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Sericin based bioactive coating for polyester fabric

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In this study, a process has been developed for durable coating of sericin on polyester. Pre modification of polyester with alkali (sodium hydroxide) has been carried out to incorporate polar entities on its inert surface. 10g/L of sericin with 30mL/L of glutaraldehyde, cured at 130°C for 2min has been optimized for its application on modified surface. Treated samples are tested for surface smoothness, moisture retention, wicking, antistat and antioxidant characteristics. Results show that polyester fabric surface becomes smoother as well as highly hydrophilic on application of sericin. Wicking properties are enhanced greatly and antisat property is improved along with the radical quenching property. These results can be used to prepare fabric having unique properties of enhanced smoothness, hygroscopicity, high wicking and radical quenching which make it suitable for applications in skin moisturizing, skin healing and anti-ageing. These results indicate that sericin can be used to develop a durable and bioactive finish on polyester for use in medical and sports garments.

Keywords: Bioactive coating, Medical garments, Polyester, Sericin, Silk, Textile finishing

1 Introduction

Polyester (PET) has acquired a major position in textile and apparel trade across the world due to its competitive pricing and versatile performance characteristics. More recently, polyester is also being considered a good candidate for biomaterial applications due to its excellent physicochemical properties, good mechanical strength and transport properties, high chemical and thermal stability with low permeability to gases. However, PET has certain undesirable features such as poor water permeability and wettability, low moisture regain and static built up due to which it is uncomfortable to wear and can cause itching and rashes on the skin of some users[1, 2]. Application of suitable biocompatible finishes can help mitigate some of these problems and make it more appropriate for specialized applications.

Some researchers have reported studies on applying sericin as a finishing agent on natural and man-made textiles. Since polyester is non-polar and hydrophobic in nature, it is difficult to apply any finish directly on it. In most of the studies, polyester has been modified either by chemical hydrolysis or by irradiation prior to application of sericin. This incorporates functional groups onto the surface which increases the hydrophilicity of the polymer for subsequent reactions[3, 4].

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Chemical approaches based on grafting and crosslinking have been used to fix a protein macromolecule such as sericin on PET. Yamada and Matsunaga[5] treated knit fabrics of hollow porous polyester fibres with an alkali solution to cause weight loss upto 15%. Further sericin was applied using glycerylpolyglycidyl ether and diethylenetriamine as crosslinking agents. The fabric was heat treated for 30s at 160°C to obtain water absorption height 12.3 cm, as against 5.2 cm for a fabric of conventional PET.

Lee *et al.*[6] grafted N-vinylformamide onto PET swollen by benzyl alcohol using 'film seal method' of electron beam irradiation technique. After the pretreatment, the fabric surface was hydrolyzed by sulphuric acid and sericin was fixed on it using ethylene glycol diglycidyl ether. A significant increase in hygroscopicity and antistatic properties was observed while the smoothness property remained unaffected. Jin *et al.*[7] applied sericin to ethylenediamine-pretreated polyester fabric along with chloromethyloxirane or cyanuric chloride as a crosslinking agent. Finished fabrics showed improved moisture absorption ability with increased harshness of sericin-treated fabrics. Some patents have also been granted on the finishing of polyester by sericin to improve its hygroscopicity. Most of the patents are being held by Seiren Co Ltd of Japan[5, 6]. Gulrajani *et al.*[8] treated PET fabric with alkali followed by application of sericin. The performance properties such as the moisture content, UV absorption, antistatic, crease recovery, of the treated fabric were tested and a noticeable improvement was observed.

This study was undertaken with the objective of developing textiles which can be used by patients suffering from specific medical conditions such as skin itching, rashes and atopic dermatitis. Atopic dermatitis is a chronic itchy and inflammatory skin disease which is extremely difficult to treat. Although effective therapeutic agents are available, they may have long-term toxic side effects. It has been suggested that non-drug based approaches such as clothing having high moisture sorption and smoother surface can be used for patients suffering from such ailments[4, 9, 10]. In this study, sericin was applied to PET fabric and treated fabrics were tested for moisture related properties, comfort characteristics, antimicrobial, antioxidant and antistat properties.

2 Materials and Methods

2.1 Materials

Mulberry silk waste was procured from Central Silk Research Institute of Technology, Central Silk Board, Bangalore, India. It was cleaned manually to remove insect waste and other impurities before use. Hundred per cent polyester fabric having plain weave, 90g/m^2 (gsm), 56 ends/cm; 34 picks/cm was used for the study. Reagent grade chemicals like sodium hydroxide (Merck), glutaraldehyde (Merck), magnesium chloride (Merck), acetic acid (Merck) and potassium chromate (Merck) were used. Analytical grade chemicals, sodium carbonate (Qualigen), sodium hydroxide (Qualigen), copper sulphate (Merck), sodium potassium tartarate (Merck), Bovine serum albumin (Spectrochem), Folin Ciocalteau's phenol reagent (Merck) were used for protein estimation. Deionized water was used for all the experiments. Lissapol N (a commercial product) was used as a non-ionic surfactant for scouring.

2.2 Methods

2.2.1 Extraction and Characterization of Sericin

Silk waste was treated in deionized water for 40 min at 100°C at a material-to-liquor ratio of 1:20 to extract sericin[11]. Infrared dyeing machine DLS 7000 (Daelim Scarlet, Korea) was used for the purpose. The protein content in sericin liquor was assayed by Lowry's method using bovine serum albumin (BSA) as a standard. Ultraviolet spectrum of sericin samples was recorded on a D-2750 UV-Vis spectrophotometer (Shimadzu, Singapore). Absorbance of solutions was measured in the range of 260–290 nm. A-ratio (A_{280}/A_{260}) was calculated by recording the absorbance value of sample at 280 nm and 260 nm, to determine the purity of protein obtained.

2.2.2 Modification of Polyester

PET fabric samples were scoured using Lissapol N to remove any impurities and temporary spin finishes that might be present on the fabric. PET is chemically inert, has no available functional groups and no affinity for an ionic protein like sericin. Hence, it was pretreated with alkali- 1M (40g/L) NaOH at 80°C for 45 min with 1:100 material-to-liquor ratio to create functional groups on its surface, prior to application of sericin. The weight loss in PET after this treatment was found to be ~6-7%.

2.2.3 Application of Sericin

Sericin was applied on modified PET with and without the use of a crosslinking agent. Effect of *p*H on uptake of sericin by modified PET was studied. Glutaraldehyde was used as a crosslinking agent.

*Effect of p*H— 20g/L of sericin solution was used. Since affinity is a function of ionic charge, solutions of *p*H 4, 5, 6 and 7 were used for application. *p*H was adjusted using 10% acetic acid. Alkali treated fabrics were padded (80% expression) with the sericin solution in a laboratory padding mangle by a 2-dip/2-nip process. The padded fabric was dried at 80°C for 3min and cured at 130°C for 2min. Cured samples were then washed and dried.

Application of Sericin with Crosslinking Agent—On pretreatment with alkali, carboxyl and hydroxyl groups are created on the surface of PET. However, sericin cannot be attached directly to the functionalized surface. Therefore, glutaraldehyde (GTA) was used as a crosslinking agent to attach sericin to alkali modified PET.

The concentration of sericin was kept at 20g/L for all the runs. To optimize the conditions of GTA crosslinking, a three level, three factorial Box and Behnken design (BNB) was used in this study. Box-Behnken design requires a lower number of actual experiments be performed, which facilitates probing into possible interactions between the parameters studied and their effect on dependant variables. Factors studied in the BNB experimental design were concentration of GTA, time and temperature of curing (Table 1).

The factor levels were evenly spaced and coded as –1 (low), 0 (central point) and +1 (high). Treated samples were dyed with a basic dye to estimate the amount of sericin deposited. The higher the colour value, the more is the amount of sericin applied. The selected response was colour value (*K/S*) of treated samples. A set of 15 experiments was performed using the above- mentioned scheme. The treatment conditions are given in the Table 2.

The analysis of data was carried out using design expert software (version 8). The module applied a quadratic polynomial equation to analyze the relationship of each response with the independent variables, as shown below:

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_1X_2 + b_5X_2X_3 + b_6X_1X_3 + b_7X_1^2 + b_8X_2^2 + b_9X_3^2 + E \quad \dots (1)$$

where b_0-b_9 are the regression coefficients; X_1, X_2 and X_3, the factors studied; E, an error term; and Y, the measured response namely colour values or *K/S* (Y_1). The statistical significance of the model equation and the multiple coefficient of determination (R^2) were also determined.

2.2.4 Characterization of Treated Fabric

All samples were conditioned for 48 h before testing. Following characterization tests were carried out.

Table 1—Variables used in the Box and Behnken design

Independent variable	Level used		
	Low(-1)	Medium(0)	High(+1)
GTA conc., mL/L (X_1)	10	20	30
Curing temperature, ^{0}C (X_2)	110	120	130
Curing time, min (X_3)	1	2	3

Table 2—Box and Behnken surface response design for crosslinking of sericin with GTA

Case	GTA conc., mL/L (X_1)	Temperature, °C (X_2)	Time, min (X_3)
1	10	110	2
2	30	110	2
3	10	130	2
4	30	130	2
5	10	120	1
6	30	120	1
7	10	120	3
8	30	120	3
9	20	110	1
10	20	130	1
11	20	110	3
12	20	130	3
13	20	120	2
14	20	120	2
15	20	120	2

SEM Study

Surface morphology of untreated and alkali modified polyester fabrics was studied under the scanning electron microscope EVO 50 (Zeiss, Germany) at 10K magnification. Also the samples treated with sericin after alkali modification were examined under scanning electron microscope.

FTIR-ATR Study

The FTIR-ATR spectra of samples was recorded on BX FTIR spectrophotometer (PerkinElmer, US) in order to determine the presence of functional groups in untreated and sericin treated PET samples.

Weight Add-on

Add-on was determined by measuring the difference in the weight of the conditioned untreated sample and sericin treated sample. It was calculated using the following equation:

$$\text{Add-on (\%)} = \frac{(W_2 - W_1)}{W_1} \times 100 \quad \dots (2)$$

where W_1 is the initial weight of the sample; W_2, the weight of sericin finished sample.

Whiteness Index

Whiteness index of the treated fabrics was measured by Gretag Macbeth, Color eye-7000A using colour eye control software. Three readings were taken for each sample, and the average values are reported.

Dyeing with Basic Dye

Since 80% of sericin is composed of amino acids that contain a high proportion of hydroxyl groups such as aspartate, serine and glycine, it can be treated with basic dyes. Basic dyes have amino or alkylamino groups, as their auxochromes. Hydroxyl groups of sericin can interact with these basic dyes to form ionic bonds (Scheme 1). Hence, the amount of methylene blue dye absorbed can be taken as an indirect measure of the amount of sericin applied on the textile.

$$\text{Dye } (D^+) + {}^-\text{OOC-Sericin-}NH_3^+ \longrightarrow D^{+-}\text{OOC- Sericin- }NH_3^+$$

Scheme 1

Sericin treated fabrics were further treated with 0.5% shade of basic dye (Methylene Blue) at 100°C for 60 min, with the material-to liquor ratio at 1:30. The treated samples were washed at 60°C and dried. *K/S* of the samples was measured with a spectrophotometer (Color Eye 7000A, Gretag

Macbeth, New York). Three readings were taken for each sample, and the average *K/S* values were recorded.

Bending Length and Tensile Strength

The bending length was measured according to ASTM Standard D 1388-96. The tensile strength of alkali treated fabric was measured in accordance with ASTM D5034 test method (grab tensile method).

Friction and Roughness of Fabric

Surface properties including friction (resistance / drag) and surface contour (roughness) were determined using the KES-FB4 automatic surface tester (Kato Tech Co. Ltd.).The equipment uses a sensor (designed to simulate the human finger) placed on the fabric surface while applying a total load at 50gf. Surface roughness is measured with the help of a vertical sensor which touches the fabric with a constant force of 10gf.

Durability of Sericin Finish

Durability of sericin finish applied by using crosslinker was assessed by washing the dyed sample with 5g/L of Lissapol N for 45min at 50°C (ISO 2). The samples were washed upto 3 washing cycles. After each wash a sample was taken out, dried and its *K/S* value was measured.

Testing of Comfort Characteristics

Air Permeability

TEXTEST air permeability tester (FX3300) was used to measure the air permeability at 98 Pa air pressure. ASTM D737 test standard was followed. An average of 3 readings for each sample was recorded.

Moisture Regain

Moisture regain was measured in accordance with ASTMD 629-99 by comparing the dry weight and moisture conditioning weight of sericin treated PET fabrics. Samples were dried in an oven at 105°C for 1 h and then weighed after cooling in a desiccator for 30 min. Moisture conditioning was carried out at 20°C and 65% relative humidity for 24 h. The moisture regain was calculated using the following equation:

$$\text{Moisture regain (\%)} = \frac{W_m - W_d}{W_d} \times 100 \quad \ldots (3)$$

where W_m is the weight of fabric in moisture equilibrium state at 20°C and 65 % relative humidity; and W_d , the weight of fabric dried at 105°C for 90 min

Drop Test

The vertical drop test of fabrics was measured in accordance with BS 4554. A drop of distilled water was allowed to fall onto the fabric sample, and the time taken by the liquid to get absorbed by the fabric completely was recorded. The shorter the time, the more wettable is the fabric.

In Plane Wicking

Gravimetric in-plane wicking tester was used for testing the wicking of fabrics. Sample of 12 cm diameter was used. Three tests were carried out for each sample and average values were plotted as amount of water absorbed (g) against time (s).

Vertical Wicking

Wicking property of samples was evaluated using the wicking strip test according to standard DIN53924. Specimen of size 200 × 25 mm, the length parallel to warp direction was prepared. Each specimen was suspended vertically with its lower end immersed in a reservoir of distilled water to which dilute potassium chromate (0.5%) was added for tracking the movement of water. Potassium chromate was chosen instead of a dye because it is an inorganic salt without having any affinity towards the fibres. After various intervals of time (1, 3, 5, 7, 10min) the height reached by water in the fabric, above the water level in the reservoir, was measured with a scale.

Performance Characteristics

Anti-static Property

A piece of fabric to be evaluated is rubbed briskly on a piece of plastic or rubber. The fabric is then placed over an ash tray containing cigarette ash. The amount of ash transferred to the fabric is an indication of the amount of static charge imparted to the fabric. This test is mainly used as qualitative tool.

Antimicrobial and Antioxidant Property

Qualitative assessment was made by agar diffusion method (AATCC-147). Antioxidant property was measured by estimating the free radical scavenging activity (RSA) of sericin finished PET fabrics. The capacity of the fabric to scavenge the stable free radical using 2,2, diphenyl-1-picryl hydrazil (DPPH) was monitored according to the method proposed by Wu *et al.*[12] with a few modifications. Sample of size 1” × 1” was added to 3.5mL of freshly prepared DPPH radical in a methanol solution (0.1M) and vortexed. After reaction for 25min at 25°C at room temperature in the dark, reaction mixtures were centrifuged at 8000rpm for 30min. The decolourizing result of the supernatant was assayed at 517nm. In addition a blind control containing 3.5ml DPPH and untreated PET instead of was also assayed. The following equation was used to calculate the RSA:

$$\text{Scavenging activity (\%)} = 1 - \frac{OD_{sample}}{OD_{blind}} \times 100 \qquad \dots (4)$$

where scavenging activity refers to the RSA percentage; OD_{sample} refers to the absorbance of the sericin treated sample; and OD_{blind} refers to the absorbance of the blind control.

3 Results and Discussion

3.1 Surface Modification of Polyester

Since sericin cannot be applied directly to PET, so PET fabric was pretreated with 40g/L of NaOH at 80°C for 45 min. Treatment with sodium hydroxide brings about changes in mechanical as well as physico-chemical properties of PET fibres. Weight loss of 6-7% and strength loss of almost 30% was observed in both warp and weft directions after alkali treatment.

Morphological structure of untreated and alkali treated samples was studied by SEM analysis at 10K magnification (Fig. 1).

Surface of untreated PET fibre (A) was observed to be smooth. After treatment with alkali, PET surface shows creation of distinct pits and cavities of about 1.1 × 0.5 ± 0.16 µm in size on the surface (B). Residual material generated due to erosion is also seen adhering to the surface. These effects lead to a micro level roughness which allows better adhesion of finishes and coating on the surface. Similar results have been reported by Dave *et al* [13].

Chemical functionalization of the fibre surface was studied by FTIR. Strong absorption at 1708.85 cm^{-1} and C-O-C stretching vibration band at 1089.25 cm^{-1} confirm ester linkage in PET. The absorption of 1237.08 cm^{-1} corresponds with the asymmetric stretching vibration of ring ester CC and C=O in plane bending[14]. Chain scission occurring at the ester linkages leads to creation of carboxyl and hydroxyl end groups on the surface (Fig. 2) [15].

Treatment with alkali induces an additional peak at 2908 cm^{-1} and 2361.05cm^{-1} attributed to carboxylic groups (-COOH) introduced on the surface due to hydrolysis of ester linkage[14]. Absorption bands on application of sericin indicate no frequency shifts or redistribution of bands. However, a sharp peak was observed at 1440cm^{-1} which is the characteristic peak of sericin, thus indicating application of sericin[16]. Intensity at 2361.05cm^{-1} was observed to reduce indicating involvement of carboxyl groups in formation of bond with sericin.

3.2 Assessment of Sericin Quality

Spectrum of extracted sericin liquor was recorded in the UV range from 220 nm to 310 nm. Assessment of protein quality was done by calculating the A ratio (A_{280}/A_{260}). A-ratio of pure protein is around 1.8(ref.17). However, A ratio of sericin extracted from silk waste was found to be 1.09. Protein estimation in sericin was

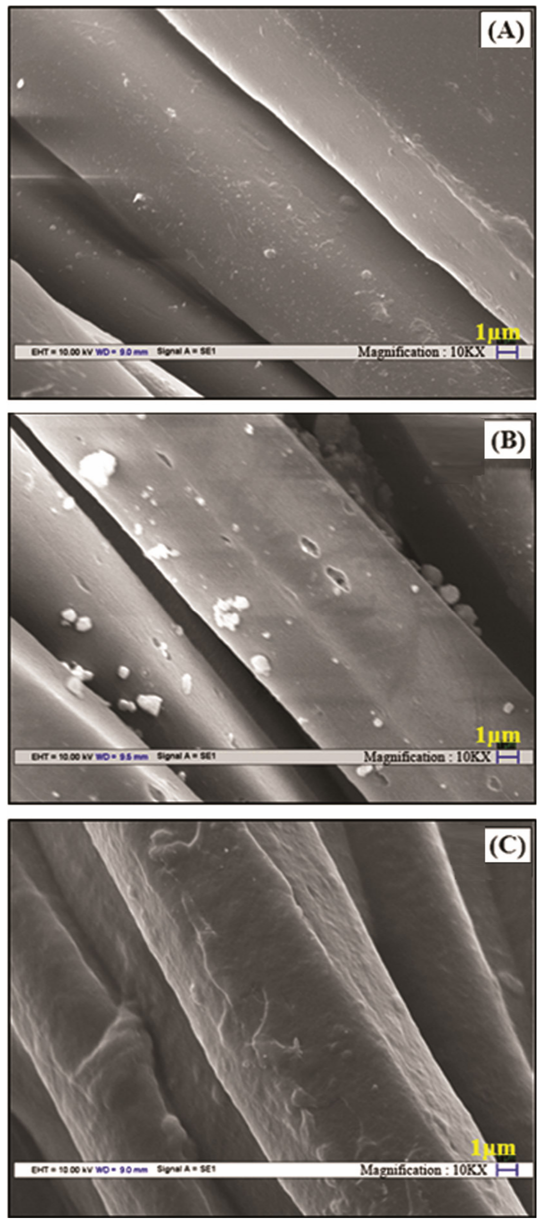

Fig. 1—SEM images of polyester (A) control, (B) alkali treated and (C) treated with alkali and 10g/L of sericin

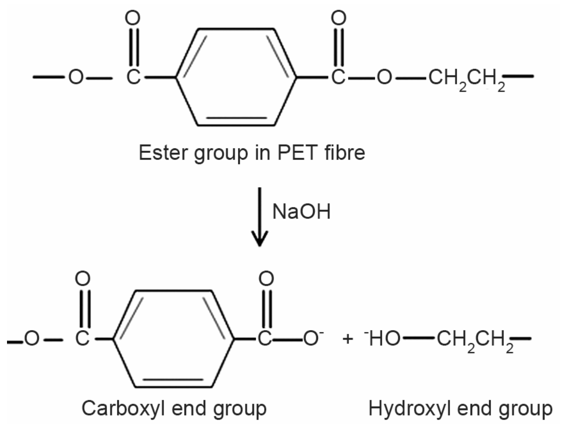

Fig. 2—Alkali hydrolysis of polyester[15]

carried out by Lowry's assay. Concentration of protein as estimated by Lowry's assay is found to be 20g/L.

Qualitative estimation of sericin in extracted liquor was carried out using spectroscopic analysis. Proteins absorb strongly in ultraviolet region mainly due to peptide bonds and aromatic acids. The aromatic amino acids like tryptophan, tyrosine and cysteine are known to absorb in the range of 260 – 290 nm due to $\pi \longrightarrow \pi^*$ transition and can be used to assess the quality of sericin protein[16]. The UV spectrum shows the characteristic peak of sericin in the region of 275.40 nm.

3.3 Application of Sericin on Alkali Modified Polyester

3.3.1 Application of Sericin at Different pH

Since sericin is amphoteric in nature, the net ionic charge on it changes, depending on the pH of the application bath[18]. To find out the optimum concentration for application of sericin, baths having *p*H of 4, 5, 6 and 7 were prepared. Sericin was found to precipitate at and below its isoelectric point i.e. *p*H 4. Thus, application was only carried out at *p*H 5, 6 and 7. The amount of sericin attached onto PET surface was estimated by dyeing with basic dyes and measuring the *K/S* value of dyed samples. Results of application are shown in Table 3.

Table 3—*K/S* of test samples used in the study

Sample code	*p*H	*K/S*
UT	-	0.58
S_{20}		0.59
AT	-	0.80
ATS_{20}	5	0.98
ATS_{20}	6	0.89
ATS_{20}	7	0.84

UT– control, S_{20}- treated with 20g/L sericin, AT– only alkali treated and ATS_{20}–alkali treated with 20g/L sericin.

It can be seen that *K/S* of samples treated at *p*H 6 and 7 is negligible while there is almost 20% increase in *K/S* values of samples treated at *p*H 5. It can also be seen that there is no change in *K/S* when sericin is applied directly to PET (S_{20}) while alkali treated fabric (AT) shows higher value of *K/S*. As a result, *p*H 5 is used for sericin application in all subsequent studies.

Treated samples were washed to check for durability of treatment. Results show that all sericin gets washed off, indicating poor adhesion of finish on the fibre. Alkali treatment creates negatively charged groups on PET. Sericin too has an abundance of negatively charged hydroxyl groups. It could be because of the presence of similar charge on the two materials, that sericin is only superficially attached on the surface and no bond formation occurs with PET. Accordingly it has been decided to use a common crosslinking agent [glutaraldehyde (GTA)] to bring about better attachment of sericin on PET.

3.3.2 Optimizing the Process Parameters for GTA Application

In this study, a 3^3 Box and Behnken statistical plan was used to optimize the process conditions for use of GTA for crosslinking of sericin on PET fabric. The experimental results were analyzed by RSM and the following response surface equation was obtained for the dependent variables i.e. Y_1.

$$K/S \text{ values } (Y_1) = 1.2025 + 0.05275X_1 - 0.016375X_2 + 0.27375X_3 - 0.000350X_1X_2 + .0015X_2X_3 + 0.00025X_1X_3 - 0.000163X_1^2 + 0.000113X_2^2 - 0.09375X_3^2 \quad \ldots (5)$$

The 20g/L sericin was used for application, whereas the concentration of GTA (X_1), temperature (X_2) and the time (X_3) of crosslinking were varied. Effect of these parameters on the concentration of sericin fixed on polyester, as measured by *K/S* value (Y_1). *P* values of GTA concentration, time and temperature of curing were to be observed less than 0.05. Thus, all the three parameters were found to be having a significant effect on *K*/*S*. From the analysis of measured responses obtained by the design-expert software, the quadratic model is found to be statistically significant for the variable i.e. colour value (*K/S*). The regression coefficient R^2 for *K/S* value is estimated to be 91.4% respectively. The response surface plots representing the colour values (*K/S*) of sericin treated PET fabric are shown in Fig. 3.

K/S values are used as an indirect measure to assess the amount of sericin attached onto the fabric surface. It can be seen that as the GTA concentration and curing temperature increase the *K/S* values increases, thus indicating better fixation of sericin due to increased concentration of crosslinker. Maximum values of *K/S* are observed at GTA concentration of 30mL/L and curing temperature of 130°C.

Glutaraldehyde (GTA) gives good attachment of sericin on modified PET because it has two aldehyde groups, and can thus react with two different chemical groups simultaneously. One aldehyde group of GTA reacts with alcohol group of modified polyester to give a hemiacetal. This hemiacetal, having another aldehyde group on the other end, can further react with the amino groups of sericin, thus resulting in its fixation over PET surface as shown in Fig. 4 (ref. 8).

Interestingly, the colour values are found to increase on increasing the time of curing from 1 to 2 min but no increase in *K/S* values is observed at 3min. Thus, 2min is taken as the optimum time for curing.

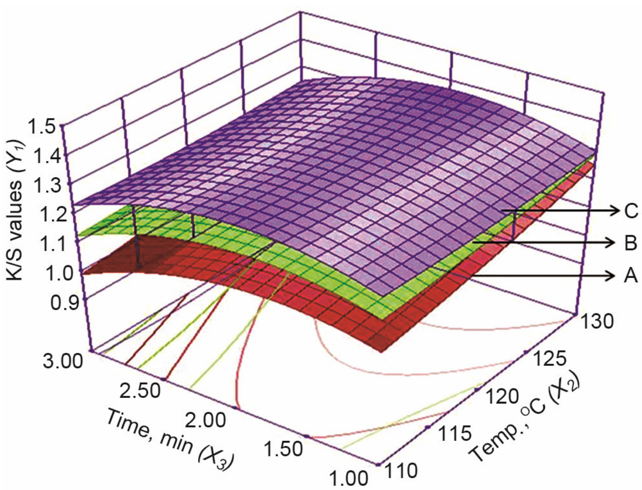

Fig. 3—Response surface plots for the *K/S* values (Y_1) versus temperature and time at different GTA concentrations (A—10mL/L, B—20mL/L, and C—30mL/L)

On the basis of the measured responses, optimum conditions for application of sericin on PET are found to be 30 mL/L of GTA and cured at 130°C for 2 min. These conditions are used for preparing the subsequent samples.

3.4 Characterization of Sericin Treated Fabric

3.4.1 Effect of Sericin Concentration on Physical Properties

Since sericin is an expensive product, it is necessary to determine the best concentration of sericin that should be used for obtaining the required performance characteristics on PET fabric. Once the optimum parameters for GTA crosslinking are identified, the next set of experiments is carried out to find the suitable concentration of sericin. Fabric samples were treated with different concentrations of sericin ranging from 5g/L to 25 g/L and then crosslinked using the optimum conditions identified earlier.

K/S values of fabrics treated with different concentrations of sericin and dyed with methylene blue were observed. Increase in *K/S* from 0.82 to 1.42 is observed in all samples treated with sericin. Amount of sericin deposited on the fabric increases with increase in concentration of sericin in application bath. Corresponding weight add on for various concentrations of sericin is found to vary between 0.3% for 5 g/L of sericin to a maximum of 1.5% for 25g/L of sericin. Significant increase in weight is observed at 10g/L concentration of sericin, however no significant difference is observed when concentration is increased from 20g/L to 25g/L, indicating a saturation limit. All subsequent tests were carried out using 10g/L and 20g/L of sericin concentration. Whiteness index of samples was measured to observe if there is any change in whiteness of samples (Table 4).

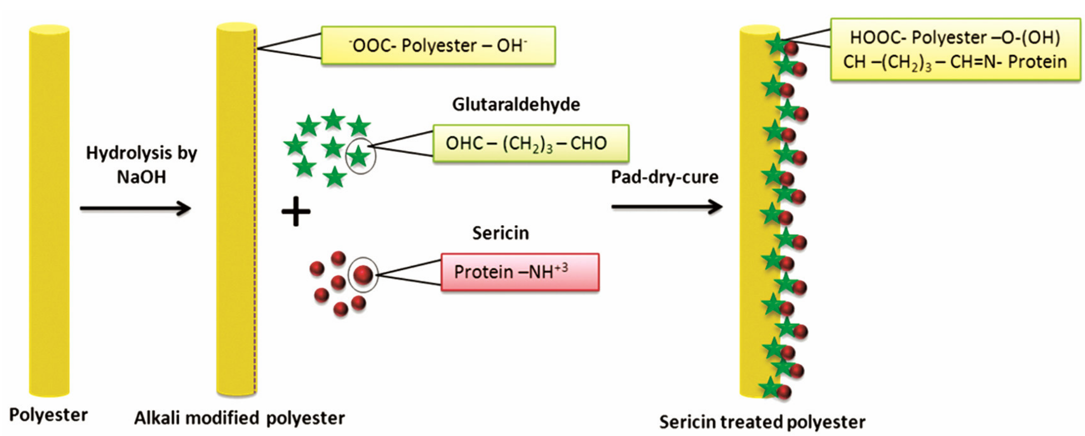

Fig. 4—Graphic representation of sericin application on modified PET using GTA

It can be seen that whiteness of fabric reduces with increase in concentration of sericin applied. Sericin powder used in the study has a slight yellow colour which is probably causing the yellowness. Wherever whiteness is a consideration, sericin can be bleached before use to take care of this matter. Feel of the fabrics, especially for use by patients suffering from skin ailments is very important. Thus, bending length of the samples was measured and results are reported in Table 4. It can be seen from the data that bending length reduces when PET is treated with alkali. All subsequent treatments lead to some increase in the stiffness of fabric with increase in concentration of sericin. This is expected as sericin is a high molecular weight compound and forms a film on the fabric surface which causes stiffness[16].

Samples treated with 10 and 20g/L of sericin were tested for durability of treatment to washing. *K/S* value of samples is recorded after each wash till 3 washes. It can be seen that the fabric treated with 10g/L of sericin is durable to washing since no decrease in colour values (1.22) on washing has been observed. However, sample treated with 20g/L sericin loses approximately 11% of its colour strength after first wash followed by further 2-3% reduction after the 3rd wash. *K/S* of sample without washing has been observed to be 1.40. 10g/L concentration is durable to washing because all of it is likely to be held by covalent bonds. Anything more than this concentration is only superfluously attached and gets removed during washing.

3.4.2 Comfort Characteristics of Treated Fabrics

Textile fabrics have an inherent roughness on the structure due to the yarn and weave structure. It has been suggested that a smoother fabric surface having good water transport properties and high breathability can have therapeutic effect on patients suffering from skin ailments. To study the effect of sericin coating on the comfort properties, treated fabric was tested for air permeability and surface friction and roughness (Table 5).

Analysis of surface characteristics shows that untreated and alkali treated PET have same surface friction and geometrical roughness. This is interesting as it indicates that the micro level roughness observed on the fibre surface in SEM analysis due to pitting, does not affect the roughness or friction characteristics, as experienced by the user.

Application of sericin enhances the surface smoothness as indicated by a lower value of MIU as well as SMD for ATS_{10} and ATS_{20}. In comparison to control, there is around 50% decrease in friction of sericin finished fabric, indicating that fabric surface is becoming smoother after the sericin treatment. This effect can be attributed to the formation of a smooth and continuous film of sericin on the fabric surface as seen in SEM pictures [Fig. 1 (C)]. Since sericin is polar in nature and therefore more hydrophilic than PET, its presence can reduce the coefficient of friction on fibre surface.

As shown in Table 5, air permeability of alkali treated PET is 32% higher than that of untreated. This could be because of alkaline hydrolysis which destroys the outer skin of PET fibres, thus making them thinner. Reduction in fibre diameter can lead to an increase in the inter yarn spaces in the fabric. However, a further increase in air permeability is observed in samples treated with 10g/L of sericin. This could be due to the coating of sericin which acts as an adhesive between fibres and binds them, thus further increasing the air spaces between yarns. To confirm this hypothesis, diameter of yarns was measured under Leica microscope. It is found that the average diameter of yarns changes from 2mm for untreated yarns to 1.8mm after alkali treatment and further reduced to 1.6mm after sericin application. Air permeability is found

Table 4—Properties of PET treated with varying concentration of sericin

Sample code	Weight add on %	Whiteness index	Bending length cm
UT	-	114.2	4.8
AT	-	111.05	3.8
ATS_5	0.3	103.51	4.6
ATS_{10}	1.1	102.29	5.8
ATS_{20}	1.4	95.38	6.3
ATS_{25}	1.5	88.96	6.4

UT–control, AT–alkali treated, ATS_5– alkali and 5g/L sericin, ATS_{10}– alkali and 10g/L sericin, ATS_{20}– alkali and 20g/L sericin, ATS_{25}– alkali and 25g/L sericin.

Table 5—Comfort characteristics of test fabrics

Sample	Surface characteristics		Air permeability $cm^3/cm^2/s$
	Friction (MIU)	Geometrical roughness (SMD)	
UT	0.2	3.3	5.88
AT	0.2	3.4	7.82
ATS_{10}	0.13	2.8	11.24
ATS_{20}	0.11	2.8	8.5

UT- control, AT- alkali treated, ATS_{10}- alkali and 10g/L sericin, ATS_{20}- alkali and 20g/L sericin.

lower for samples treated with 20g/L of sericin. This could be due to the reason that higher amounts of coating can clog up the yarn spaces and reduce the fabric porosity and hence the air permeability.

3.4.3 Moisture Management Properties of Sericin Treated Samples

The moisturizing effect of sericin is well established and a wide range of skin moisturizing and hair treatments based on sericin is commercially available[19,20]. But the moisture management properties of sericin on textiles has not been studied enough. The following paragraphs report the findings of moisture related properties of sericin treated fabrics. Moisture regain, in plane wicking and vertical wicking properties have been studied.

Moisture Regain

Moisture regain is the tendency of most fibres to pick up or give off ambient atmospheric moisture until they reach equilibrium moisture content at a given temperature and humidity level. Untreated PET has a low moisture content of 0.60±0.10%. Polar groups generated on the surface after alkali hydrolysis lead to improved hydrogen bonding capacity with water molecules, and thus better water wetting.

It can be seen from the results (Table 6), that on pretreatment with sodium hydroxide, the moisture content increases up to 0.88± 0.20%. Since sericin is composed of 80% amino acids that contain hydrophilic groups such as serine, aspartate and glycine it can absorb moisture very well[21]. A further increment in the moisture content on treatment with sericin is observed. Sericin concentration of 10g/L and 20 g/L gives a moisture content of 1.84± 0.10% and 2± 0.15%.

In Plane Wicking

Transverse wicking is the most significant factor that affects garment comfort. It is a unique phenomenon with respect to the water transfer behavior of the fabrics, since it has no directional effect[22]. It is evident from the results (Fig. 5) that there is a slight increase in the in plane wicking of alkali modified PET in comparison to control fabric. The water uptake (in grams) of sericin treated fabrics is found to increase by upto 23% as compared to control fabric. This establishes that even hydrophobic fibres like PET can be imparted high water binding capacity by treating them with sericin. The high hydroxy amino acid content of sericin, coupled with its amorphous nature makes it highly hydrophilic[21].

Table 6—Moisture related properties of test fabrics

Sample	Moisture regain, %	Vertical wicking height rise (cm) in 10min
UT	0.60 ± 0.10	7.8
AT	0.88± 0.20	12.3
ATS_{10}	1.84± 0.10	12.8
ATS_{20}	2± 0.15	12.6

UT– control, AT– alkali treated, ATS_{10}– alkali and 10g/L sericin, ATS_{20}– alkali and 20g/L sericin.

Vertical Wicking

Fabric wicking is a function of the capillary movement of water through the fabric. Capillary action occurs when the adhesive intermolecular forces between the liquid and the substrate are stronger than the cohesive intermolecular forces within the liquid[23]. It can be seen from Fig. 6 that the wicking in fabric

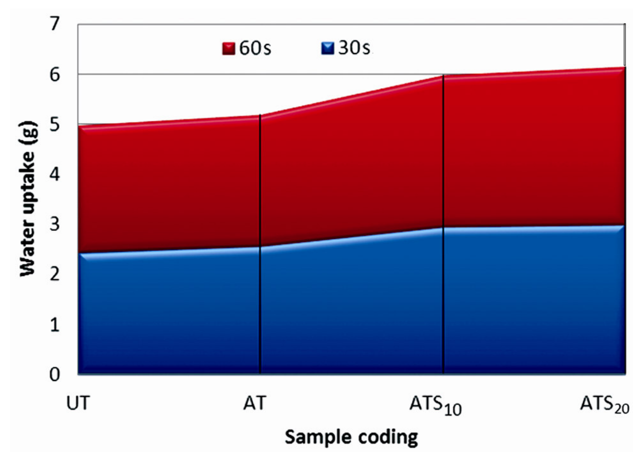

Fig. 5—In plane wicking values of untreated and sericin treated polyester fabric (UT–control, AT– alkali treated, ATS_{10}–alkali and 10g/L sericin, ATS_{20}–alkali and 20g/L sericin)

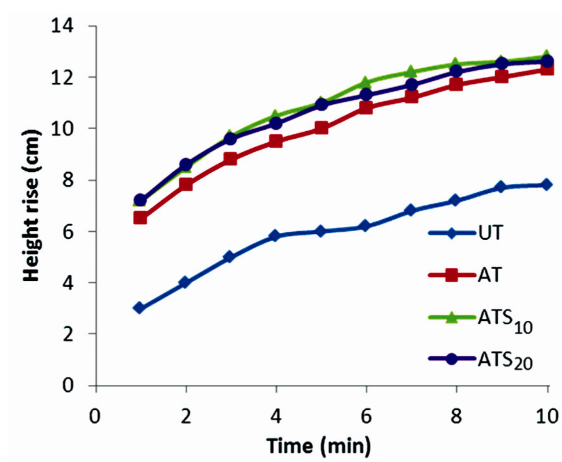

Fig. 6—Vertical wicking values of control and sericin treated polyester (UT–control, AT– alkali treated, ATS_{10}– alkali and 10gpl sericin, ATS_{20}– alkali and 20gpl sericin)

improves by almost 57% after treatment with alkali because of the generation of polar groups on the fabric surface. Application of sericin further enhances the vertical wicking values by 4% (Table 6). These high values of wicking are similar to those shown by specialized moisture management yarns like Coolmax (12.7cm height rise in 30 min) [24].

3.4.4 Performance Properties of Sericin Coated Fabrics

Antistatic Property

Polyester is a hydrophobic polymer with problem of static build up which causes problems during the processing of the fabric and also makes it uncomfortable for the wearer. Static behavior of untreated and treated PET was examined by qualitative analysis using the ash test (Fig. 7).

As can be seen that the amount of ash transferred to the untreated PET fabric (A) is much higher than that for the alkali modified PET fabric (B). On the other hand, almost nil amount of ash gets transferred onto sericin treated PET (C), indicating the hydrophilic nature of treated fabric surface. Easy dissipation of charge on the fibre surface occurs because of the enhanced conductivity of the hydrophilic coating. This indicates improvement in antistatic properties of PET fabric on sericin application.

Antimicrobial Activity

The results of agar diffusion test against the standard test organism *E. coli* (Gram negative) are shown in Fig. 8.

There is no zone of inhibition around the control fabric (A) thus, indicating an absence of antimicrobial activity. Similar results are seen in fabrics treated with sericin (B) against the test organisms. This is an interesting finding since sericin has been attributed with antimicrobial activity by several researchers[25, 26]. However, in our studies we find that it has no activity whatsoever. Antimicrobial activity of sericin is primarily due to the presence of a compound called

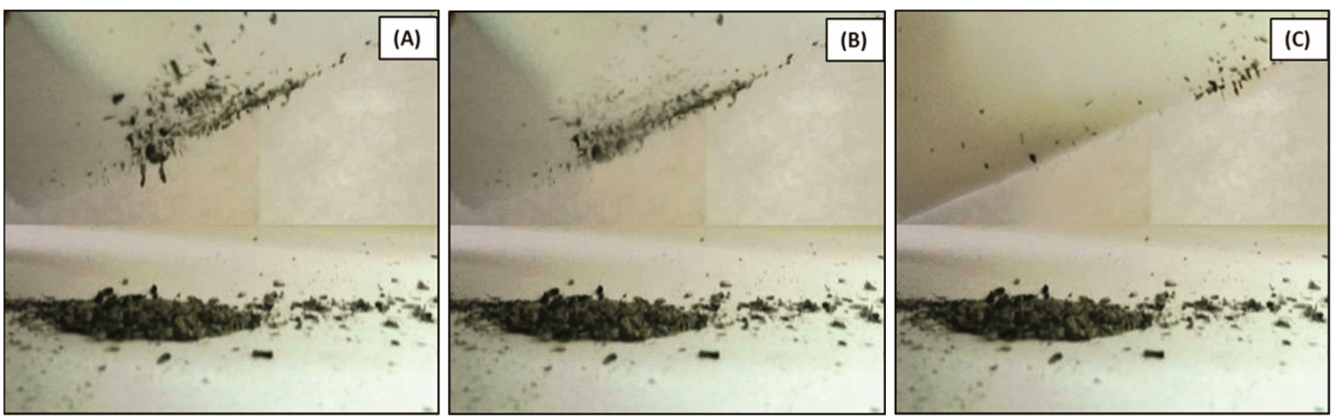

Fig. 7—Ash test for assessment of antistatic property (A) control, (B) alkali treated, and (C) alkali and 10g/L of sericin treated

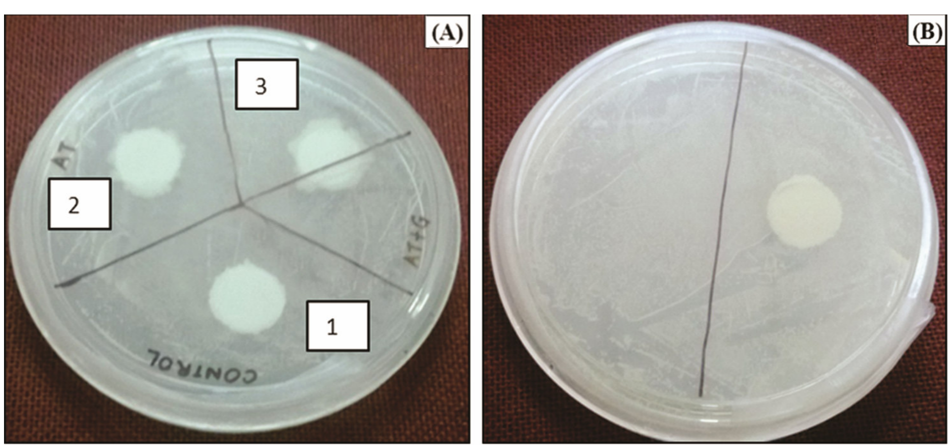

Fig. 8—Photograph showing zone of inhibition using agar diffusion method with *E.coli* (A) [1-control, 2-alkali treated, 3-alkali and GTA treated] , and (B) alkali and 10g/L sericin treated

seroin which is present in the cocoon. However, seroin gets degraded or destroyed during the process of degumming or protein extraction, leading to the loss of antimicrobial activity of sericin[27].

Free Radical Scavenging Activity (RSA)

Free radicals are natural by-products of our body metabolism. They are unstable, react readily and attack biological molecules such as lipids, proteins, enzymes, DNA and RNA, leading to cell or tissue injury associated with degenerative diseases[28]. Antioxidant agents, particularly those from natural sources are much in demand since they function as free-radical scavengers and chain breakers[29]. As a natural protein, silk sericin has functional groups like cysteine, tyrosine and histidine with flavonoids which contain electron donors. These donors react with free radicals and convert them to more stable products and terminate the radical chain reaction[30]. Radical scavenging activity of fabrics treated with sericin improves by 60% on sericin application. A marked increase in RSA from 12.44 in case of alkali treated samples to 30.12 for sericin treated samples is observed.

4 Conclusion

Results of this study show that durable sericin based finish can be applied on alkali modified PET using glutaraldehyde as crosslinking agent. Pre modification treatment with alkali affects the fibre significantly by creating voids on its surface. Application of sericin leads to improvement in comfort properties of PET. Fabric becomes smoother, hydrophilic and antistatic, and antioxidant property is found to improve. Enhancement in all these multifunctional properties makes sericin treated PET suitable for medical garments.

References

1 Vigneswaran C & Anbumani N, *J Text Inst,* 102 (2011) 140.
2 Kan C W & Yuen C W M, *Nucl Instrum Methods,* 266(2008) 127.
3 Zhang Y Q, *Biotechnol Adv*, 20 (2002) 91.
4 Irena G, Jolanta B & Karolina Z, *Appl Surf Sci,* 255 (2009) 8293.
5 Yamada H & Matsunaga A, *Japan Pat 06017373* (to Seiren Co.), 1994.
6 Lee S R, Miyazaki K, Hisada K & Hori T, *Sen'I Gakkaishi,* 60 (2004) 9.
7 Jin P Z, Igaeashi T & Hori T, *Sen'i Kogyo Kenkyu Kyokai Hokoku,* 3(1993) 44.
8 Gulrajani M L, Brahma K P, Kumar P S & Purwar R, *J Appl Polym Sci,* 109 (2008) 314.
9 Gimenez J C, *Alergol Inmunol Clin,* 15 (2000)279.
10 Ring J, Alomar A, Bieber T, Deleuran M, Fink-Wagner A, Gelmetti C, Gieler U, Lipozencic J, Luger T, Oranje A P, Schafer T, Schwennesen T, Seidenari S, Simon D, Stander S, Stingl G, Szalai S, Szepietowski J C, Taeb A, Werfel T, Wollenberg A & Darsow U, *J Eur Acad Dermatol Venereol,*26 (2012) 1045.
11 Gupta D, Agrawal A, Chaudhary H, Gupta C & Gulrajani M L, *J Clean Prod,* (2013) 1.
12 Wu J H, Wang Z & Xu S Y, doi:10.1016/j.foodchem.2006.10.042 (13.9.2013).
13 Dave J, Kumar R & Srivastava H C, *J Appl Polym Sci,* 33(1987) 455.
14 Natarajan S & Moses J J, *Indian J Fibre Text Res,* 37(2012) 287.
15 Zeronian S H, Wang H Z & Alger K W, *J Appl Polym Sci,* 41(1990) 527.
16 Zhang X M and Wyeth P, *Sci China*, 53 (2010) 626.
17 Boucau J, *Enzymatic and structural characterization linked to mycobacterium tuberculosis pathogenically of proteins*, Ph.D. thesis, The University of Toledo, 2008, 39.
18 Oh H, Kim M K & Lee K H, *Macromol Res,* 19(3) (2011) 266.
19 Kongdee A, Bechtold T & Teufel L, *J Appl Polym Sci,* 96(2005)1421.
20 Sehnal F, *Entomol Res,* 38 (2008)1.
21 Sothornvit R, Chollakup R, Suwanruji P & Songklanakarin J, *Sci Technol,* 32(2010)17.
22 Raja D, Kouhik C V, Ramakrishnan G, Babu V R & Subramanium V, *Indian J Fabric Text Res,* 37(2012) 381.
23 Sharabaty T, Biguenet F & Viallier P, *Indian J Fibre Text Res,* 33 (2008) 419.
24 www.advancedfibres.eu (accessed on 12.7.2013).
25 Mondal M, Trivedy K & Kumar S N, *Caspian J Env Sci,* 5(2)(2007) 63.
26 Rajendran R, Balakumar C, Sivakumar R, Amruta T & Devaki N, *J Text Inst*, (2011) 1.
27 Aramwit P, Kanokpanont S, Punyarit P & Srichana T, *Wounds,* 21(8) (2009)198.
28 Devasagayam T P A, Tilak J C, Boloor K K, Sane K S, Ghaskadbi S S & Lele R D, *J Assoc Physicians India,* 52(2004)794.
29 Sarovart S, Sudatis B, Meesilpa P, Grady B P & Magaraphan R, *Rev Adv Mater Sci*, 5 (2003)193.
30 Fan J B, Wu L P, Chen L S, Mao X Y & Ren F Z, *J Food Biochem,* 33(1) (2009) 74.