

## Development of Medical Textiles from Sericin Coated Polyester Materials

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#### ABSTRACT

Sericin, a silk protein, has high potential for use in biomedical applications. It has important attributes such as excellent oxygen permeability, cell protecting and antioxidant action, moisture regulating ability, protection from ultraviolet (UV) radiation and microbes, wound healing, anticancer and anticoagulant properties. Sericin, however, has no direct affinity for textiles. In this study conditions for imparting a durable finish to polyester, based on sericin have been optimized. Ten grams per litre of sericin concentration with 10 mL/L of glutaraldehyde cured at 130°C for 2 min was found to give best application. Sericin content in finished samples was estimated by measuring the colour value of treated fabrics dyed with Methylene Blue. SEM analysis showed creation of nano-roughness on the surface of polyester after exposure to UV light and smoothening of fabric surface after application of sericin. Treated samples showed enhanced vertical wicking and moisture regain. They also exhibited improved antistat, ultraviolet protection and radical scavenging activity. These properties make sericin-treated fabrics suitable for use as medical textiles in wound dressings and for healing abrasive skin injuries in patients suffering from atopic dermatitis, pressure ulcers and rashes.

Keywords: Polyester; Surface Modification; Silk Sericin; Textile Finishing; Medical Textiles; Excimer Radiation

Abbreviations: UV: Ultraviolet; PET: Polyethyene Terephthalate; GTA: Glutaraldehyde

#### Introduction

In recent years, the population explosion and environmental pollution have increased the interest of researchers in the discovery of new health and hygiene-related products for the well-being of mankind. Among the possible approaches initiated by the textile industry, the use of low environmental impact technologies with use of sustainable biopolymers presents a novel possible avenue for largescale development of medical textiles. Medical textiles account for a huge market owing to the widespread need for them, not only in the hospital, hygiene and healthcare sectors but also in hotels and other environments where hygiene is required [1]. Textiles characterized by high moisture sorption, smooth surface, formaldehyde free, are being specially designed to heal inflammatory reactions in patients suffering from atopic dermatitis and pressure ulcers [2,3]. Some other desirable properties of medical textiles include non-toxicity, sterilizability, biocompatibility, good absorbability, anti microbial activity and freedom from additives and contaminants [4,5]. A wide number of chemicals have been employed to impart these properties on textile materials. These chemicals include inorganic salts,

organometallics, phenols, antibiotics, urea and related compounds, formaldehyde derivatives, amines, etc. [6]. However, majority of such agents are toxic to humans and are not environmental friendly [7]. In view of these ecological and environmental concerns, biopolymers like sericin and chitosan that are natural in origin have the potential to become key resources in the development of sustainable medical textiles. Over the past few years, enormous attention has been paid to use chitosan in different application fields like textile processing, pharmaceuticals, food industry, etc [8-10]. However, sericin is a comparatively new biopolymer which has not been fully explored. Silk sericin is a biopolymer with a unique structure leading to enhanced performance properties. It is a water-soluble globular protein derived from silkworm Bombyx mori, and represents a family of proteins whose molecular mass ranges from 10 to 310 kDa [11]. It constitutes about 20-30% of the total cocoon weight and consists of 18 amino acids most of which have strong polar side chains such as hydroxyl, carboxyl and amino groups [12]. Its hydrophilicity arises from the high content of serine (33.4%) and aspartic acids (16.7%) which have a high content of hydroxyl groups [13,14]. Sericin has gained importance because of its unique properties like biocompatibility, biodegradability [15], antibiotic-antibacterial activity, UV resistance, oxidative resistance and moisture absorption ability [16,17]. In clinical studies, sericin has exhibited biological activities such as tyrosinase inhibition activity (Kato, et al. [18]), cell healing, cell proliferation and pharmacological functions such as anticoagulation, anticancer and cryoprotection activity [18-21]. It enhances the elasticity of skin and has shown anti-wrinkle and anti-aging effects via its collagen promoting activity [21]. Because of its antioxidant activity sericin inhibits the oxidation reaction of free radicals and prevents growth of microbes [22]. These properties allow wide-spread use of sericin in medical, cosmetics and food industry (Kundu, Dash, Dash, & Kaplan, 2008) [23]. Sericin can be cross-linked, copolymerized or blended with other polymers to produce new biodegradable materials with improved properties [24]. Fabrics treated with sericin have been reported to prevent abrasive skin injuries and development of rashes in products such as diapers, diaper liners and wound dressings [15]. Amongst man-made fibres, Polyethyene terephthalate (PET) is a very useful biomaterial. However it is non-polar, hydrophobic and inert in nature, making it difficult to apply any finish directly on it. To overcome this problem, surface modification as pretreatment can be carried out. Techniques of surface functionalization results in nanostructuring and helps in formation of reactive functional groups on the fabric surface which further aid in adherence of finish on fabric surface [25]. The current study aims to explore green chemistry approaches to develop polyester textiles for aesthetic, hygienic and medical applications. In this study, the conditions for application of sericin on polyester have been optimized in terms of concentration, time and temperature of curing. The sericin treated fabrics were studied on surface morphology, moisture related properties and performance properties like UV protection, free radical scavenging activity (RSA) and anti-static properties which are required for medical textiles.

## **Technical Details**

The materials used include the following

- Raw silk waste
- Dionized water
- Sericin extract
- Plain woven polyester fabric
- Reagent grade glutaraldehyde
- Magnesium chloride
- Acetic acid
- Analytical grade Sodium carbonate
- Sodium hydroxide
- Copper sulphate
- Sodium potassium tartarate
- Bovine serum albumin
- Folin Ciocalteau's phenol reagent
- Deionized water
- Lissapol N

The following equipments have been used

- Infra red dyeing machine
- Xenon excimer UV lamp
- Sericin has been applied on irradiated polyester fabrics
- (Price & Nairn, 2009)

The following procedures have been followed

The treated polyester fabrics have been characterized by

- SEM analysis
- Weight add on
- Dyeing with basic dyes
- Durability of sericin finish

The following moisture related properties have been determined

- Moisture regain
- Vertical wicking

The following performance properties have been determined

- Anti static properties
- Antimicrobial properties
- Antioxidant properties
- Ultra violet protection factor

All quantitative results were expressed as means  $\pm$  SD. Statistical analysis was carried out using the unpaired Student's t-test. A value of p < 0.05 was considered to be statistically significant.

## The Findings

PET is non-polar and inert in nature. Thus to apply sericin on its surface, pre-irradiation with excimer was carried out. Fabric samples were irradiated for different time duration (1, 3, 5, 7, 10, 12, 15 min) to bring about surface modification. The assessment of modification on the fabric surface was done by the following methods.

#### Surface Modification of Polyester

PET is hydrophobic and has no polar groups. The excimer lamp emits intense monochromatic light with high-energy UV photons at 172 nm capable of generating polymer chain scissions of the weakest bonds of the polyester surface, creating very reactive chain-end free radicals, which then react with the oxygen at the PET fibre surface, yielding oxidized carboxyl groups. These carboxyl groups are polar species capable of increasing the surface energy of the polyester. The increase in hydrophilicity of the PET fabric surface after the VUV excimer treatment is due to formation of polar groups when the PET surface is irradiated with the VUV excimer [26]. A second reaction, which is simultaneously occurring, is due to the presence of oxygen at the fibre surface which absorbs the high-energy photons to form highly reactive excited oxygen through the ozone cycle, which then reacts with the PET surface to form polar groups that increase surface wettability [27]. The amount of anionic groups (carboxyl and hydroxyl) generated on the fabric surface was estimated indirectly by dyeing the treated PET with a cationic dye. Results have been plotted. Untreated PET fibre has no functional group and hence has no affinity towards basic dyes which develop a positive charge in the dye bath. After treatment with excimer, carboxyl and hydroxyl groups are generated on the surface which serve as sites for attachment of cationic dye. It is because of this that while the untreated polyester remains colourless after dyeing (K/S 0.586), the sample irradiated for 15 min is dyed a deep blue shade with K/S value of 7.083.

#### **SEM Analysis**

The SEM analysis was carried out to investigate the change in morphology of excimer-treated fabric. (Figure 1(a & b)). It can be seen that while the surface of untreated PET is smooth and distinct, (Figure 1(a)), the irradiated fibre shows a roughened surface after treatment, (Figure 1(b)). Since 172 nm energy is absorbed strongly at the surface, the etching effect is restricted largely on the fibre surface. No thermal damage such as melting or bubble formation was seen in or around the exposed region. Therefore, it can be inferred that the etching process with incoherent UV radiation of excimer lamps is mainly of a photolytic nature. Similar results have been reported, with a 222 nm excimer lamp [28].

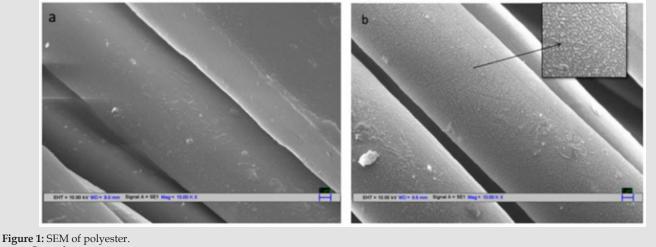


Figure 1: SEM of polyester.a. Controlb. Excimer treated (15 min)

## **Wicking Properties**

Surface etching and surface polarization of PET brought about by exposure to excimer are expected to affect the moisture transport properties of the treated fabric. Wicking properties of samples irradiated for different periods of time have been determined. During the initial irradiation period (up to 3 min), there was about 15% increase in the rise of water. On irradiation for more than 5 min, an increase of >35% in wicking properties was observed. Irradiation seems to increase the wicking height of the PET fabric considerably to about 60% after an exposure of 15 min to lamp. Improvement in capillary uptake after excimer treatment has also been reported [29]. From the above results, it could be seen that exposure of 15 min brings about maximum effect on the surface of fabric, thus this exposure time was used for all subsequent studies.

## **Application of Sericin**

Application of biopolymer sericin on VUV excimer modified PET

was carried out using glutaraldehyde (GTA) as cross-linking agent. The action mechanism is given in (Figure 2).

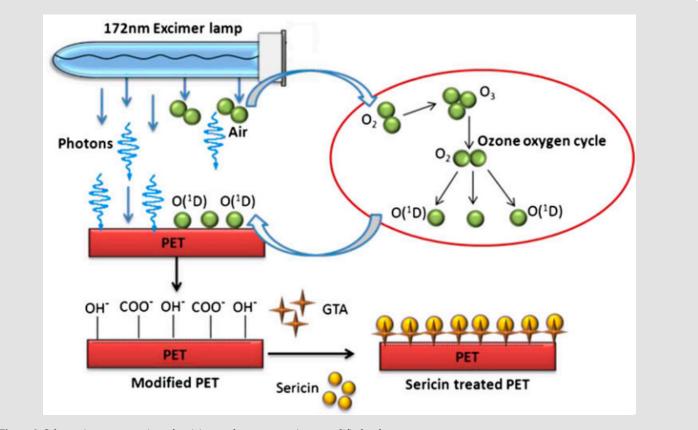


Figure 2: Schematic representation of sericin attachment on excimer modified polyester.

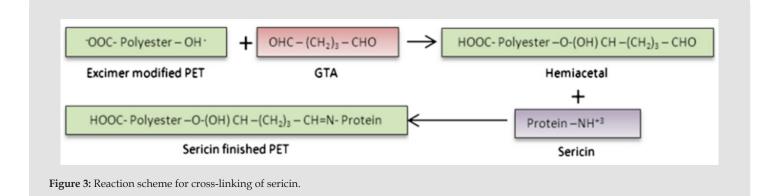
## **Concentration of Cross-Linking Agent**

The K/S values of the fabrics dyed after sericin treatment with different concentrations of GTA have been determined. It was found that the K/S value increases from 7.083 to 8.532 with increase in the concentration of GTA from 5 to 10 mL/L. The increase in dye uptake may be attributed to the amount of sericin fixed on the fabric which is proportional to the concentration of GTA. It was also observed that there was no significant increase in dye uptake beyond 10 mL/L, in fact it is probable that all free radicals have formed bonds with GTA, so the fabric is saturated at a concentration of 10 mL/L. Glutaraldehyde (GTA) gives good attachment of sericin on modified polyester 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 [30]. 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 figure 3. Similar trend was observed in case of weight add on. The results have

been determined. With least concentration of 5 mL/L, there was no increase in weight add on. However on increasing the concentration of GTA to 10 mL/L, an increase of 86% in weight add on was observed. Though no significant increase in weight add on was observed beyond 10 mL/L of GTA concentration. Curing conditions Curing conditions for cross-linking were optimized with respect to the curing temperature and time. Excimer irradiated PET fabrics was padded with a solution containing GTA (10 mL/L) and sericin (10 g/L). The modified PET fabric was cured at different temperatures (100, 110, 120, 130°C) keeping curing time as constant, 2 min. After washing, to estimate the sericin fixed onto the fabric surface, the samples were dyed with basic dyes. The results have been determined. The K/S values were found to increase by 18% with an increase in the curing temperature from 100 to 130°C. Moreover the weight add on was observed to increase from 0.13 to 1.42%. This indicates that the fixation of sericin on the fabrics increases with increase in temperature. To optimize the curing time, sericin-treated fabric was cured at 130°C for various intervals of time. K/S values increase from 7.9 to 8.5 on increasing the curing time from 1 to 2 min. However,

no significant increase in K/S values was observed beyond 2 min of curing time. Similar trend was observed in case of weight add on. The results have been determined. On the basis of these results, 10 mL/L concentration of GTA and curing at 130°C for 2 min appeared to be the best conditions for cross-linking of sericin as they yield maximum application of sericin onto PET fabric. These conditions were used for the next study to estimate the optimum concentration of sericin for application on PET. Concentration of sericin Fabrics pretreated with 172 nm excimer lamp were treated with different concentrations of sericin from 5 to 25 g/L using the cross-linking conditions optimized earlier. K/S values of fabrics treated with different concentrations of sericin and dyed with methylene blue have been determined. An increase of 5% in colour values was found on applying 5 g/L of sericin on irradiated PET. Further, an increase of 21% in colour values was calculated on increasing the sericin concentration to 10 g/L. These results indicate an increase in amount of sericin deposited on PET with increasing concentration of sericin in solution. Weight add on for various concentrations of sericin varies from 0.3% for 5 g/L of sericin to a maximum of 1.58% for 25 g/L of sericin. The results have

been determined. Significant increase in weight add on was observed at 10 g/L concentration of sericin. However, no significant increase in weight add on and colour value was observed on increasing the sericin concentration to 20 and 25 g/L of sericin. It can be inferred that fabric reached a saturation level at 10 g/L of sericin concentration beyond which no more sericin could be applied. Thus for all further work, concentration of sericin liquor was kept constant at 10 g/L. Properties of sericin finished fabrics Fabrics treated with 10 g/L of sericin, 10 mL/L of GTA, cured at 130°C for 2 min were tested for various performance parameters relevant to medical and sports textiles. Fabrics suitable for medical applications are expected to meet a few criteria such as good absorbency, creating a moist environment, non-toxic, non-allergenic, antibacterial, antistat and possessing sufficient mechanical integrity for handling. Keeping these in mind, sericin-treated fabrics were tested for various performance properties. Durability to washing of sericin finish applied on PET was tested. There was no change in the colour value of samples exposed to three launderings indicating that the finish is durable to washing.



#### **Moisture Related Properties**

Excessive transepidermal water loss is one of the causes of dry skin which results in generation of frictional forces between fabric and skin underneath. Sericin has resemblance with natural moisturizing factor which is naturally a part of our human makeup. Serine which is an important moisturizing amino acid has a high concentration in sericin, thus attributing moistening properties to it. Moisture-related properties of sericintreated fabric were studied as they play an important role in fabrics that are in direct contact with the dermal layer especially for patients suffering from skin diseases [17]. Moisture regain Moisture regain is the tendency of most fibres to pick up or give off ambient atmospheric moisture until they reach an equilibrium moisture content at a given temperature and humidity level. Virgin PET has a moisture content of  $0.60 \pm 0.10\%$  as it has a low degree of amorphous regions and a high degree of crystalline region. On irradiation with excimer, the moisture content increases up to  $1.02 \pm 0.05\%$  due to generation of polar moities on fabric surface.

There is a further increment in the moisture content on application of sericin. Sericin concentration of 10 g/L gives a moisture content of  $2.36 \pm 0.04\%$ . This effect can be attributed to the formation of uniform coating of sericin on the fabric surface, as seen in SEM pictures, Figure 4. Since sericin is composed of 80% amino acids that contain hydrophilic groups such as serine, aspartate and glycine it can absorb moisture very well. Vertical wicking Surface energy of both fabric and liquid influence the wicking characteristics of a fabric. The vertical wicking height depends on the balance between capillary force of fibres and the weight of the liquid in fibres. Therefore, the higher rising value of liquid means higher surface energy and stronger capillary forces of fabric [31]. Results of vertical wicking test have been determined. Wicking height values of untreated PET fabric was found to be about 7.8 cm, while irradiated and sericin-treated sample (ExTS) showed a height rise of 12.8 cm in 10 min. On application of sericin there was almost 64% rise in height of water as compared to untreated PET. Antistatic property The problem of static charge

generation is particularly common in hydrophobic fibres such as PET and limits their use in medical and other functional applications. It is extremely uncomfortable especially for patients with skin problems. Static behavior of treated PET was examined by qualitative analysis using the ash test. Results are shown in (Figure 5(a & b)). The amount of ash transferred to untreated PET fabric (Figure 5(a)) is much higher than that on treated PET fabric (Figure 5(b)). This can be attributed to the improved hydrophilicity of the fabric surface. Antimicrobial property The results of agar diffusion test against the standard test organism E. coli (Gram negative) have been determined. No zone of inhibition around the control fabric was observed thus indicating an absence of antimicrobial activity. Similar results were seen in fabrics treated with sericin against the test organisms. Several papers report the antimicrobial property of sericin but in this study no activity was observed [12,32]. This could be because the antimicrobial activity of sericin is primarily due to the presence of a compound called 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 [33]. Free RSA Oxygen-centred free radicals and other reactive oxygen species (ROS) can be generated as by-products during oxidative progresses of living organisms. Many human diseases, including accelerated ageing, cancer, cardiovascular disease, neurodegenerative disease and inflammation, are linked to excessive amounts of free radicals [34]. The antioxidants are necessary to cure these diseases [35]. Antioxidant agents, particularly those from natural sources are much in demand since they function as free-radical scavengers and chain breakers. RSA of sericin-treated PET was found to be 56% higher than excimer-treated PET. Results are shown in (Table 1). As a natural protein, silk sericin has functional groups like cysteine, tyrosine and histidine with flavonoids which contain electron donors [36]. These donors react with free radicals and convert them to more stable products and terminate the radical chain reaction. Because of this unique property of sericin, it is employed in cosmetics as an antiageing agent.

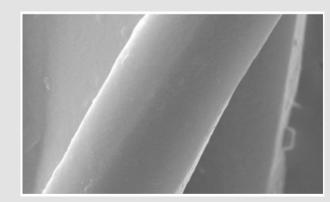


Figure 4: SEM of sericin treated PET fibre.

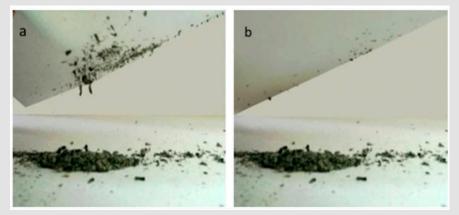


Figure 5: Ash test for assessment of antistatic property. a. Control

b. Excimer and 10 gpl of sericin

#### **Ultraviolet Protection Factor**

Impact of the UV rays on various living organisms, especially humans and the relationship between skin cancer and UV dosage is well correlated. UV radiation can generate ROS, which influences skin pigmentation [37]. Excessive UV radiation leads to cell damage and causes inflammation of human skin, the obvious consequences of which are erythema or sunburn. The degree to which a fabric protects the skin from UV radiations is given as its UPF. PET has an inherent protection to ultraviolet radiations because of aromatic rings in its structure which absorb UV light. It can be seen from the (Table 1), that the UPF rating was observed to increase from  $55 \pm 5.57$  to 125 ± 6.37 on application of sericin. Sericin acts as a UV absorbers thus converting electronic excitation energy into thermal energy. The high-energy, short-wavelength ultraviolet radiation excites the UV absorber to a higher energy state; the energy absorbed may then be dissipated as longer-wavelength radiation thus avoiding skin degradation [38]. In earlier reported studies, sericin has been found to exert a photo protective effect against ultraviolet-B-induced tumour promotion and damage to the skin [16,39].

Table 1: RSA and UPF rating of test samples.

	RSA (%)	UPF rating
Control	0	$55 \pm 5.57$
Excimer Treated	$18.18 \pm 2.02$	$90 \pm 4.88$
Excimer and 10 gpl sericin	41.68 ±4.33	$125 \pm 6.37$

## Conclusion

The results of this study show that durable sericin-based finish can be applied on excimer modified polyester using glutaraldehyde as cross-linking agent. The sericincoated fabric showed enhanced wicking and moisture regain property making the fabric suitable for direct contact with the dermal layer especially for patients suffering from skin diseases. The treated fabric was not found to have antimicrobial property. Simultaneously, an improvement in antistat, ROS scavenging and UV absorption properties was observed making it suitable for applications in skin moisturizing, cell healing and antiageing. Hence, it can be concluded that fabric based on sericin could be developed for dermatological and medical purposes.

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