

Synthesis and Application of Sericin/ β -Cyclodextrin Material for Skincare Textile

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Abstract: In this study, we report the synthesis, characterization and application of Sericin/ β -Cyclodextrin material for skin care finish on cotton fabric. The Sericin/ β -Cyclodextrin material was successfully synthesized by using citric acid. Sericin was characterized by SDS-PAGE for molecular weight analysis and calculated A-ratio for the purity of sericin by using UV spectra analysis. The Sericin/ β -Cyclodextrin material is characterized by Scanning electron microscopy (SEM) and Dynamic light scattering (DLS) confirmed that the synthesized material was spherical and flat in shapes varied from micro to nano range. Fourier Transform Infrared (FTIR) Spectroscopy spectra and ferric hydroxamate test confirmed the successful synthesized the material. Thermogravimetric analysis (TGA) confirmed that the stability of material at a higher temperature. SEM image and Fourier Transform Infrared (FTIR) Spectroscopy spectra were confirmed to presence of material over cotton fabrics. α -tocopherol loaded material treated cotton samples were displayed very good scavenging activity ranges 62-70% against DPPH free radical after 20 washes. Treated cotton samples were displayed good to very good UPF value varies from 18 to 26. Treated fabrics were shown good mechanical, Moisture transport and optical properties. The synthesized finishing material has a lot of potentials to use for skincare textile applications.

Keywords: Biopolymer, Sericin, β -Cyclodextrin, Citric acid, Scavenging activity, Skincare.

I. INTRODUCTION

As on date, petroleum is a vital resource for a lot of synthetic materials. The supply of petroleum being limited, and as per higher demand, there is requirement to find an alternative resource to produce various synthetic materials. Petroleum-based synthetic materials are also generating severe environmental issues due to its lack of biodegradability, sustainability, and eco-friendly nature and their hazardous nature impact on life over the water, air, and soil [1, 2]. These problems can be resolved by using bio-based materials that can quickly be adopted by our environment. Biopolymers are bio-based materials that are derived from renewable resources [3]. Biopolymers are linearly or branched chain-like structured molecules that have a monomer unit of either saccharide derived from sugars, amino acid proteins, or nucleic acid of nucleotides [4]. Biopolymers have a hierarchical structure that contains various units of monomer. The secondary structure is formed by the local molecular arrangement of monomeric units, which obtain a three-dimensional framework to configure a tertiary structure.

Biopolymers have now become an exciting area of research to develop new and emerging products. They can produce from renewable resources such as microorganisms, biomass, agro, and fossil [5, 6]. They are generally polymeric chain like structural molecules, which has made by repeating unit [7]. Biopolymers can be produced a renewable resource to make them biodegradable, non-toxic, biocompatible, and sustainable [8]. They have several specific properties, such as biological, physical, chemical, degradation, and biomechanical properties which, attract researchers [9, 10]. These unique properties of biopolymers are created attention to utilize them in various industries and applications, such as textiles, food production, geotechnical engineering, erosion control, drug delivery, medical, paper, agriculture and cosmetic industry [11-14]. Individual biopolymers have limited properties and applications which can be enhanced by using their composites or by grafting them to other biomaterials. These materials have displayed their improved performance due to combining effect and broader their application area. In the current scenario, the industry has a higher demand and requirement of biodegradable products. So, biopolymers are combined with other bio-based materials to make new products and finishes.

Silk is a protein-based biopolymer produced by *Bombyx mori* silkworm [15]. Silk is composed of two proteins, i.e., fibroin and sericin. The silk fibre has fibrous core protein fibroin, and this core protein is coated with glue-like protein sericin [16]. Sericin is an amorphous, globular, and hydrophilic polymer, which consists of 18 different amino acids [17]. These amino acids have made by a bulkier strong polar group, such as carboxyl, hydroxyl, and amino groups [17]. The

silk industry produces sericin as a by-product and produced after the process of silk degumming [18]. Sericin has various unique properties, such as good antioxidant, UV protective and moisturizing properties, which have resulted in lot of researchers to use them in numerous applications [19, 20]. Sericin has utilities in various industries and products, such as textiles, food dietary, cosmetics, drug delivery, and biomedical applications [20-23].

The cyclodextrins are a cyclic oligosaccharide consist of glucose units that are linked together by α -1,4-glycosidic bonds [24]. Starch or sugar is the raw material of cyclodextrins that produced by enzymatic degradation process. Cyclodextrins can be categorized into three forms, i.e., α , β , and γ -cyclodextrin, which consist of 6, 7, and 8 α -1,4-glycosidic bonds [24]. Cyclodextrins have unique properties, such as the ability to form an inclusion complex with hydrophobic materials, water-soluble, and biodegradable [25]. Cyclodextrin is used for several applications, such as textiles, food, agriculture, cosmetic, pharmaceutical, and chemical industries [26-30]. Cyclodextrins also have several areas of textiles applications such as pretreatment, dyeing, functional finishes to entrapping of hydrophobic materials [31, 32]. Cyclodextrin grafted cotton fabrics were modified their function properties and utilized for fragrance release, UV protection, odor absorption, antimicrobial [33, 34]. Dehabadi et al. were used cyclodextrin derivatives such as monochlorotriazinyl- β -CD on cotton that directly reacted to the hydroxyl groups [35]. Cyclodextrins was also grafted with biopolymers for different textile applications. β -Cyclodextrin Grafted Chitosan were synthesized and applied on cotton fabric with active material like lavender oil, Doxorubicin hydrochloride to enhance antimicrobial and fragrance activity [36, 37]. For improvement of fragrance property, β -Cyclodextrin was combined with Butyl Acrylate, maleic anhydride, β -Cyclodextrin, 3-Glycidoxypropyltrimethoxysilane (GPTMS) and Tetraethoxy orthosilicate (TEOS) [38-40]. Chitosan/Poly Nisopropyl acrylamide composite material was synthesized for smart and fragrance applications [41].

The silk industry produces sericin as a by-product in a considerable amount and discharge into wastewater. Sericin has excellent moisturizing, antioxidant, and UV protective properties. These properties of sericin can utilize to enhance the functional properties of textiles. The sericin has a lot of potentials to use as textile finishes. The aim of this research paper is to the synthesis and application of sericin/ β -cyclodextrin material for different textile applications, such as fragrance, antimicrobial, skincare textiles, etc. The sericin/ β -cyclodextrin material has ability to formed inclusion complex with fragrance, antimicrobial, skincare hydrophobic substances, such as Vitamin E, essential oils, thyme oil, etc. The treated textile material can give antioxidant, moisturizing effects to human skin by transfer of active substances through treated textiles. There is no research available of sericin based grafted compound which used for skincare finish of textiles.

II. MATERIALS AND METHODS

2.1. Materials

Sericin was collected from Central Silk Technological Research Institute, Bangalore. β -cyclodextrin (98%) was purchased from TCI, Mumbai. Citric acid and Sodium hypophosphate (98%) were procured from Central drug honor Ltd, New Delhi.

2.2. Methods

The Sericin/ β -cyclodextrin material was synthesized by using above chemical.

2.2.1. Silk sericin solution preparation method

Silk sericin solutions were prepared from sericin powder (average molecular weight 175 KDa). 2 gms of sericin powder were added in 50 ml of di-water and heated at 90°C for 30 min with proper stirring.

2.2.2. Synthesis of sericin/ β -cyclodextrin material

Prepared sericin aqueous solution was used for the synthesis of sericin/ β -cyclodextrin material. The different weight ratios of sericin and β -cyclodextrin were dissolved in 30 ml of di-water and slowly added for prepared sericin solution at 85°C and stir at 300 rpm for 30 min. Citric acid was dissolved in 20 ml of di-water with a total β -cyclodextrin concentration of 1 mol/L. The Citric acid and Sodium hypophosphate solution was added drop wise at 130°C and stirred at 300 rpm for 2 hrs. The slurry was cooled down at room temperature and stored.

2.3. Characterization methods of sericin/ β -cyclodextrin material

2.3.1. SDS-PAGE Analysis

The distribution of the molecular weight of sericin was analyzed by SDS-PAGE analysis. The SDS-PAGE buffer sample was prepared and mixed with the sericin sample, according to Gamo et al. [42] the mixer was boiled for 5 min. The stacking and resolving gel amounts were 5% and 12%, respectively. The electrophoresis was performed by a Power pro electrophoresis system (Cleaver Scientific, U.K.) for 4 hrs at a constant voltage of 90V. After electrophoresis, Coomassie Brilliant Blue R-250 was used for staining the gel. Staining was carried out for 30 min, and then the de-staining process

was executed. The distribution of Molecular weights was analyzed by a molecular weight marker (MWM) purchased from Himedia Laboratories Pvt. Ltd., Bangalore.

2.3.2. UV spectroscopy analysis

The purity of sericin samples was determined by UV-vis spectrophotometer (Shimadzu - UV-2450) to measure absorption at wavelength 260 nm and 280 nm with the help of A-ratio. The standard ratio is 1.8 for pure sericin material.

$$A - \text{ratio} = \frac{\text{Absorption at } 280 \text{ nm}}{\text{Absorption at } 260 \text{ nm}}$$

2.3.3. Particle size analysis (PSA)

The intensity of average particle size of sericin/ β -cyclodextrin materials was determined by Dynamic light scattering. The results were recorded using Zetasizer Nano ZS90 (Malvern, U.K.). All samples were prepared in aqueous media and sonicated for 30 min.

2.3.4. Scanning electron microscopy (SEM)

The surface topology of sericin/ β -cyclodextrin material was studied by using scanning electron microscopy EVO 50 (Zeiss, Germany) running at 20 kV at 10K magnification. The powder sample was mounted over metal sample holder by using carbon tape. Samples were gold coated with help of an EMS 850 sputter-coating device before the sample imaging.

2.3.5. Fourier Transform Infrared (FTIR) Spectroscopy

The FTIR spectra of samples were analyzed on the FTIR spectrophotometer (Nicolet 6700) to find out the functional groups presence in sericin/ β -cyclodextrin material in transmission mode scan with scanning range from 4000 nm to 400 nm.

2.3.6. Ferric Hydroxamate Test

Esterification reaction between β -cyclodextrin and sericin was identified through Ferric Hydroxamate Test. The sample was dissolved in a various amount of hydroxylamine hydrochloride (0.5N), 95% ethanol (95%), and NaOH (6N) and test followed according to Goddu et al. [43]. 5% ferric chloride solution was used as an indicator. A deep burgundy color was a positive test, and yellow was negative.

2.3.7. Thermogravimetric analysis (TGA)

The thermal degradation behavior was analyzed by Thermogravimetric Analyzer (Parkin Elmer, USA). The samples were analyzed on 50-800°C at a rate of heating of 10°C/min. The nitrogen atmosphere was maintained during the process with 50 ml/min flow rate to avoid the oxidation of samples.

2.4. Preparation and application on cotton fabric samples

Various compositions of sericin/ β -cyclodextrin were synthesized by the citric acid route. Citric acid and β -cyclodextrin were used in a molar ratio (1:1). These sericin/ β -cyclodextrin materials were loaded with various amounts of α -tocopherol. α -tocopherol was mixed with ethanol and stirred for 30 min as per the quantity given in Table 3.1. Ethanol containing α -tocopherol solution was added with sericin/ β -cyclodextrin solution and stir for 1 hr at 60°C for making inclusion complex with α -tocopherol.

5% sericin/ β -cyclodextrin aqueous solution was made with 50 gpl of citric acid and 10 gpl of Sodium hypophosphate (ratio of 5:1) under magnetic stirring at 200 rpm. Bleached cotton fabrics were treated with above prepared sericin/ β -cyclodextrin in various add-ons (5% & 7%) by pad-dry-cure. Treated samples were dried at 80°C for 2 mins and cured at 130°C for 5 min.

2.5. Evaluation of their activities on cotton fabric samples

2.5.1. Scanning electron microscope (SEM)

Surface morphology of sericin/ β -cyclodextrin functionalized cotton fabrics were evaluated under the Scanning Electron Microscope (SEM) analysis. The functionalized fabrics were scanned at different places on the sample using EVO 50 (Zeiss, Germany) at 10K magnification running at 20 kV. The fabric samples were cut into small pieces (3 x 3 mm²) and mounted over a metal sample holder with the help of carbon tape. The sample was coated with a thin layer of gold using an EMS 850 sputter-coating device before taking image.

2.5.2. Fourier Transform Infrared (FTIR) Spectroscopy

The chemical structure of functionalized and untreated cotton fabric samples was analyzed by FTIR-ATR spectra using an IR spectrophotometer (Nicolet 6700, USA). The scan was performed between the ranges from 4000 to 400 cm⁻¹, at a resolution of 4 cm⁻¹ in absorbance mode.

2.5.3. Scavenging activity %

Scavenging activity of α -tocopherol loaded sericin/ β -cyclodextrin treated cotton fabric samples was measured by estimating the free RSA. The scavenging capacity of α -tocopherol loaded sericin/ β -cyclodextrin treated cotton fabric samples against the stable free radical DPPH was measured according to the antioxidant test method. 1" x 1" size sample was taken and added to 3.5 ml of freshly prepared DPPH radical in a methanol solution (0.1 M) and kept for 30 min at 37°C in the dark under sacking. The decolorizing of the supernatant was analyzed at 517 nm by UV-vis spectrophotometer (Shimadzu - UV-2450) and compared with a blank control DPPH methanol solution. The baseline of spectroscopy was fixed by using methanol. The RSA percentage was calculated using the Equation:

$$\text{Scavenging activity (\%)} = 1 - \frac{OD \text{ sample}}{OD \text{ blind}} \times 100$$

Where scavenging activity percentage represents to the free radical scavenging percentages, OD sample represents the absorbance of the functionalized cotton fabric; OD blind represents the absorbance of the blind control.

2.5.4. Weight add-on %

Add-on % was analyzed by measuring the difference in the weight of the conditioned functionalized and control sample. Weight add-on % was calculated using the Equation:

$$\text{Weight add - on (\%)} = \frac{W2 - W1}{W1} \times 100$$

Where, W1 is the initial weight of the control sample and W2, the weight of the functionalized sample.

2.5.5. The release profile of α -tocopherol

2 x 2 cm size of α -tocopherol loaded sericin/ β -cyclodextrin functionalized cotton samples were incubated with 95% ethanol under agitation (20 rpm) for 72 h, and the α -tocopherol release profile was evaluated along time in ethanolic conditions by using UV-vis spectrophotometer (Shimadzu - UV-2450) at 292 nm. A calibration curve was plotted at various concentrations of tocopherol (0.0005, 0.001, 0.0025, 0.005, 0.01 and 0.02 mg/mL) in the same conditions.

2.5.6. Ultraviolet protection factor

UV protection properties of control and functionalized fabric samples were evaluated against the ultraviolet protection factor (UPF) by using a UV spectrophotometer (Labsphere UV-2000S, USA), following the AATCC Test Method 183-2000 test method. An average of five scans was taken in the wavelength range of 290–400 nm.

2.5.7. Wash durability of the finish

Durability of α -tocopherol loaded sericin/ β -cyclodextrin finished samples were measured by the AATCC 61A test method. In this method, each washing cycle was performed with 2 g/L non-ionic detergent at 50 °C for 45 min at 40 rpm with 10 iron balls in Rota dyer (RBE India Launderometer). The washed fabric was dried at 50 °C for 30 min after each cycle of washing. The weight loss of the washed fabrics was analyzed after every 5 washing cycles.

2.5.8. Drop absorbency test

Water absorbency of treated and control samples was measured according to AATCC 79 method. A water drop was fallen into the fabric through the burette from 9.5 mm distance, and time was recorded until a drop of water absorbed completely.

2.5.9. Vertical wicking Test

Wicking property of samples was evaluated using the wicking strip test according to standard DIN53924. Specimen of size 200 x 25 mm, the warp direction parallel length was prepared. The lower end of the sample was immersed in a reservoir of distilled water to which contains 0.5% dilute potassium chromate (0.5%) for tracking the movement of water. The height of the water was measured with a scale that moves above the fabric in the reservoir after various intervals (1, 3, 5, 7, 10, 15 min).

2.5.10. Stiffness Test

The bending length of control and functionalized cotton fabric samples were measured according to ASTM Standard D 1388-96.

2.5.11. Wrinkle resistance Test

The crease recovery angle of control and functionalized cotton fabric samples were measured according to AATCC 66-2008.

2.5.12. Whiteness Index

The CIEXYZ measurement for whiteness index of control and functionalized cotton fabric samples were recorded according to AATCC TM110 by using Computer color matching (Premier Colorscan SS 5100H). The spectrophotometer has been calibrated by using reference and black tiles by using D65 light at 10 ° observers.

III. RESULTS AND DISCUSSION

3.1 SDS-PAGE for molecular weight distribution

The SDS-PAGE method is a quantitative method to determine the molecular weight distribution of the protein. The molecular weight distribution of the sericin sample shown in **Figure 1 (a)**. The sericin sample S1 appears the molecular weight distribution range 63 - 245 kDa in SDS-PAGE diffusion band, while sericin sample S2 appears higher broader the molecule weight distribution ranges 20-245 kDa. Sericin sample S2 was found some proportion of lower molecular distribution and partially soluble in water at room temperature, but that could be easily soluble in hot water. Sericin sample S1 was located molecular distribution at the higher side that could be readily soluble in hot water but difficult to be soluble in water at room temperature. Lower molecular weight sericin (< 50 kDa) is readily soluble in water at room temperature, but a higher molecular weight range 50-300 kDa can only soluble at the higher temperature (90°C). The broader range of molecular weight has been found to affect by the properties and applications of sericin [44].

3.2 UV spectra analysis

The purity of sericin samples was analyzed by UV spectra shown in **Figure 1 (b)**. Two different sericin samples S1 and S2 were collected from a different source and measured for absorbance at the ultraviolet region. Sericin has shown absorbance peak in the ultraviolet region at 275.4 nm due to the presence of peptide bonds and aromatic acids. Sericin has aromatic acids such as tyrosine, tryptophan, and phenylalanine, which absorb UV radiation at 260-280 nm range due to $\pi \rightarrow \pi^*$ transition. Sericin samples showed a characterization peak at 275.4 nm that confirm the material. The purity of sericin samples was analyzed by A-ratio (A_{280nm}/A_{260nm}) [44]. Proteins show UV absorption at 280 nm (A_{280nm}) due to the presence of amino acids with an aromatic ring. Nucleic acids also present in sericin, which shows UV absorption at 260 nm (A_{260nm}). Higher the A_{280nm} value displays a higher purity of sericin. Sample S1 shows a lower A-ratio 1.05, and sample S2 shows a higher value that is 1.4. Sample S2 has shown much closer to the ideal value of 1.8. Samples shows differ in A-ratio due to extraction methods as well as a source of extraction of sericin. Higher purified sample S2 was used for the synthesis of further material.

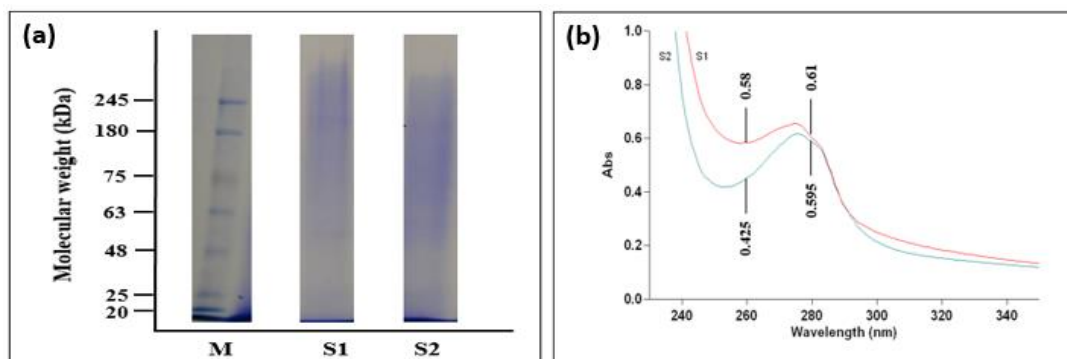


Figure 1: (a) SDS-PAGE analysis of different sericin samples [M - Marker, sericin (S1) - 20 mg/mL, sericin (S2) - 20 mg/mL] and (b) UV spectra of different sericin samples at 260 nm & 280 nm (absorbance value)

3.3 Particle size analysis of synthesized material

Dynamic light scattering (DLS) is a usual technique that can determine the particle size in a colloidal solution. These particles have suspended in the solvent and moving randomly through brownian motion that can be responsible for scattering the light. The average intensity of particle size distribution of sericin was found 440 nm in diameter shown in **Figure 2 (a)**. The particle scattering intensity for biopolymers and biological materials is proportional to the square of the molecular weight. Therefore, the sericin sample has a higher intensity of molecular weight 22 kDa. The average intensity of the particle size distribution of sericin/ β -cyclodextrin was found to be 1882 nm in diameter, shown in **Figure**

2 (b). Results indicated that average particle size increased after grafting between sericin and β -cyclodextrin due to both the reactants bonded covalently and formed a stable complex with increase their molecular weight. Single peak with very narrow width distribution denotes that the sample has monomodal, monodisperse, and near-spherical or spherical.

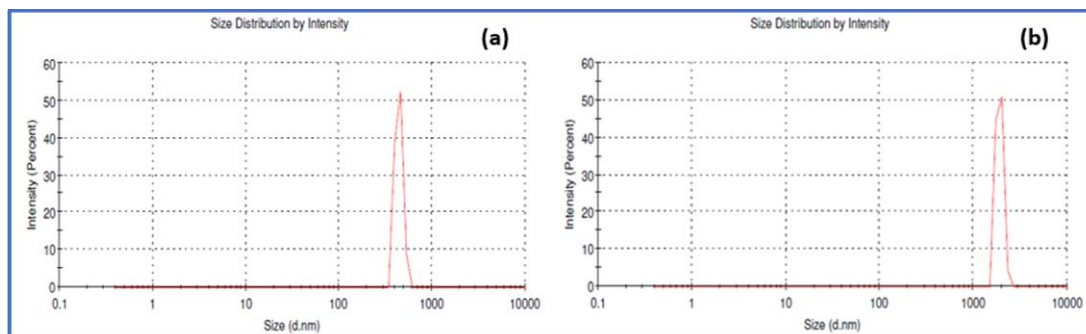


Figure 2: Dynamic light scattering (DLS) particle size analysis, (a) Particle size distribution by intensity % of sericin in aqueous solution after sonicated for 30 min, (b) Particle size distribution by intensity % of sericin/ β -cyclodextrin material after sonicated for 30 min

3.4 SEM analysis

SEM analysis of sericin/ β -cyclodextrin samples was done to observe the surface morphology of the particles. The sericin/ β -cyclodextrin has a spherical ball-like morphology in micro-size in aqueous solution, and also form a film as shown in **Figure 3 (a)**. Results indicated that size distribution of spherical ball-like particles widely varied in size. They appeared in highly aggregate form, and that could create a problem of solubility. SEM images also indicated that the formation of a film of synthesized material that helps to form a film over textile material and provides a platform to slow release of a guest substance from the inclusion complex. Another SEM image of sericin/ β -cyclodextrin was studied that extracted from freeze-drying. SEM image of sericin/ β -cyclodextrin from freeze-drying showed flat structure in nano-size with broad size distribution shown in **Figure 3 (b)**. Therefore, the size distribution and shape have significantly depended on the extraction method of the particles.

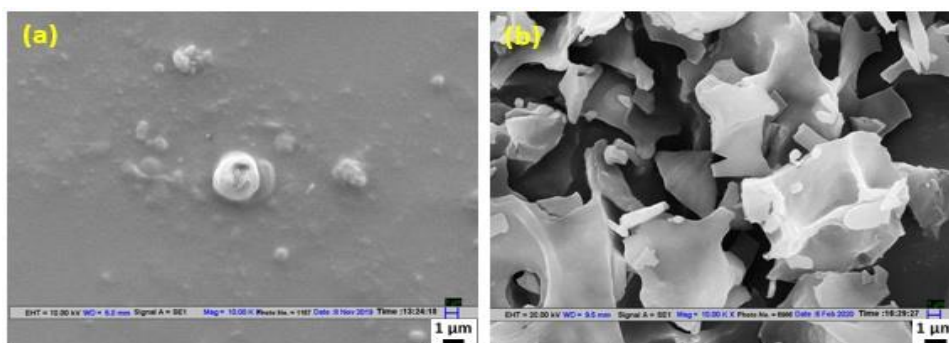


Figure 3: Scanning electron microscopy (SEM) images of sericin/ β -cyclodextrin material, (a) SEM images of sericin/ β -cyclodextrin material in aqueous solution, (b) SEM images of sericin/ β -cyclodextrin material in powder form extracted through freeze-drying

3.5 FTIR spectra of material

In FTIR spectra of sericin show the absorption peak of amide I, amide II and amide III vibration bands at 1632 cm^{-1} , 1533 cm^{-1} , and 1230 cm^{-1} respectively. The amide I vibration band denotes C-O stretching. The amide II and amide III absorption peak denote secondary N-H bending and C-N functionalities, respectively, shown in **Figure 4 (a)**. The protein materials conform to the positions of these bands. The random coil and β -sheet of amide I is confirmed by absorption peak at 1632 cm^{-1} . The random coil of amide I and amide II is confirmed by absorption peak at 1533 cm^{-1} and 1230 cm^{-1} , respectively [45]. The β -cyclodextrin structural characteristics is confirmed by the adsorption peaks at 1139 , 1034 , 939 , and 867 cm^{-1} shown in **Figure 4 (b)**. The sericin/ β -cyclodextrin sample shows the absorption peak at 1200 cm^{-1} and 1731 cm^{-1} , which corresponds to C-O stretching vibration and C-O-C stretching vibration of ester and carboxyl groups. The absorption peak at 1731 cm^{-1} indicate a cross-linking reaction between the carboxyl group of sericin and the hydroxyl groups of β -cyclodextrin with the carboxyl groups presence in citric acid and formed ester linkage shown in **Figure 4 (c)** [46].

3.6 Ferric hydroxamate test

The ferric hydroxamate test of samples was performed to the identification of ester formed during the esterification reaction. The formed ester is reacted with hydroxylamine hydrochloride and formed a hydroxamic acid (HOHN-C=O), which gave a positive ferric chloride test and shown deep burgundy color. As evident from **Figure 5 (a)**, unreacted sericin and β -cyclodextrin showed yellow color. The yellow color indicated the negative results of the formation of ester. Sericin/ β -cyclodextrin showed a deep burgundy color, which means the presence of ester in grafted material and confirms the possible reaction shown in **Figure 5 (b)** [47]. This deep burgundy color is obtained due to a complex between the ferric ion and the hydroxamic acid. Hydroxamic acid contains three metal-binding groups ($-\text{CONHO}^-$), which can complete the coordination sphere of the iron (III) and form a more stable 1:1 complex.

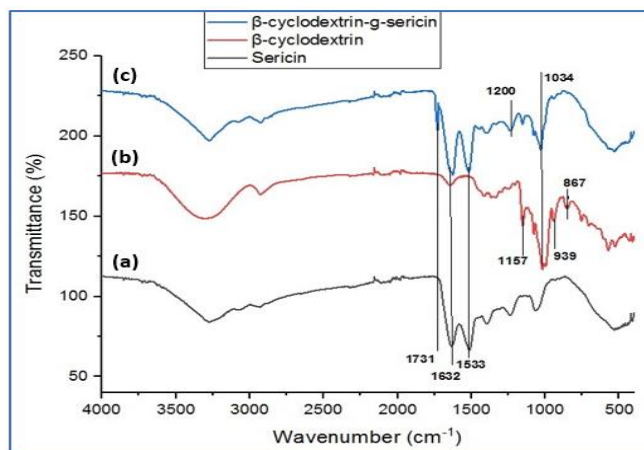


Figure 4: FTIR spectra, (a) Sericin, (b) β -cyclodextrin, and (c) sericin/ β -cyclodextrin

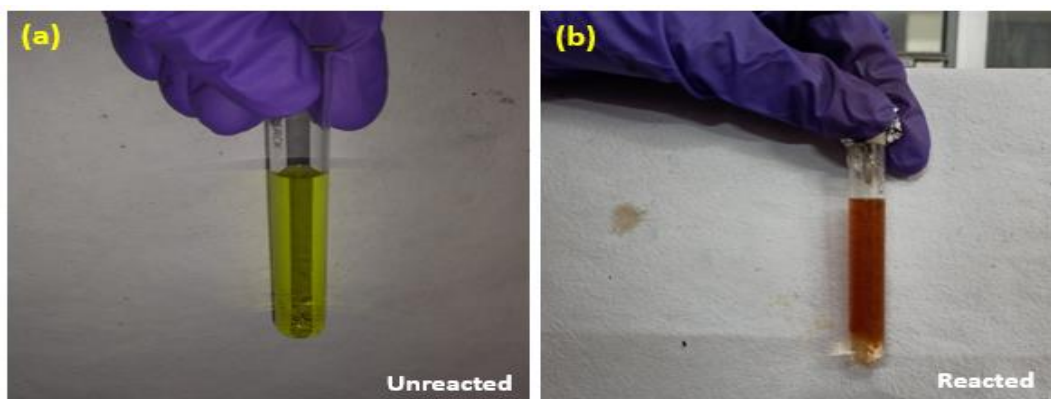


Figure 5: The ferric hydroxamate test to the chemical identification of ester, (A) Unreacted β -cyclodextrin and sericin, (B) sericin/ β -cyclodextrin

3.7 Thermal Gravimetric analysis

TGA curve of samples are depicted in **Figure 6**. Sericin exhibited weight loss below 100°C due to the evaporation of water. TGA analysis of mulberry sericin has shown no significant changes in weight up to 210°C , and then degradation started sharply illustrated in **Figure 6 (a)**. The sericin was lost its 60 % weight at 340°C . More than 80% of the thermal degradation of sericin was observed at 800°C . According to Tsukada [48], sericin shows decomposition reaction goes through a molecular flowing and scission at 220°C , and then thermal degradation occurred. Tsukada [49] stated that the amorphous region of sericin has thermally decomposed at 201°C . Sericin/ β -cyclodextrin has shown 10% degradation at 300°C , and then sharp degradation was observed illustrated in **Figure 6 (b)**. 40% thermal degradation of sericin/ β -cyclodextrin was found at 720°C . Results indicated that 55% weight loss was observed at 800°C in sericin/ β -cyclodextrin. Therefore, the sericin/ β -cyclodextrin has shown a low thermal degradation rate as compare to reactants which is indicative of its enhanced thermal stability. Higher thermal stability of sericin/ β -cyclodextrin material means that it can be used for textile finishing at a higher temperature.

3.8 SEM analysis of fabric samples

SEM images of untreated and treated cotton fabric were confirmed to deposition sericin/ β -cyclodextrin over fabrics. The untreated cotton sample was observed to be a smooth surface, shown in **Figure 7 (a)**. However, in the case of treated fabrics, the SEM analysis shows uniform distribution of sericin/ β -cyclodextrin on the surface of cotton fabric shown in **Figure 7 (b)**. Synthesized sericin/ β -cyclodextrin was cross-linked with cotton in the presence of binder and catalyst.

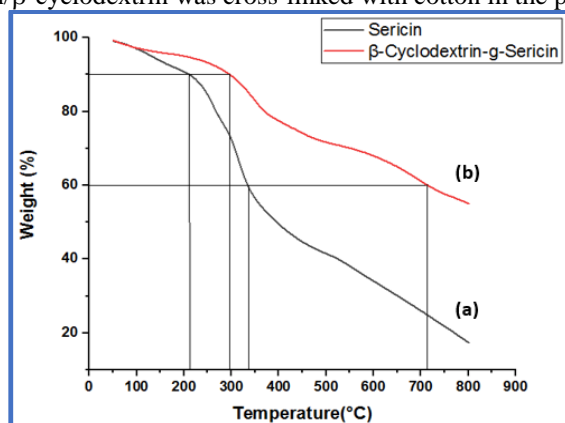


Figure 6: Thermal gravimetric (TG) analysis curve, (a) sericin and (b) sericin/ β -cyclodextrin material

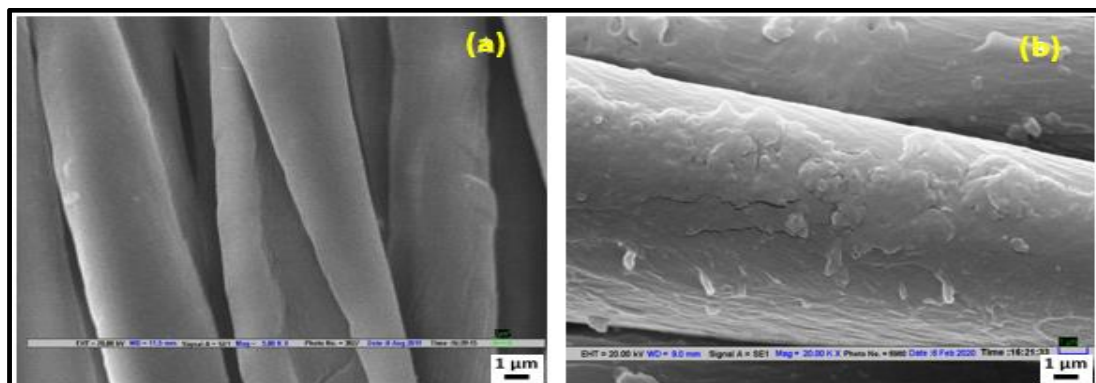


Figure 7: Scanning electron microscopy (SEM) images, (a) Untreated cotton fabric, (b) Cotton fabric treated with sericin/ β -cyclodextrin material.

3.9 FTIR spectra of fabric sample

The FTIR spectra of bleached and treated cotton were shown in **Figure 8**. Untreated cotton fabric shows characteristic vibration bands in 3334 cm^{-1} for -OH stretching due to the presence of hydroxyl group in cotton fabric. The absorbance peak at $2,901\text{ cm}^{-1}$ corresponds to CH_2 stretching vibration. The peak at 1731 cm^{-1} observes in treated cotton fabric with sericin/ β -cyclodextrin. This is attributed to the C-O stretching vibration of carboxyl groups and ester groups due to the formation of an ester bond between the sericin carboxyl group and the hydroxyl group of cotton through citric acid.

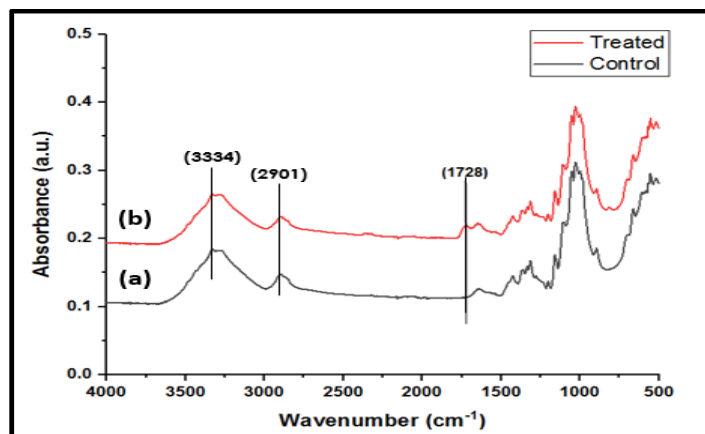


Figure 8: FTIR spectra (a) Untreated cotton, (b) treated cotton with sericin/β-cyclodextrin

Figure 9 illustrates a schematic chemical reaction between sericin/β-cyclodextrin and cellulose by using citric acid as a cross-linking agent. Carboxyl group present in citric acid has possibly react with an amide group of sericin and hydroxyl group of cellulose through amide linkage as well as carboxyl group present in sericin/β-cyclodextrin and the hydroxyl group of cellulose through an ester linkage in presence of heat. Amide and carboxyl group present in sericin/β-cyclodextrin has possible to react with the hydroxyl group by hydrogen bonding.

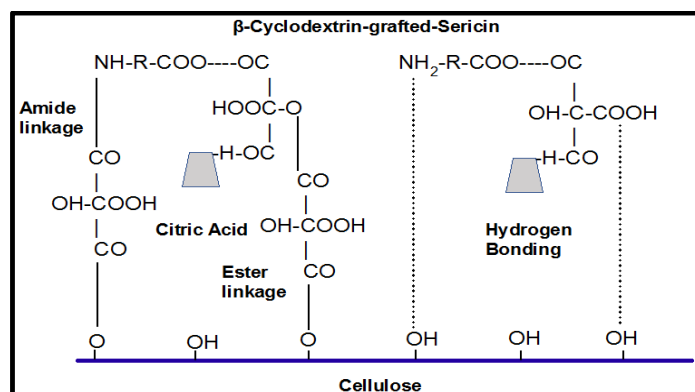


Figure 9: Schematic illustration of the chemical reaction between sericin/β-cyclodextrin and cellulose through citric acid.

3.10 Scavenging activity of fabric sample

Antioxidant properties of treated and untreated cotton fabric were observed at different wash cycles presented in Table 1. Untreated cotton fabric was shown a deficient scavenging activity below 6%. Without loading α-tocopherol. Sericin/β-cyclodextrin treated cotton fabric were displayed scavenging activity 69.18%, 58.2%, 50.2%, and 40.6% respectively for un-wash, 5, 10, and 20 washes sample illustrated in Figure 10. After loading of α-tocopherol, the samples were displayed excellent scavenging activity as compared to unloaded fabric samples that shown 97.28%, 88.45%, and 68.45% respectively for un-wash, 5 and 20 washes in 1:1 ratio of Sericin and β-cyclodextrin. β-cyclodextrin has lipophilic nature that facilitates to form a host-guest complex with α-tocopherol. β-cyclodextrin cavity has hydrophobic nature from inside due to a line of hydrogen atoms and glycosidic oxygen bridges. Non-bonding electron pairs of glycosidic oxygen bridges are arranged inside the cyclodextrin, which holds the hydrophobic substances, such as α-tocopherol. After washing, the scavenging activity of samples was displayed significantly lower due to the washout of antioxidant agents, which presents on the fabric surface as well as leach out from the inclusion complex due to the external stimuli, such as light, oxygen, and temperature. Lower Sericin and β-cyclodextrin ratio was shown excellent scavenging activity in an un-wash sample approx. 97-98% for 1:0.75 ratio and 96-98% for 1:0.5 ratio. After washing, higher sericin and β-cyclodextrin ratio samples were shown lower scavenging activity as compare to un-wash due to less amount of inclusion complex formation between β-cyclodextrin and α-tocopherol. These samples have shown scavenging activity between 85-88% and 80-82% respectively for sericin and β-cyclodextrin ratio of 1:0.75 and 1:0.5 after 5 washes. After 20 washes, these treated cotton fabrics were shown good scavenging activity ranges from 62-67% and 56-68%, respectively. These samples have higher

wash durability due to the slow release of α -tocopherol from the cavity of β -cyclodextrin. Sericin has an excellent ability to form a film. This film-forming capability of Sericin helps to slow the release of α -tocopherol. β -cyclodextrin is grafted with sericin that forms a film over fibre surface. The grafted film has provided a slow release of α -tocopherol and protects α -tocopherol from external stimuli, such as light, oxygen, and temperature. Sericin has very good hygroscopic material due to the presence of polar group amino acids. When the human body releases perspiration, the fabric absorbs with the help of sericin due to hydrophilic nature. Fabric pH changes after absorption of perspiration that help to release of α -tocopherol. The released α -tocopherol provides an antioxidant effect on human skin due to a hydroxyl group in its chromane ring that has reduced available free radicals by donating a hydrogen atom. Sericin has itself provided good scavenging activity due to the presence of hydroxyl group that has reduced available free radicals on human skin by donating a hydrogen atom.

Table 1: Scavenging activity of fabric samples at different proportions and wash duration

Sample Code	Sericin: β -Cyclodextrin Ratio (w/w)	α -tocopherol ml/ltr	Scavenging Activity (%)			
			Un-wash	5 Wash	10 Wash	20 Wash
Control			5.58			
SB11-W	1:1	0	69.18	58.2	50.2	40.6
SB11-1	1:1	1	97.21	88.45	81.54	68.45
SB11-2	1:1	2	98.23	89.2	83.1	68.3
SB11-3	1:1	3	99.12	90.21	83.3	70.5
SB12-1	1:0.75	1	97.12	85.2	77.2	62.1
SB12-2	1:0.75	2	98.1	85.2	80.1	66.24
SB12-3	1:0.75	3	98.89	88.22	82	67.1
SB13-1	1:0.5	1	96.25	80	70.2	56.5
SB13-2	1:0.5	2	98.2	80.1	70.8	58.5
SB13-3	1:0.5	3	98.6	82.5	75.4	66.5

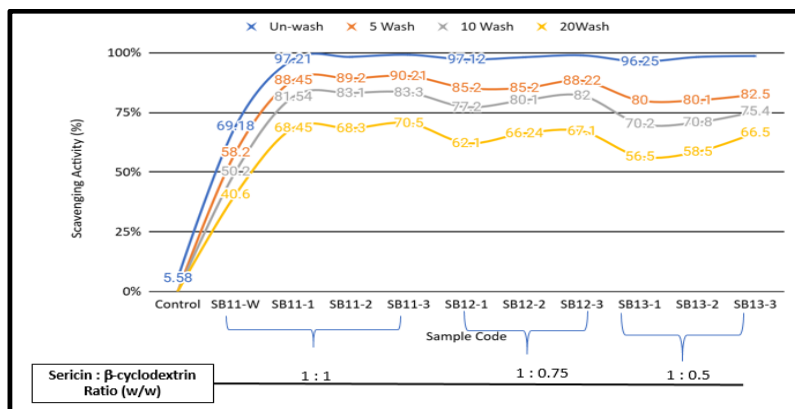


Figure 10: Scavenging activity % of α -tocopherol loaded sericin/ β -cyclodextrin after different ratio and washing cycle. The results indicated that scavenging activity % was increased in respect of time illustrated in **Figure 11** The treated samples were displayed good scavenging activity % even after 60 min that evident treated fabric has enough α -tocopherol that donated hydrogen atom to reduce free radicals for very long time. The results supported that α -tocopherol release slowly from the inclusion complex formed with sericin/ β -cyclodextrin against DPPH free radicals. Without α -tocopherol loaded samples were shown increment of scavenging activity % after 30 min due to the presence of sericin on the fibre surface.

3.11 Release of α -tocopherol from treated fabric samples

Figure 12 is shown a suitable linear Equation of absorbance value of α -tocopherol has arrived at a different concentration at 292 nm by UV-vis spectroscopy with the help of the calibration curve. **Figure 13** is presented the quantitative release behavior of α -tocopherol from sericin/ β -cyclodextrin in various amounts of α -tocopherol loaded cotton fabric at the different wash cycles. The results indicated that α -tocopherol was released from the treated fabric even after 20 washes. The treated samples have shown excellent durability against laundering due to the slow release of α -tocopherol from β -cyclodextrin inclusion complex. Sericin/ β -cyclodextrin material has formed a thin film over fibre surface, which helps to release a sufficient amount α -tocopherol from the inclusion complex against the various stimuli. Un-washed treated

samples were shown a higher amount of α -tocopherol available in the sample that formed the maximum amount of inclusion complex. The amount of α -tocopherol has decreased after washing of treated fabric.

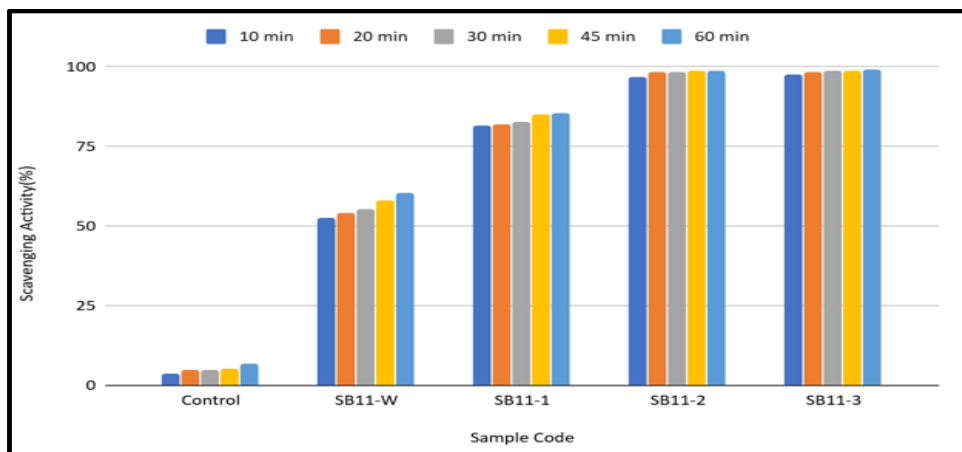


Figure 11: Scavenging activity % of α -tocopherol loaded sericin/ β -cyclodextrin at a different time duration.

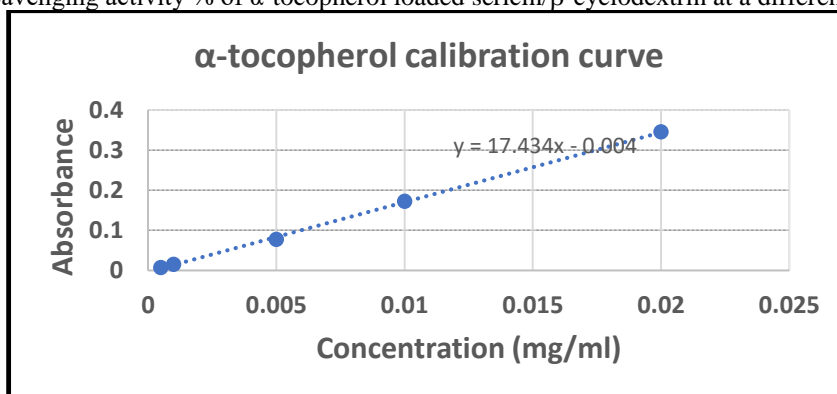


Figure 12: Calibration curve of different concentration of α -tocopherol at 292 nm by UV-vis spectroscopy.



Figure 13: Release of α -tocopherol from sericin/ β -cyclodextrin treated cotton fabric samples at different washing cycle.

3.12 Effect of add-on % on Scavenging activity % of fabric samples

The results indicated that the higher weight add-on % samples were shown higher scavenging activity due to the presence of higher α -tocopherol on fabric samples. There was found no significant difference in scavenging activity at 5% and 7% weight add-on. The fabric sample has a sufficient amount of α -tocopherol on the fibre surface as well as between the yarn voids that provides a sufficient amount of hydrogen atom to neutralize free radicals illustrated in Figure 14. Higher weight add-on samples were shown higher scavenging activity due to a higher amount of material cross-linked with fabric that formed a highly covered film over fibre surface. Higher β -cyclodextrin ration must entrap the higher amount of α -tocopherol in its cavity and shows higher scavenging activity.

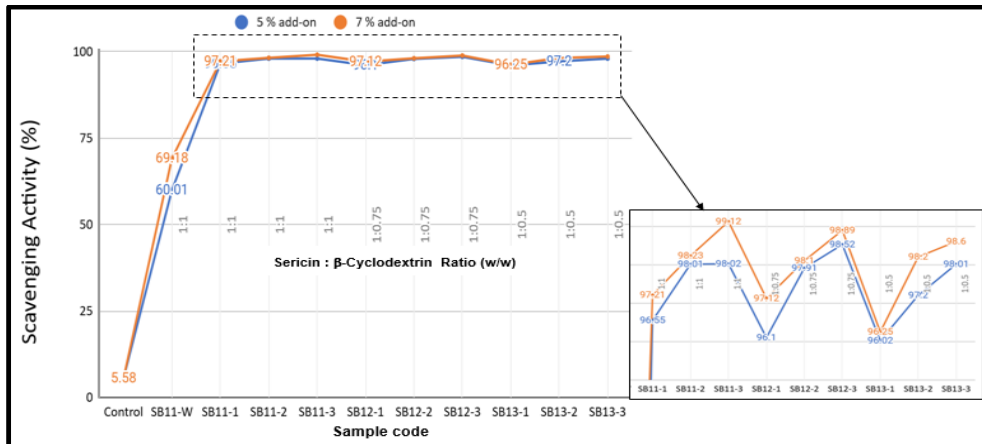


Figure 14: Scavenging activity % of fabric samples at different fabric weight add-on before washing.

3.13 Ultraviolet protection factor (UPF)

Ultraviolet protection factor (UPF) is the most crucial property of textiles that wear users in the warm climate area. This property of textiles protects the substrate from the ultraviolet radiation of the sun and protects human skin. The specific wavelength ultraviolet radiation falls on the fabric; the energy of UV radiation strikes the fibre surface. There UV radiation may be absorbed, reflect, and transmitted through fabric. These depend on the type of fibre structure, smoothness, porosity, and applied finishes (Tahlawy et al., 2015). The untreated cotton sample was shown poor UPF value mentioned in Table 2. Sericin with higher α -tocopherol samples was displayed good to very good UPF value varies from 18 to 26. Sericin has consisted of various aromatic amino acids, such as tyrosine, tryptophan, and phenylalanine, which can absorb UV B radiation at 280 nm and protect human skin from damage. These aromatic acids can absorb short-wavelength ultraviolet radiation with higher energy, which excites them to a higher energy state. Absorbed higher energy has dissipated as longer wavelength radiation, which is responsible for damaging human cells (Das 2010). Higher α -tocopherol concentration was shown good to very good UPF value. α -tocopherol prevents photo-oxidative damage of skin cells due to UVB-induced ultraviolet radiation (Maalouf et al., 2002). α -tocopherol has a heterocyclic structure with a chroman ring that can be responsible for make them photoprotective. Higher fabric weight add-on may give significantly higher UPF value.

Table 2: UPF of fabric samples at 5% fabric weight add-on before washing

Sample Code	Sericin: β -cyclodextrin Ratio (w/w)	α -tocopherol ml/ltr	UPF Value	UPF Rating	Protection category
Control			5	5	Poor
SB11-1	1:1	1	20	20	Good
SB11-2	1:1	2	20	20	Good
SB11-3	1:1	3	26	25+	Very good
SB12-1	1:0.75	1	19	15+	Good
SB12-2	1:0.75	2	19	15+	Good
SB12-3	1:0.75	3	20	20	Good
SB13-1	1:0.5	1	18	15+	Good
SB13-2	1:0.5	2	19	15+	Good
SB13-3	1:0.5	3	19	15+	Good

3.14 Moisture Transport properties

Wicking is one of the transport mechanisms that transport liquid in porous media through capillary force in textiles. Table 3 mentioned the wicking height of various cotton samples at different time duration. The results indicated that treated cotton samples were shown higher moisture transport in terms of wicking as compared to the control sample. Sericin has more than 80% amino acid in hydrophilic nature that contains bulkier side groups that easily hold the moisture. Sericin covers the fibre surface and provides a path to transfer moisture from sericin to fibre capillaries that can promote the wicking of liquid. Higher weight add-on samples were shown higher wicking height due to a higher amount of sericin

available on the fibre surface. Lower ratio of sericin and β -cyclodextrin were displayed higher wicking height due to a higher amount of unreacted sericin that holds higher liquid and creates a higher capillary force to drag the fluid against the gravity. Warp direction wicking has been found to be higher than weft direction due to tightness of warp in fabric, which provides straight capillary to transport water through capillary force. There was not found a significant change in water absorbency of treated samples that readily absorbs water droplets quickly due to the hydrophilic nature of sericin and β -cyclodextrin.

Table 3: Moisture management parameter of fabric samples at different weight add-on %

Sample Code	Sericin: β -cyclodextrin Ratio (w/w)	Add-on %	Absorbency sec	Vertical wicking height (cm)							
				Warp				Weft			
				1 min	5 min	10 min	15 min	1 min	5 min	10 min	15 min
Control			3	2	3.7	4.8	5.5	1.9	3.3	4	4.5
SB11	01:01	5	3	2.4	4.3	5.5	6.5	2.2	3.7	4.3	5
SB12	01:00.7	5	3	2.4	4.3	5.6	6.7	2.1	3.6	4.3	4.7
SB13	01:00.5	5	3	2.6	4.6	5.8	6.7	2	3.7	4.3	4.7
SB11	01:01	7	3	2.6	4.6	5.9	6.8	2.3	3.8	4.7	5.2
SB12	01:00.7	7	4	2.5	4.5	5.7	6.6	2.2	3.6	4.3	4.8
SB13	01:00.5	7	3	2.5	4.4	5.7	6.6	2.2	3.6	4.2	4.7

3.15 Fabric stiffness

Bending length represents the stiffness of the fabric. Higher bending length means the stiffer fabric. The bending length of the samples is presented in **Table 4**. The results indicated that the treated samples were shown higher stiffness as compared to the control sample. sericin/ β -cyclodextrin is attached to cotton by using a cross-linker that occupies a lot of hydroxyl group of cotton. These fabric samples are not able to form a new hydrogen bond; this may become fabric stiffer. Also, sericin/ β -cyclodextrin is formed a thin layer over the fibre surface as well as binding the fibre inside the yarn than can be made fabric stiffer and resistant to bending. Higher weight add-on samples were shown stiffer. Stiffness of warp direction fabric samples was shown higher than weft direction Warp is a high tension condition than weft that cannot bend easily as compared to the weft.

3.16 Crease recovery

Crease recovery of treated samples was shown better crease recovery than the control sample. The reason behind the higher crease recovery is that the finished material is attached to the cotton hydroxyl group with the help of a cross-linker. During the bending of cotton fabric, new hydrogen bond formation is restricted due to the lack of hydroxyl group available on cotton, which helps to recover cotton fabric in its original position. That results low crease formation in treated materials, and the fabric becomes crease-resistant. The results indicated that Crease recovery angle was significantly dependent on fabric weight add-on. The fabric samples with 7% weight add-on were exhibited higher crease recovery than 5 %. More number of hydroxyl groups have blocked on cotton by the higher add-on, that resists to the formation of a new hydrogen bond during wetting or bending of fabric. That results in lower crease formation on fabric. **Table 4** is mentioned the value of the crease recovery angle (CRA) and the whiteness of various samples.

3.17 Whiteness Index

The results indicated that the control sample was shown whiter than treated in **Table 4**. Sericin has a slightly yellowish nature, which affects the whiteness of the fabric samples. Citric acid was used as a cross-linking agent to attached finish on cotton; they also one of the reasons to made fabric yellow. Citric acid has three carboxyls and one hydroxyl group. The two-carboxyl groups of citric acid are attached to the hydroxyl group of cotton and β -cyclodextrin by esterification reaction and formed a double bond. The formation of an alternative double bond has display yellow in nature. Higher weight add-on samples were shown lower whiteness index due to higher sericin loading on cotton as well as more ester linkage.

Table 4: Mechanical and optical properties of cotton fabric samples

Sample Code	Sericin:β-Cyclodextrin	Add-on	Bending length (cm)		Crease recovery angle	Whiteness Index
	Ratio (w/w)	%	Warp	Weft	(°)	CIE
Control			4.7	2.6	181	65.3
SB11	1:1	5	4.9	2.9	198	58.4
SB12	1:0.75	5	4.9	2.9	198	56.3
SB13	1:0.05	5	4.8	2.8	195	55.1
SB11	1:1	7	5.0	2.9	208	56.0
SB12	1:0.75	7	5.0	2.9	205	52.9
SB13	1:0.05	7	4.9	3.0	205	52.2

IV. CONCLUSION

Sericin/β-cyclodextrin materials are synthesized successfully in a different ratio. Sericin is characterized by SDS-PAGE for molecular weight analysis and calculates A-ratio for the purity of Sericin has been determined using UV spectra analysis. Sample S2 has a higher A-ratio 1.4 and molecular range 50-245 kDa, which has been used for further synthesis. Particle size distribution and SEM have proper evidence to synthesize finishing materials that vary from micro to nano-size in diameter. After a combination of sericin/β-cyclodextrin, synthesized materials have increased their particle size. FTIR spectra and ester identification test of synthesized material have confirmed the grafting of Sericin with β-cyclodextrin. TGA of sericin/β-cyclodextrin has confirmed that it increases the thermal stability of synthesized material. A SEM image of treated cotton samples shows the presence of sericin/β-cyclodextrin on the surface of the fabric. FTIR spectra of sericin/β-cyclodextrin treated has confirmed cross-linking on cotton fabric. The sericin/β-cyclodextrin treated cotton fabric samples show very good scavenging activity ranges 56-70% after 20 washes. Sample SB11-3 shows higher scavenging activity before and after washing. Treated samples show the slow release of α-tocopherol from sericin/β-cyclodextrin and shown excellent scavenging activity after 60 min. Higher add-on 7% shows better scavenging activity than 5%. Higher Sericin and β-cyclodextrin ratio shows higher UPF value varies from 20-26, which is good to very good. Treated samples have shown excellent water absorbency as well excellent moisture transport property. Wicking height has been found to be varying from 5.5 to 5.9 cm in the warp direction and 4.2 to 4.7 cm in the weft direction after 10 min. Stiffness of fabric has increased with higher add-on after the finish application on cotton. Warp direction stiffness has increased by 4-6% and weft direction 7-15% as compared to the control sample. Treated samples have displayed increment of crease recovery angle ranges from 195 to 208° as compare to control, which is 181°. At 7% add-on shows higher crease recovery angle range from 202 to 208° but shows lower whiteness index. Treated samples have a lower whiteness index of approximately 11-20% as compare to the control sample. This finish has a lot of potentials to provide excellent antioxidant properties, good UV resistant and good moisture transmission properties. Finished cloths can be used as cosmetotextiles, which can enhance the ability to take care of human skin after several washes.

ACKNOWLEDGMENT

The authors would like to thank **Dr. Subhas V. Naik**, Director, Central Silk Technological Research Institute, Bengaluru for providing sericin for this research work. The authors are also thankful to the Nano Research Facility (NRF) and Central Research Facility (CRF) of Indian Institute of Technology Delhi (IIT Delhi) for providing various characterization facilities.

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