pretreated with sodium hydroxide were padded with different concentrations of sericin ranging from 5 to 40 g/L along with optimized conditions of the crosslinking agent. The fabrics were padded in a 2-dip/2-nip process, cured, and washed. Then, the washed, dried, and conditioned fabrics were taken for characterization.

### Nitrogen content of the treated fabrics

It is known that polyester fabrics do not contain nitrogen. On the application of sericin to polyester, it



**Figure 4** Relation of the nitrogen content of the treated fabric with the sericin content in the padding liquor.



**Figure 5** Relation of the amino groups in the treated fabric with the sericin content in the padding liquor.

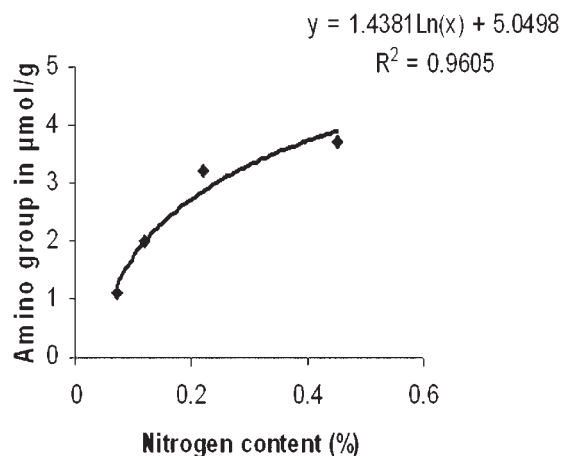
was expected that the treated fabrics would have traces of nitrogen. The estimated nitrogen content of the treated fabrics is given in a graph in Figure 4. The graph shows that with an increasing amount of sericin in the padding liquor, the nitrogen content of the treated fabrics increases. The increase in the nitrogen content of the treated fabrics indicates the presence of an increasing amount of sericin on the fabric surface. The fixation efficiency of the sericin, as calculated from the nitrogen content of the padding bath and that of the fabric, was found to be 10–14%.

### Amino group estimation of the finished fabric

The estimated amino groups of the treated fabrics are given in Figure 5. Like the nitrogen content, the amino group content also increases with an increasing concentration of sericin in the padding recipe. This is also due to the increasing amount of sericin in the treated fabrics.

### Correlation between the nitrogen content (%) and amino groups

It was expected that both the nitrogen content and amino groups would increase with the increase in the sericin content of the padding liquor. The relation of the amino end groups with the nitrogen content (%) is presented in Figure 6. The amino groups ( $\mu\text{mol/g}$ ) increase with the increase in the percentage of nitrogen in the treated fabrics. The amino groups ( $\mu\text{mol/g}$ ) can be correlated with the nitrogen content with the equation  $y = 1.4381 \ln(x) + 5.05$ . This equation gives an acceptable correlation with a coefficient of correlation of 0.96.



**Figure 6** Correlation between the amino groups and nitrogen content.

### Dyeing studies of sericin-pretreated polyester fabrics

The amino groups of sericin can interact with acid dyes to form ionic bonds. The sericin-treated fabrics also can be dyed with reactive dyes to form covalent bonds. Navimill Yellow 56N was used for acid dyeing, and Remazol Black B was used for reactive dyeing. Remazol Black B is a homobifunctional reactive dye. The reactions of sericin with acid and reactive dyes are given in Illustration 4.

The  $K/S$  values of the fabrics dyed with acid and reactive dyes are given in Table III. There is an

#### Reactive dyeing



#### Relation of $K/S$ to the amino groups

The relationship of  $K/S$  of the acid-dyed fabrics with the amino end groups is presented in Figure 7. The  $K/S$  values increase with an increase in the number of amino groups of the fabrics.  $K/S$  values of the treated fabrics can be linearly related with the amino end groups with the equation  $y = 0.93x + 0.61$ . This equation follows a very good correlation with a coefficient of correlation of 0.95.

#### Performance properties of the finished fabrics

The performance properties of the sericin-treated samples in terms of the moisture content, antistatic property, UV-protective property, crease recovery angle, and bending length are given in Table IV. The

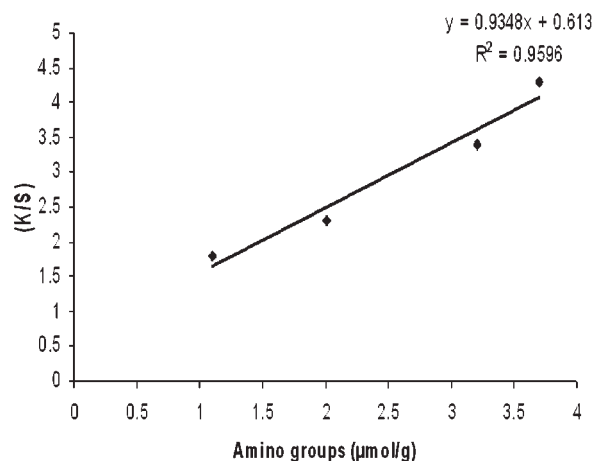
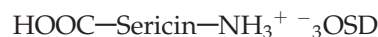
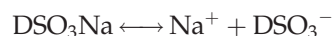
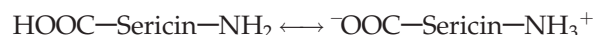
**TABLE III**  
 **$K/S$  Values of the Fabrics Dyed After Sericin Treatment**

Sample code	$(K/S)_{\text{corr}}$	
	Navimill Yellow 56N	Remazol Black B
P	0.3	0.1
UT	0.5	0.1
Blank	0.5	0.2
5SPET	1.8	0.5
10SPET	2.3	1.1
20SPET	3.4	1.6
40SPET	4.3	2.3

increase in the dye uptake with an increase in the sericin content in the padding liquor. This can be attributed to the increase in the amount of sericin on the fabrics. From the results, it is noticed that the dye uptake is comparatively much less in the original and pretreated fabrics as they do not have any amino groups on their surface.

#### Illustration 6

##### Acid dyeing



**Figure 7** Correlation between  $K/S$  of the acid-dyed fabric and the amino groups.

TABLE IV  
Moisture Content, UPF, Half-Decay Time, Crease Recovery Angle, and Bending Length Values of Sericin-Treated Fabrics

Sample code	Moisture content (%)	Mean UPF	Rated UPF	Half-decay time (s)	Crease recovery angle (°) <sup>a</sup>	Bending length (cm)
P	0.82	19.5	15	7.5	283	3.5
UT	1.47	18.6	15	4.6	286	3.2
Blank	1.43	19.0	15	4.8	284	3.1
5SPET	1.78	18.3	15	4.8	278	3.2
10SPET	1.91	20.6	15	4.2	276	3.5
20SPET	2.09	27.9	20	2.8	258	3.8
40SPET	2.34	30.9	25	2.3	247	4.3

<sup>a</sup> Warp (W) + filling (F).

original polyester fabric has a moisture content of 0.82%. On pretreatment with sodium hydroxide, the moisture content increases up to 1.47%. There is a further increment in the moisture content on the treatment with sericin. This increase depends on the sericin content of the padding liquor. A sericin concentration of 20 g/L in the padding liquor gives a moisture content of 2.09%. Similar results were reported by Lee et al.<sup>6</sup>

The absence of polar groups in polyester causes static charge generation. It has been estimated that sericin, having polar groups, can interact with the air moisture and bind the water molecules, reducing static buildup. The half-decay time of the treated fabric is lower than that of the untreated one (Table IV). This may be due to the increase in the moisture content of the fabric in the presence of sericin on the fabric surface.<sup>6</sup> No substantial change is noticed at lower sericin contents; the reduction in the half-decay time is more prominent at a higher content of sericin (20–40 g/L).

There is no change in the rated UPF of the fabrics at a lower sericin content. The rated UPF increases considerably at higher levels of sericin (20–40 g/L). This shows that polyester fabric treated with sericin has a UV absorption property.

The fabrics treated with sericin up to a 20 g/L concentration do not show any change in their bending length. At a 40 g/L concentration of sericin, there is a change in the bending length, indicating that the fabric becomes stiff. The crease recovery angles of the treated fabrics are less than that of the controlled one.

## CONCLUSIONS

Sericin was prepared from a boiled solution of silk cocoons. Salting-out with ammonium sulfate, followed by filtration and drying, resulted in sericin powder. The obtained sericin powder had a nitrogen content of 15.8%. In the FTIR spectra, it showed distinct amide A, amide B, amide I, and amide II peaks. Sericin was applied to polyester fabric with glutaraldehyde as a crosslinking agent in a pad-dry-cure process.

The presence of amino groups on the finished fabric imparted acid and reactive dyeability to the polyester fabrics. Fabrics treated with higher sericin content in the finishing liquor had a higher percentage of nitrogen, more amino groups, and higher dye uptake. A good correlation was found between the acid dye uptake and the number of amino groups. The sericin concentration of 20 g/L in the padding liquid was sufficient to improve the comfort properties of the polyester fabric, that is, the moisture content (2.09%) and antistatic property (half-decay time = 2.8 s), without affecting the feel of the fabric. In addition, the fabric showed a UV absorption property.

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