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# Fabrication of sustainable functional cotton fabric with silk sericin and chitosan for protective textiles

Md Ibrahim H. Mondal<sup>a,\*</sup>, Shimul Chandra Sarker<sup>a</sup>, Firoz Ahmed<sup>b</sup>, Md Nahid Pervez<sup>a,c</sup>, Joykrisna Saha<sup>a,d</sup>

<sup>a</sup> Department of Applied Chemistry and Chemical Engineering, Polymer and Textile Research Lab, University of Rajshahi, Rajshahi, 6205, Bangladesh

<sup>b</sup> BCSIR Laboratories Rajshahi, Bangladesh Council of Scientific and Industrial Research, Rajshahi, 6206, Bangladesh

<sup>c</sup> Department of Textile Engineering, Southeast University, Dhaka, 1208, Bangladesh

<sup>d</sup> Dept. of Textile Engineering Mawlana Bhashani Science and Technology University, Santosh, Tangail, 1902, Bangladesh

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# ABSTRACT

The use of bio-based sources for producing multifunctional cotton fabric with UV-protective and antibacterial properties is essential. Hence, the primary aim of this work was to develop sustainable functional cotton fabric (antibacterial and UV-protective) by applying silk sericin and chitosan using a simple and environmentally benign pad-dry curing method. The modification as well as functional properties of the treated fabric were evaluated in terms of antimicrobial efficacy, moisture management, UV protection, scavenging activity, surface morphology, thermal stability and mechanical strength. The results indicated that the concentration of chitosan around 10 mg/mL demonstrated remarkable antibacterial efficacy against gram-positive S. aureus bacteria. The quantitative analysis revealed an 87 %, reduction of bacteria, which surpassed the reduction rates in cotton fabric treated with sericin at concentrations of 10 mg/mL and 20 mg/ mL. The chitosan/sericin treated fabric showed antioxidant i.e. radical scavenging activity (RSA) of 47.44 % while the fabric treated with chitosan and sericin individually had RSAs of 42.35 % and 53.04 %. Compared to cloth treated with chitosan and sericin separately, the fabric treated with chitosan/sericin showed significantly better resistance to ultraviolet (UV) light (UFP 16.80). Based on the study, it is possible to create sustainable, multipurpose cotton fabrics with potential uses in protective textiles by combining silk sericin with chitosan.

# 1. Introduction

Textile products, particularly those of natural fibres, provide favourable environments for the proliferation of microorganisms owing to their extensive surface area and capacity to retain moisture. Numerous compounds have been employed to confer antibacterial properties to textile products. The substances encompassed in this category consist of amines, heterocyclic compounds featuring anionic groups, nitro compounds and their derivatives, inorganic salts, organometallic compounds, iodophors, phenols and thiophenols, onium salts, and nitro compounds [1]. Nevertheless, many of these substances pose a significant risk to human health and exhibit considerable resistance to decomposition within the natural environment. Accordingly, the textile industry seeks

\* Corresponding author.

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E-mail address: mihmondal@gmail.com (M.I.H. Mondal).

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environmentally-viable and human-body-friendly alternatives to deleterious textile chemicals.

Sericin, a naturally occurring protein derived from silkworm cocoons, exhibits unique attributes, such as biodegradability, nontoxicity, resistance to oxidation, antibacterial properties, UV radiation, and moisture absorption [2,3]. There are two distinct protein types that comprise natural silk: amorphous sericin and crystalline fibroin [4,5]. The protein sericin, comprising approximately 20–30 % of the cocoon's total weight, forms adhesive layers that sequentially envelop the fibroin fibre, facilitating the cocoon's formation [6]. Silk sericin, a water-soluble globular protein, is produced by *Bombyx mori* silkworms [7,8]. Sericin has a significant amount of serine (32 %), aspartic acid (16.8 %), and glycine (8.8 %), resulting in a high concentration of hydroxyl groups [9]. Sericin has considerable promise as a biological material. It may be combined with other polymers to prepare functional membranes, films, and fibres [3,10]. Besides, fibres coated with sericin can provide protection to the skin against the development of abrasive wounds and rash growth [11].

Chitosan, a polysaccharide found in nature, consists of N-acetyl-D-glucosamine (acetylated unit) and  $\beta$ -(1  $\rightarrow$  4)-linked D-glucosamine (deacetylated unit) in a random distribution. Chitin derived from the exoskeletons of shrimp undergoes a process known as alkaline deacetylation to transform it into chitosan [12–14]. Multiple research studies have provided evidence to substantiate the efficacy of chitosan as a finishing agent with antibacterial and antioxidant [15–18]. Although chitosan and sericin possess several beneficial characteristics, their concurrent application in the textile industry to enhance multifunctional capabilities, such as antibacterial, antioxidant, and UV-resistant properties, has not been documented thus far. Previous studies reported that a chitosan and sericin combination improved the structural configuration, such as porosity, rigidity, biocompatibility, and reactivity, more than individual application, which is necessary for the cotton fabric.

The present investigation involves the modification of cotton fabric using bioactive chitosan, sericin and chitosan/sericin to improve its functional properties, specifically antibacterial, antioxidant and UV protection. According to the authors, this is the first instance of such a treatment being reported in the literature. The physicochemical properties were thoroughly analyzed, and the functionalization was confirmed through various analytical techniques, including FTIR, SEM, and TGA. Subsequently, the antibacterial and UV protection capabilities of the treated fabric were evaluated and documented. This research suggests that textile manufacturers and consumers could benefit from the development of new biomaterials that are both environmentally friendly and safe for human health, offering an alternative to the toxic, petroleum-based finishing agents currently in use.

## 2. Experimental

# 2.1. Materials

Akij Textile Ltd., in Bangladesh, provides 100 % bleached cotton-poplin woven fabric. The fabric's specifications were  $40 \times 40/133 \times 72$  and 122 GSM (Grams per square metre). The waste silk cocoons of *Bombyx mori* were collected from the Bangladesh Sericulture Research Training Institute (BSRTI) in Rajshahi. Shrimp shell debris accumulated from the Khulna shrimp processing area and was used for chitosan manufacturing. Citric acid was utilised as a cross-linking agent. All other reagents and chemicals were used without additional purification.

#### 2.2. Methods

## 2.2.1. Silk sericin extraction

The silk sericin extraction process followed the procedure reported by Khalifa et al. and Shah et al. [5,19], with certain modifications. 50 g of silk cocoons were sliced into little pieces for this. They were then filled with deionised water and autoclaved at 120 °C for 40 min at a bath ratio of 1:15 (w/v). After autoclaving, the silk fibroin mixed solution was filtered via Whatman filter paper. After filtering, the sericin solution was given a shot of ethanol in a 3:1 ratio to precipitate it. The sericin was collected in a Buchner funnel and dried in an oven at 100 °C. The extraction yield of sericin was 13.33 %.

#### 2.2.2. Chitin and chitosan preparation

The shrimp shell powder was treated with 1 mol/L HCl at 100 °C, for 4 h, using a chitin to HCl solution, in the ratio 1:50, and then rinsed with distilled water. After being treated with 1 mol/L NaOH at 100 °C for 4 h, with a chitin to NaOH solution ratio of 1:50, the alkali-treated material was subsequently rinsed with distilled water [20–22]. The resultant solid, chitosan, was neutralised, rinsed, and then dried in a vacuum oven for 20 h at 50 °C.

## 2.2.3. Chitosan- and sericin-treated bleached cotton woven fabric

The Buşilă method [23], for applying chitosan and sericin to cotton woven fabric, was used, with minor modification. The necessary amounts of chitosan and sericin extract were applied to the cotton fabric using the pad-dry-cure procedure, with a padding machine, 2-bar pressure, and a material-to-liquor ratio of 1:15. Cotton fabrics were immersed in the prepared finishing solutions, which contained sericin, citric acid, chitosan, and the catalyst NaH<sub>2</sub>PO<sub>2</sub>, for 30 min at room temperature with a 1:15 liquor ratio. The pH of the solution was maintained at 4.5. The fabric was padded, dried at 80 °C for 3 min and, cured at 150 °C for 2 min, conditioned for 24 h at 25 ± 2 °C in 65 ± 5 % relative humidity.

#### 2.3. Characterisation of finished fabrics

#### 2.3.1. Moisture absorption study

The finished and washed cotton fabrics were stored in a humidity chamber at room temperature (70°C) for one week, and humidity was maintained at a saturation level [24,25]. After that, the moisture of the cotton fabric was analyzed using the following equation:

Moisture absorption, (%) = 
$$\frac{(Wf - Wi)}{Wi} \times 100$$

where Wf represents the wet sample's weight, and Wi represents the weight of the dry sample.

#### 2.3.2. Wicking property

The absorbency of the finished fabric, modified with chitosan and sericin, was evaluated using the wicking method. Through the capillary action of the liquid, wicking was accomplished by distributing the liquid vertically or horizontally through a medium [26].

#### 2.3.3. Water vapour permeability

This test was carried out by a water vapour transmission cabinet (Model: YG501D, Brand: Fanyan; Origin: China). According to the BS 7209 test [27], water vapour permeability was determined using the following formula:

Water vapour permeability = 
$$\frac{24 M}{At} \text{ gm}/\text{m}^2/\text{day}$$

where M is the mass loss in gm, A is the exposed test specimen's area in  $m^2$ , and t is the interval in hours between subsequent measurements of the assembly.

# 2.3.4. Air permeability

The amount of air that can move through a fabric's one square centimetre surface at a pressure of 1 cm of water in 1 s is known as the fabric's air permeability. Air permeability was assessed by using the airflow method [28]. Each group underwent testing on three samples, which were measured in cm<sup>3</sup>/cm<sup>2</sup>/s.  $4 \times 4$  cm<sup>2</sup> was the sample size. A digital air permeability tester has been used for this measurement (Model: YG461E, Brand: Fanyar; Origin: China).

## 2.3.5. Tensile test

The Grab test was used to assess the samples' tensile strength (ASTM D5034-95, 2001) test [29]. The test sample was split into 4 inch  $\times$  6 inch in the warp direction. The top and bottom jaws were used to clamp the samples. The machine started after the pre-tension had been optimised. Its cell load was 3000 N, and the machine works at a constant rate of extension (CRE) of 100 mm/min. After a certain time, it broke. Titan universal tensile strength tester (James Heal, USA) has been used.

#### 2.3.6. Soil degradation test

The Swain method was modified slightly to carry out the soil deterioration test. In four pots containing roughly 750 gm of soil and 250 gm of cow dung each treated and untreated samples were buried six inches beneath the soil. At predetermined times, 100 mL of sparkling water was poured into the pots. Weight loss after 30 days was used to gauge the integrity of the samples. The samples were removed from the soil, gently rinsed with pure water, and then dried in the sun [30]. The following equation was used to calculate the weight loss:

Weight loss (%) = 
$$\frac{W_1 - W_2}{W_1} \times 100$$

Where W<sub>1</sub> represents the initial weight and W<sub>2</sub> represents the weight after burial.

## 2.3.7. Fourier transform infrared spectrophotometer

The samples, including potassium bromide (KBr), were dried at a temperature of 105 °C for 10 h. The samples were mixed with potassium bromide (KBr) in a mortar and pestle to produce a powdered mixture with a mass ratio of 1:100 (1 mg sample to 100 mg KBr). The mixed samples were dried at a temperature of 105 °C for 10 h [3,31]. The material was then evaluated using a Fourier transform infrared (FTIR) spectrophotometer (Spectrum-100, PerkinElmer, USA) in the 400-4000 cm<sup>-1</sup> scanning range.

#### 2.3.8. Scanning electron microscopy (SEM) analysis

An electron microscopy machine was used to study the surface morphology of raw and finished cotton fabrics using scanning electron microscopy (Model-Phenom G2 pro, Netherlands). Samples were tested at a magnification of  $5000 \times$  and were scanned at 10.0 kV.

# 2.3.9. Thermal analysis

A simultaneous thermal analyser was used to conduct thermal analysis of chitosan, and sericin-treated samples (STA 8000,

PerkinElmer, USA). The temperature range for the analysis was 20  $^{\circ}$ C–600  $^{\circ}$ C, with a heating rate of 10  $^{\circ}$ C/min in a nitrogen atmosphere. The weight loss of the sample, as a function of temperature, was continually recorded.

## 2.3.10. Quantitative antibacterial test

The gram-positive bacteria *S. aureus* was used in tests to determine the antibacterial activity of the modified textile. The AATCC-100 standard [32] determined the bacterial population (total colony-forming units) in treated and untreated samples. A test sample of 2 inch by 2 inch was sliced, as required, and placed into five conical flasks of 50 mL each, containing 20 mL of nutrient broth and 20  $\mu$ l of microbial culture. For 24 h, 200 rpm and 37 °C were maintained, in a shaker incubator, with all of the flasks. The test culture that had been incubated in the nutrient broth was diluted five times with sterile, distilled water. 20  $\mu$ L of each dilution was applied in a Petri dish with nutrient agar,. All of the infected plates (both treated and untreated samples) underwent a 24 h incubation period at 37 °C. The number of surviving cells was determined after 24 h. The following equation was used to determine the percentage reduction:

Bacterial colony reduction (%) = 
$$\frac{(B-A)}{B} \times 100$$

where A is the number of surviving cells (CFU/mL) in the flasks containing the treated samples and B is the number of surviving cells (CFU/mL) in the flasks containing the untreated samples.

# 2.3.11. Qualitative antibacterial test

According to the AATCC-147 Standard [8,33], the inhibition zones of treated and untreated samples were identified. The following equation was used to determine the zone of inhibition:

$$W = \frac{T-D}{2}$$

Where W is the clear zone of inhibition's width in mm, T is the test specimen's overall diameter and the clear zone of inhibition's total diameter in mm, and D is the test specimen's diameter in mm.

#### 2.3.12. Wash durability test

The treated fabric was washed using an industrial machine, and the tested sample's antibacterial activity was assessed using the AATCC-124 and AATCC 100 test standards, respectively [32,34].

#### 2.3.13. Antioxidant activity of treated fabric

The free radical scavenging activity (RSA) of chitosan- and sericin-treated cotton textiles was measured to determine their antioxidant properties. Wu et al. (2007) detected free radical scavenging activity of such materials even lightly-treated [2]. Here, three tests were conducted on each sample, and it was found that, on average, from 3.5 mL of freshly-treated material, 0.004 percent DPPH radical was found in the methanol. The reaction mixtures were centrifuged at 6000 rpm for 5 min after 25 min of reacting, at 25 °C room temperature, in the dark. The decolorised solution was compared to a blank sample, with 1 inch  $\times$  1 inch chitosan or sericin treated cloth and pure methanol instead of DPPH at 517 nm.The following equation was used to assess radical scavenging activity:

Radical scavenging activity (RSA) = 
$$\left(1 - \frac{\text{Sample absorbance}}{\text{Control absorbance}}\right) \times 100$$

#### 2.3.14. Measurement of UV protection factor

The ultraviolet protection factor (UPF) was calculated using a UV spectrophotometer (Shimadzu 1650, Japan). The following equation was used to determine the UPF in accordance with the AATCC test method [35,36]:

Table 1
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Fabric type	Moisture absorption (%)	Absorbency height (cm)	Water vapour permeability (g/m <sup>2</sup> / day)	Air permeability (cc/cm/sec)	Tensile Strength (N)	Biodegradation (% wt loss at 30th day)
Control	$15.8\pm0.20$	$4.6\pm0.15$	$1352.2\pm0.91$	$131.8\pm0.60$	$\begin{array}{c} 1134.0 \pm \\ 1.62 \end{array}$	$\textbf{74.50} \pm \textbf{0.77}$
4 mg/mL Chitosan Finished	$23.4\pm0.2$	$5.1\pm0.2$	$862.33\pm0.85$	$122.4\pm0.65$	$\begin{array}{c} 812.53 \pm \\ 1.46 \end{array}$	$34.71 \pm 0.96$
4 mg/mL Sericin Finished	$\textbf{35.83} \pm \textbf{0.4}$	$\textbf{5.7} \pm \textbf{0.25}$	$885.83 \pm 0.85$	$126.5\pm0.81$	$\begin{array}{c} 834.03 \pm \\ 1.65 \end{array}$	$55.02 \pm 0.52$
<sup>a</sup> Combined Finished	$\textbf{28.73} \pm \textbf{0.45}$	$\textbf{5.6} \pm \textbf{0.12}$	$873.3\pm0.90$	$124.13\pm0.83$	$\begin{array}{c} \textbf{822.83} \pm \\ \textbf{1.74} \end{array}$	$\textbf{44.41} \pm \textbf{0.80}$

<sup>a</sup> Combined means 2 mg/mL chitosan and 2 mg/mL sericin.

$$\text{UPF} = \frac{\sum_{280}^{400} E_{\lambda}.S_{\lambda}.\Delta_{\lambda}}{\sum_{280}^{400} E_{\lambda}.S_{\lambda}.\Delta_{\lambda}.T_{\lambda}}$$

where  $\lambda$  is the wavelength in nanometers,  $T_{\lambda}$  is the fabric's spectral transmission, and  $\Delta_{\lambda}$  is the measured wavelength intervals in nanometers.  $E_{\lambda}$  is the erythemal spectral effectiveness.

## 3. Results and discussion

# 3.1. Physico-chemical characteristics

Table 1 displays the moisture absorption, absorbency height of washed and finished cotton fabrics, water vapour permeability, air permeability, tensile strength, and biodegradation results of the cotton fabrics that were washed and finished.

Sericin has a good attachment capacity on cotton fabric, and sericin-treated cotton fabric shows higher moisture absorption. This is because, sericin on cotton fabric provides numerous hydrophilic hydroxyl (-OH) and primary amine (-NH<sub>2</sub>) groups than that of chitosan. The results of the wicking test showed a combined-treated fabric's absorbency of  $5.6 \pm 0.12$  cm, which was higher than that of chitosan-treated fabric but lower than that of sericin-treated fabric.

Additionally, Table 1 demonstrates that the chitosan/sericin treated fabric showed higher tensile strength ( $822.83 \pm 1.74$  N) than a fabric treated with chitosan but a lower tensile strength than a fabric treated with sericin. The tensile strength of the sericin-treated cloth was the highest of the three, but it was less than that of the untreated fabric ( $1134 \pm 1.62$  N). Hence, treatment decreases the fabric's tensile strength by 27–30 %.

The untreated sample experienced the greatest biodegradation. Chitosan-treated fabric experienced the least biodegradation, shown as  $34.71 \pm 0.96$  % weight loss. This ability to resist attack by soil bacteria, reducing biodegradation, also suggests potent antibacterial properties. The combined treated sample's soil deterioration, with a weight loss of  $44.41 \pm 0.80$  %, was a little better than no treatment at all. For samples that had only been treated with sericin, weight loss was  $55.02 \pm 0.52$  %. Chitosan showed the most potent antibacterial activity. The findings of the soil degradation test showed that, despite the treated samples' modest resistance to

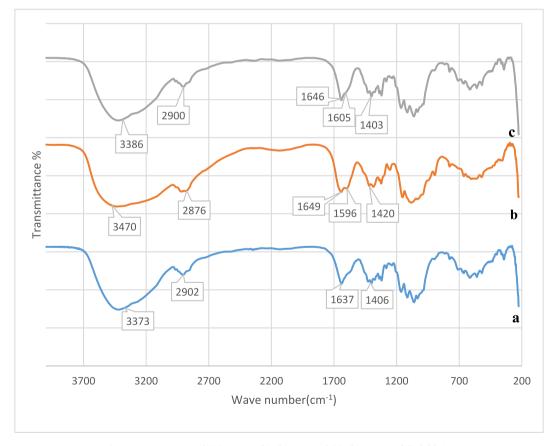


Fig. 1. FTIR Spectra of: (a) cotton, (b) chitosan and (c) chitosan modified fabric.

biodegradation in soil, they were still biodegradable and, therefore, environmentally-friendly.

The rate of airflow through the cloth is measured by its air permeability. The finishing process has an impact on a fabric's capacity to "breathe". The fabric's thickness was increased by applying antibacterial chemicals as a finishing touch. In turn, this resulted in a small reduction in air permeability [37]. According to the test results shown in Table 1, fabrics treated with chitosan and sericin had less air permeability than untreated materials. The air permeability of the fabrics decreased as a result of chitosan and sericin treatments that filled the pores of the materials. In comparison to sericin-treated fabric, the chitosan-treated fabric had a thicker coating layer. As a result, chitosan-treated fabric has a lower air permeability than sericin-treated material. Discomfort develops when the air permeability diminishes.

The capacity of water vapour to permeate clothing is known as water vapour permeability. If water vapour cannot pass through, humidity will build up between the skin and the fabric. Heat builds up in the body when moisture is trapped on the skin. Discomfort results from rising temperature rise and humidity. Therefore, less fabric mass-per-square-metre and thickness, allowing for the simple flow of water vapour through the materials, are preferred.

As shown in Table 1, the combinational treatment of chitosan and sericin generated a fabric with a higher water vapour permeability (873.3  $\pm$  0.90 g/m<sup>2</sup>/day) than one treated with chitosan but less than one treated with sericin. The sericin-treated fabric had the best results for water vapour permeability among the three but was lower than the untreated fabric (1352.2  $\pm$  0.91 g/m<sup>2</sup>/day).

From the data table of physico-chemical properties of modified and unmodified fabrics, we see that chitosan/sericin treated fabrics showed the moderate results whereas chitosan treated showed lower and sericin modified showed the higher. This is due to the fibre reactivity of chitosan and sericin. The fibre reactivity of sericin is higher than that of chitosan. That's why when cotton fabrics treated with chitosan/sericin combination the physico-chemical properties lies between chitosan treated and sericin treated fabrics. Thus, according to physico-chemical properties, fabrics treated with sericin are more pleasant to wear in hot conditions. Chitosan-treated fabric feels warmer to wear and will be suitable for slightly cold weather. Whereas, chitosan/sericin treated fabrics can be used in both weather conditions in terms of comfortness.

# 3.2. FTIR analysis

The FTIR *spectra* of scoured bleached cotton show a prominent peak at 3373 cm<sup>-1</sup>. This peak is associated with the stretching vibration of hydroxyl (OH) groups in cellulose within the range of  $3100-3600 \text{ cm}^{-1}$ . This observation is depicted in Fig. 1a. The cotton fabric that had undergone bleaching and scouring exhibited distinctive peaks at 1406 cm<sup>-1</sup> and 2902 cm<sup>-1</sup>, which were identified as

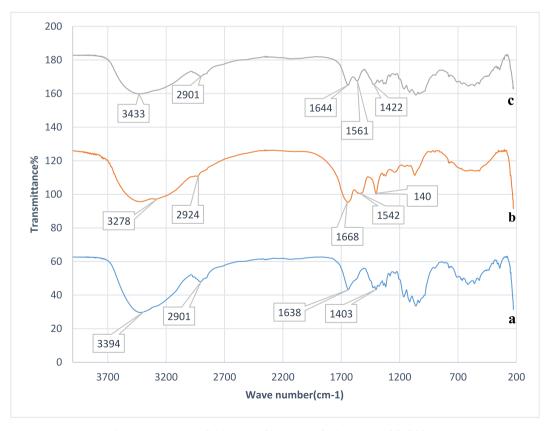


Fig. 2. FTIR Spectra of: (a) cotton, (b) sericin and (c) sericin modified fabric.

the C-H bending and stretching bands, respectively. Moreover, the signal at 1637 cm<sup>-1</sup> is attributed to the H-O-H stretching vibration of water molecules absorbed inside carbohydrates [36,38].

The prominent peak seen at 3470 cm<sup>-1</sup> in Fig. 1b corresponds to the stretching vibration of O-H and N-H groups in chitosan. The characteristic peaks seen at wavenumbers 1596 cm<sup>-1</sup> and 1649 cm<sup>-1</sup> in the chitosan spectrum may be attributed to the bending vibration of N-H groups and the stretching vibration of C=O groups in acetyl groups, as reported in the literature [3,39]. After undergoing chitosan treatment, the sample exhibited intermolecular hydrogen bonding between the cotton and chitosan, as shown by the presence of a peak at a wavelength of 3386 cm<sup>-1</sup>. In addition, it was observed that the sample treated with chitosan exhibited peaks at wavenumbers of 1646 cm<sup>-1</sup> and 1605 cm<sup>-1</sup>. The peaks shown in Fig. 1c indicate the successful integration of chitosan into the fabric.

The N-H stretching vibration of sericin exhibits peaks within the range of about  $3500-3000 \text{ cm}^{-1}$ , with a specific peak observed at  $3278 \text{ cm}^{-1}$ , as seen in Fig. 2b. The spectral analysis revealed the presence of the stretching vibration of the C=O bond, which was seen at a wavenumber of  $1668 \text{ cm}^{-1}$ . The analysis of the weaving properties seen at a wavenumber of  $1542 \text{ cm}^{-1}$  confirms the existence of N-H bending functional groups in sericin. The peak at  $1405 \text{ cm}^{-1}$  corresponds to CH<sub>3</sub> bending and CH<sup>2</sup> deformation [8,40–42]. The presence of a new peak at  $1561 \text{ cm}^{-1}$  was seen in the treated cotton fabric (Fig. 2c), which was absent in the untreated cotton fabric (Fig. 2a) due to the lack of sericin treatment. Furthermore, the cloth that had sericin treatment exhibited altered peaks at  $1644 \text{ cm}^{-1}$  and  $1422 \text{ cm}^{-1}$ . The peaks shown in Fig. 2c indicate the effective integration of sericin into the cotton fibre.

## 3.3. Surface morphology

The scanning electron microscope was used to examine the surfaces of both the treated and untreated cotton fabric. The untreated fabric exhibits comparatively smooth surface, as shown in Fig. 3a. In contrast, the treated cotton fabric display rough and uneven surfaces, as illustrated in Fig. 3b, c, 3d, as compared to the untreated fabric. The presence of chitosan or sericin can be seen in Fig. 3b, c, 3d. These granuler materials contribute to the uneven surface of the treated fabric and serve as proof of the effective attachment of the finishing agent to the previously untreated fabric surface. The uneven surface provided the higher surface area as well as higher functionalization towards the physico-chemical, antimicrobial, UV protection, anti-oxidation, wash durability, etc. properties of the treated fabrics.

## 3.4. Thermal analysis

The thermal behaviour of control and finished cotton fabrics is depicted in Fig. 4. The TGA of the control sample can be justified through the literature by means of similar behaviour [43]. Based on the analysis of TGA thermograms in Fig. 4, both modified and unmodified or control cotton fabrics undergo a process of thermal degradation in two distinct phases. The first phase, of weight reduction, in the temperature range of 90–120 °C, is attributed to physiological dehydration. The second phase, of significant and rapid reduction in weight, in the temperature range of 220–380 °C, is attributed to the thermal degradation of the glycoside bonds. After the second phase degradation the residual char was produced which achieved a fixed weight. The thermal stability of control and finished cotton textiles may be categorised based on their initial decomposition temperature (T<sub>i</sub>), following a specific sequence. The initial decomposition temperature (T<sub>i</sub>) of control, chitosan modified, sericin modified and chitosan-sericin modified cotton fabrics were 240 °C, 280 °C, 260 °C and 310 °C respectively. Here, it has been seen that thermal stability increased due to modification because of the more thermally stable chitosan and sericin coatings. Among these chitosan-sericin composite finished fabric showed better results

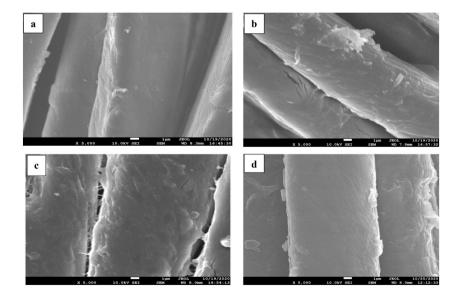


Fig. 3. SEM of: (a) Control, (b) Chitosan finished, (c) Sericin finished, and (d) Chitosan-sericin finished cotton fabrics.

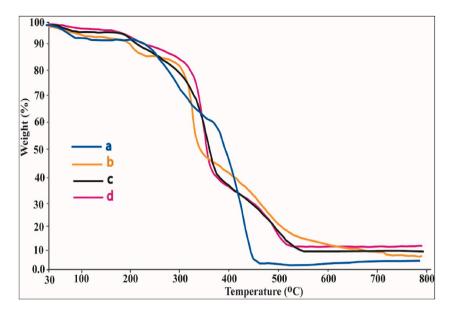


Fig. 4. TGA of: (a) control, (b) chitosan finished, (c) sericin finished, and (d) chitosan-sericin finished cotton fabrics.

in terms of  $T_i$  due to the synergistic effect of the usage of chitosan and sericin together. At the final stage of thermal degradation, it has been seen that control or unmodified cotton fabric showed the least amount of residual char whereas modified fabrics showed approximately 10 % char. Because, chitosan and sericin remained organic salt form i.e. contained minerals as both were derived after several successive chemical treatments. From the thermal analysis data it can be concluded that cotton textiles were treated with a combination of chitosan and sericin finishes to achieve control over their thermal properties.

## 3.5. Anti-bacterial activity measurement

Fig. 5b depicts a solution containing 10 mg/mL of sericin extract, while Fig. 5c depicts a solution containing 5 mg/mL of chitosan. The untreated sample, shown in Fig. 5a, did not exhibit any zone of inhibition. A distinct zone of inhibition was visible in Fig. 5b and (c). The chitosan solution created a better zone of inhibition than the sericin-extract solution.

Table 2 demonstrates an inverse relationship between the concentration of chitosan and sericin and the treated fabric's antibacterial effectiveness, meaning the inhibition zone's size and the percentage reduction of bacteria on the fabric. The chitosantreated fabric had bacterial reduction rates of 5 mg/mL and 10 mg/mL against *S. aureus*, which were 65 % and 87 %, respectively. Bacterial reduction rates for cloth treated with sericin at 5 mg/mL and 10 mg/mL were 51 % and 68 %, respectively. The prepared biocomposite (5 mg/mL chitosan and 5 mg/mL sericin) treated fabric had a 78 % reduction rate against germs. Chitosan, however, at a concentration of 10 mg/mL, had a higher rate of bacterial reduction than sericin-treated and combined-treated fabrics.

A longer-term study assessed the effects of repeated washing on the anti-bacterial properties of treated and untreated fabrics. The findings clearly show that subsequent washing has a negative impact on bacterial reduction. After 10 washes, the bacterial reduction rate for *S. aureus* declined from 87 % to 63 % for 10 mg/mL chitosan-treated cloth and from 68 % to 42 % for 10 mg/mL sericin-treated fabric. All of the treated cloth samples were shown to preserve 60 %–70 % antimicrobial activity after 10 washing cycles.

As seen in Table 2, the circular inhibitory zone grows with rising sericin and chitosan concentrations. An inhibitory zone of 2.5 mm

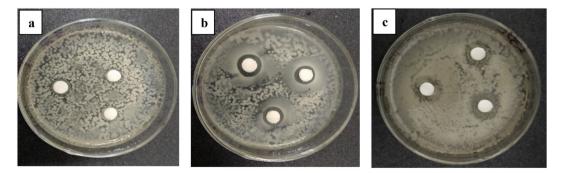


Fig. 5. Antimicrobial activity of: (a) control, (b) sericin extract and (c) chitosan solution against S. aureus gram-positive bacteria.

#### Table 2

The antibacterial activity of bleached cotton treated with chitosan and sericin against S. aureus.

Test Fibres	Number of colonies	Bacterial reduction (%)	Bacterial reduction (%) after five washes	Bacterial reduction (%) after ten washes	Inhibition zone in mm
Untreated	816	00	00	00	00
5 mg/mL chitosan treated	285	65	51	42	1.9
10 mg/mL chitosan treated	106	87	75	63	2.8
5 mg/mL sericin treated	399	51	40	31	1.6
10 mg/mL sericin treated	261	68	54	42	2.2
<sup>a</sup> Combined sericin- chitosan treated	179	78	68	56	2.5

<sup>a</sup> Combined sericin-chitosan treated means 5 mg/mL chitosan and 5 mg/mL sericin.

against *S. aureus* bacteria is visible on combined-treated fabric. On the other hand, at a concentration of 10 mg/mL, fabrics treated with sericin and chitosan had inhibitory zones of 2.8 mm and 2.2 mm, respectively. The interaction of the positively-charged chitosan with the negatively-charged residues at the cell surface of many fungi and bacteria results in substantial cell surface alteration and changes in cell permeability. These are the bases of chitosan's better antibacterial results. The weakened cell walls allow the emptying out of the bacterial cells' electrolytes, UV-absorbing chemicals, proteins, amino acids, glucose, and lactate dehydrogenase. Chitosan subsequently prevents bacteria from metabolising food intake normally, ultimately resulting in these cells' death from inadequate nutrition [44].

Table 2 and Fig. 6a–d shows the zone of inhibition due to the antimicrobial effects of untreated, chitosan-treated, combined-treated, and sericin samples on cotton fabric. Again, chitosan demonstrates, in every test, that it is superior to sericin in anti-bacterial effects (Fig. 6b). However, chitosan and sericin together functioned better than sericin alone in terms of the size of the inhibition zone and reduction of bacterial numbers ((Fig. 6c).

# 3.6. Antioxidant activity of treated fabric

For all samples, the antioxidant activity increases as the concentration of the treatment agent rises. Table 3 shows that 10 mg/mL sericin-finished cotton fabric had the highest radical-scavenging percentage when compared to chitosan- and chitosan-sericin finished cotton fabrics. Because it's residual free  $-NH_2$  groups can react with free radicals (DPPH Solution) to generate stable molecules and  $-NH_2$  groups can form ammonium groups ( $NH_4^+$ ) by collecting hydronium ions from the solution, silk sericin works as an antioxidative agent. Other authors in the scholarly literature similarly attest to sericin's antioxidant properties, as they found that sericin totally inhibited lipid peroxidation [7,45,46].

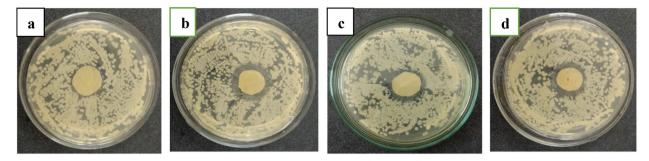
As a result, sericin-finished cotton fabric has better radical scavenging activity (RSA) than other finished fabrics. Radical scavenging activity (RSA) of finished cotton fabrics may be rated as sericin-finished, chitosan-sericin-finished, and chitosan-finished cotton fabrics.

#### 3.7. The UV protection factor of treated fabric

Table 4 shows that sericin-finished cloth has the best UV protection factor. The UV protection factor of chitosan-sericin finished cloth is ranked first as the chitosan-sericin composite showed the synergistic effect regarding this case. The UPF rating is inversely proportional to the transmittance value of the specimens [36]. As a result, the transmittance value of the chitosan-sericin treated cloth was higher than that of other fabrics. N-acetylglucosamine and glucosamine are two chromophoric groups found in chitosan [15]. These are made from chitin that has been partially deacetylated. UV protection is aided by these chromophoric groups. Sericin, on the other hand, has peptide linkages between amino acids that help with UV protection [47].

#### 4. Conclusions

The fabrication of sustainable functional cotton fabric using silk sericin and chitosan has shown promising results, underscoring the potential of these natural biopolymers in advancing protective textiles. Through the application of silk sericin and chitosan, the cotton fabric exhibited enhanced physico-chemical properties like enhanced moisture management, wicking height, water vapour and air permeability, all while mechanical strength decreased. The results obtained from this research can be valuable in developing protective textiles that possess properties such as resistance to bacterial growth, protection against ultraviolet radiation, high levels of radical scavenging, higher thermal stability and environmentally friendly characteristics. Nevertheless, the application of sericin or chitosan treatments did not result in any noticeable effects on the comfort properties of the cotton fabric. The results of soil degradation indicate that the treated samples are ecologically benign and capable of biodegradation. In conclusion, this study highlights the effectiveness of silk sericin and chitosan in developing functional and sustainable textile solutions. The approach not only aligns with global sustainability goals but also opens new possibilities for developing advanced protective textiles.



**Fig. 6.** Images of the zone of inhibition for treated and untreated samples that were tested using a qualitative methodology: (a) untreated, (b) chitosan treated, (c) combined treated, and (d) sericin treated).

# Table 3 Test results of radical scavenging activity of treated fabric.

Concentration	RSA % of sericin- treated fabric	RSA % of chitosan- treated fabric	RSA % of chitosan-sericin treated fabric
2 mg/mL	$29.36\pm0.63$	$25.04\pm0.85$	$27.10\pm0.60$
4 mg/mL	$35.02\pm0.83$	$29.24 \pm 0.59$	$32.43 \pm 0.58$
6 mg/mL	$42.33\pm0.51$	$33.42\pm0.51$	$38.44 \pm 0.60$
8 mg/mL	$48.12\pm0.44$	$38.14\pm0.31$	$42.13\pm0.78$
10 mg/mL	$53.04 \pm 0.23$	$42.35\pm0.40$	$47.44\pm0.50$

#### Table 4

Test results of the UV protection factor value of treated fabric.

Test Fabric	UV protection factor (UPF) rating
Untreated fabric	$3.52\pm0.5$
5 mg/mL sericin finished	$5.3\pm0.4$
10 mg/mL sericin finished	$6.81\pm0.7$
5 mg/mL chitosan finished	$6.08\pm0.5$
10 mg/mL chitosan finished	$8.33\pm0.8$
5 mg/mL chitosan-sericin finished	$12.40\pm1.0$
10 mg/mL chitosan-sericin finished	$16.80\pm0.9$

# CRediT authorship contribution statement

**Md Ibrahim H. Mondal:** Writing – review & editing, Supervision, Resources, Funding acquisition, Formal analysis, Conceptualization. **Shimul Chandra Sarker:** Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Formal analysis. **Firoz Ahmed:** Writing – review & editing, Visualization, Investigation, Formal analysis. **Md Nahid Pervez:** Writing – review & editing, Visualization, Methodology, Investigation, Formal analysis. **Joykrisna Saha:** Writing – review & editing, Visualization, Methodology.

# Data availability

All relevant data are within the manuscript.

# Additional information

Correspondence and requests for materials should be addressed to MIHM.

#### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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