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Cotton Fabrics Finished by Natural and Sulfated β-Cyclodextrin Inclusion Complexes of Silver Nanoparticles for Biomedical Applications

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ABSTRACT

In this study, it was aimed to compare the properties of cotton fabrics finished by natural (neat, pure) and sulfated β-cyclodextrin complexes of silver nanoparticles (AgNPs) for biomedical applications. For this aim, sulfated β-cyclodextrin (S-β-CD) was obtained from β-CD and they were applied to cotton fabrics with and without EDTA crosslinking agent. Then, all the fabrics were treated with AgNPs and inclusion complexes were formed. Within the scope of the study, antibacterial activity, washing stability, add-on, tensile strength, handle and color change of the samples were tested and compared to each other. In addition, SEM and EDX were carried out on the samples to characterize the effects of finishing, FT-IR analysis was carried out to characterize the chemical structures of β-CD and S- β-CD powders and XRD analysis was carried out to characterize the AgNPs. As a result of the study, the treatment of S-β-CD complex with AgNPs and crosslinking this complex to cotton sample by means of EDTA was found to be the most favorable method.

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1. INTRODUCTION

Textile products have long been known as susceptible media that support the growth and reproduction of microorganisms such as bacteria and fungi. These microorganisms are almost everywhere in the environment and can multiply rapidly when basic requirements such as moisture, nutrients and temperature are met [1-8]. Therefore, it is of great importance that the fabrics form a protective shield and protect them from all kinds of microbial attacks [2, 4, 6-10]. There are many antibacterial agents that kill or inhibit the growth of microorganisms. Due to being biocompatible and having a strong antibacterial effect and high thermal stability, silver is generally preferred in textile products over other antibacterial agents [2, 3, 7, 10-17]. The antibacterial property of silver is directly related to the amount and the ability of the silver released to inactivate target bacteria and fungal cells. Ionized silver is highly effective in antibacterial sense since it binds to tissue proteins, creates structural deformations in the bacterial cell wall and cell membrane through ionic interaction, and causes cell degradation and even death [8, 9, 15, 17-21]. Although the silver has considerable antibacterial activity, in order to be durable to washing and longly effective, silver particles should have high bonding performance and controlled release mechanism on the surface of the textile materials [6, 8, 17, 21]. For this aim, alternative methods like surface modification of textile material [6, 7, 22], nanocomposite formation [15, 21, 23-25], crosslinking [6, 16, 26, 27], capsulation [11, 28] and complexation [12, 14, 17, 29, 30] have been suggested. On the other hand, it has been reported that when silver is used as nanoparticles, it gives some extra advantages like good and uniform dispersion without agglomeration on the textile surface and high affinity and interaction with microorganisms thanks to their large surface area to volume ratio and high surface energy [18, 19, 27, 30-32]. However, against the studies that present the advantages of silver nanoparticles, some studies point out the

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possible toxic and irritating effect of silver nanoparticles on human health. These studies suggest alternative methods that can be useful to decrease the toxicity problem of silver towards human cells by increasing biocompatibility, bioavailability, weak antigenicity and biodegradability and by controlling the silver releasing properties. For instance, using green synthesized silver nanoparticles [4, 7, 10, 19, 28, 31-35] and/or combination with biopolymers like cellulose, chitosan, collagen, sericin and cyclodextrin have come into prominence in recent studies [24, 28, 35-37].

Cyclodextrins are obtained by enzymatic degradation of starch that results in a mixture of α-, β-, γ-cyclodextrins and acyclic dextrins [38-46]. Cyclodextrins seem to be a truncated cone and have a ring structure. There are three types of cyclodextrins based on the number of glycopyranose bonds as alpha (α) cyclodextrin, beta (β) cyclodextrin and gamma (γ) cyclodextrin. Each cyclodextrin unit has a cavity that can act as a host for a guest [41-45, 47, 48]. Even though β-cyclodextrin (β-CD) is the least water soluble, due to the simple production method, medium cavity diameter and low price, it is the most commercially produced type of the three natural cyclodextrins [39, 42-44, 49]. Cyclodextrins can form an inclusion complex (guest-host structure) with aromatic compounds or various antibacterial agents thanks to Van der Waals forces, hydrogen bonding and hydrophobic interactions [38, 39, 42-46]. On the other hand, natural cyclodextrins for instance β-CDs have limited solubility in most commonly used solvents and water since the hydroxyl groups in the cyclodextrin molecule form hydrogen bonds with each other [39, 42, 47]. So, in order to increase the solubility of natural cyclodextrins and the interaction of cyclodextrins with the guest molecule, derivatization possibilities of cyclodextrins have been studied. In derivatization reactions, some groups have been connected to the cyclodextrin structure and by this way, functional properties like solubility, stability and reactivity can be changed [50]. In addition, more controlled and durable inclusion complexes against physical abrasion and washing can be obtained [38, 39, 45, 47, 49, 51]. By inserting guest molecules into the cyclodextrin spaces attached to the fiber surface, the properties of the textile material can be improved. For instance, by this way, the finished textile material turns into a UV protective, insect repellent, antibacterial effective, bad odor catching or fragrance releasing material [40-42, 44, 45, 47, 48, 52]. Moreover, they possess controlled release mechanism that can be longly durable [42, 45, 47, 49].

There are many studies about silver nanoparticles that give considerable antibacterial activity to textile materials and formation of inclusion complexes between cyclodextrin and silver nanoparticles. George et al. [11] synthesized silver nanoparticles and encapsulated them into the β-cyclodextrin and found efficient antibacterial activity against *P.aeruginosa, S. aureus, S. marcescens, E. coli* and *K.pneumoniae*. Jaiswal et al. [12] formed an inclusion complex between the silver nanoparticles (AgNPs) and β-cyclodextrins and the antibacterial activities of the complexes against *E. coli, P. aeruginosa* and *S. aureus* bacteria were tested by agar diffusion methods. They found that the use of silver nanoparticles with β-cyclodextrin complex was more effective than the use of silver nanoparticles alone. Sathiya Priya et al. [30] formed an inclusion complex with silver nanoparticles (AgNPs) and three different β-cyclodextrin solutions at 5, 10 and 15 mM and examined the antibacterial effect of the prepared complex against *S. aureus* bacteria. They observed that the antibacterial effect of β-cyclodextrin complexed with silver nanoparticles was higher than the non-complexed silver nanoparticles. In addition, it was observed that this antibacterial activity increased as the concentration of β-cyclodextrin increased. Hedayati et al. [23] synthesized silver nanoparticles on β-cyclodextrin/ ketoconazole composite and then loaded them on cotton fabric by using a crosslinking agent. This treatment showed tremendous antimicrobial activities without cytotoxicity effects and drug release regularity with excellent washing durability. But, the use of cyclodextrin derivatives and the comparison of the effects of natural cyclodextrin and derivative cyclodextrin on textiles have not been studied in detail. Popescu et al. [20] synthesized monochlorotriazine-β-cyclodextrin derivative using compounds with multiple functions and achieved four simultaneous effects as large wrinkle recovery angle (WRA), hydrophilicity, antibacterial capacity and a good breaking resistance on cotton. Aubert-Viard et al. [53] designed a wound dressing consisting of methylated β-cyclodextrin and chitosan molecules and found an effective antibacterial activity against gram positive (*S. aureus*) and gram negative (*E. coli*) bacteria for several weeks. Popescu et al. [29] formed a complex with monochlorotriazine-β-cyclodextrin (MCT- β-CD) with silver nanoparticles and enabled these complexes to be transferred to cellulosic fabric. They observed that fabrics treated with silver nanoparticles complexed with MCT-β-CD had strong antibacterial activity against *E. coli* and *S. aureus* bacteria. Selvam et al. [54] obtained sulfated-β-cyclodextrin as a result of the chemical treatment of β-cyclodextrin with sulfuric acid. Then sulfated-β- cyclodextrin was complexed with different metal nanoparticles and bound to cotton fabric with crosslinker, EDTA. They reported sulfated-β-cyclodextrin was important for biomedical applications in textile but they did not investigate the physical properties of cotton in detail.

In this study, silver nanoparticles (AgNPs) were used as an antibacterial agent and inclusion complexes of β-cyclodextrin and its derivative (sulfated β-cyclodextrin) with silver nanoparticles were formed to reduce the toxic effects of AgNPs and applied to cotton fabrics with and without crosslinking chemical (EDTA) by pad-dry-cure method. EDTA was selected due to its chelating ability towards metal ions and binding ability towards cyclodextrins. Due to the bifunctional effect of EDTA, the

adsorption capacity of AgNPs to the cyclodextrins and the binding ability of cyclodextrins to the cotton surfaces via crosslinking reaction were concurrently enhanced. In order to compare the effects of the treatments, antibacterial tests, physical tests and characterization tests were applied in this study and the results were discussed in detail distinctively from the preceding studies.

2. MATERIAL AND METHOD

2.1 Materials

Desized and bleached woven 100% cotton fabric with a weight of 245 g/m^2 was used in the study. Warp yarn density was 45 threads/cm and weft yarn density was 20 threads/cm. The properties and supplier information of the chemicals used in the study are given in Table 1.

Table 1 The properties and supplier information of the chemicals

Chemicals	Properties and supplier information
β-cyclodextrin	1135 g/mole, Roquette (Kleptose)
Sulfuric Acid (H_2SO_4)	95% - 98%, Tekkim
Calcium Carbonate ($CaCO_3$)	98%, 100.09 g/mole, Aromel Chemistry
Ethyl Alcohol (C_2H_5OH)	96%, Merck
Silver Nanoparticles (AgNPs)	99.99%, Nanografi
EDTA (Titriplex III)	292.25 g/mole, Emir Chemistry

2.2 Methods

2.2.1 Preparation of β-cyclodextrin Derivative (Sulfated β-cyclodextrin)

The β-cyclodextrin derivative (sulfated β-cyclodextrin) was prepared in accordance with the method specified by preceding studies [54, 55]. Firstly, 10 g of β-cyclodextrin and 3 mL of sulfuric acid mixture was kept at 0-5 °C for 2 h in an ice bath. Then, 50 g/L calcium carbonate solution was added to this mixture and filtered. After filtering, 95% ethanol was added to the particles and kept at 0-5 °C overnight. The ethanol in the solution was evaporated and the particles were dried in the oven at 110 °C for about 45 min.

2.2.2 Finishing Treatments

Five different finishing treatments were studied and four of these were carried out in two steps. These treatments are application of β-cyclodextrin, sulfated β-cyclodextrin with and without crosslinking agent EDTA in the first step and application of silver nanoparticles (AgNPs) to form inclusion complexes on the cotton surface in the second step. On the other hand, only in Treatment 1 that was used as a reference, silver nanoparticles were applied to the cotton surface alone in other words without complexation in one step. The details of the finishing treatment recipes are given in Table 2.

Then the cotton samples were dipped into these solutions for 15 min and padded by a 90% wet pick-up in the laboratory type padding machine (Prowhite/Y002). After padding, they were dried at 80 °C for 5 min and cured at 140 °C for 3 min by the laboratory type stenter machine (Prowhite/Y003). In the second step, pad-dry-cure processes were repeated in the same conditions. A schematic representation of the applied mechanism is given in Figure 1.

Table 2 The details of the finishing treatment recipes

Finishing treatments	First step	Second step
Treatment 1	50 mg/L AgNPs	-
Treatment 2	50 g/L β-cyclodextrin	50 mg/L AgNPs
Treatment 3	50 g/L Sulfated β-cyclodextrin	50 mg/L AgNPs
Treatment 4	50 g/L β-cyclodextrin + 5 g/L EDTA	50 mg/L AgNPs
Treatment 5	50 g/L Sulfated β-cyclodextrin + 5 g/L EDTA	50 mg/L AgNPs

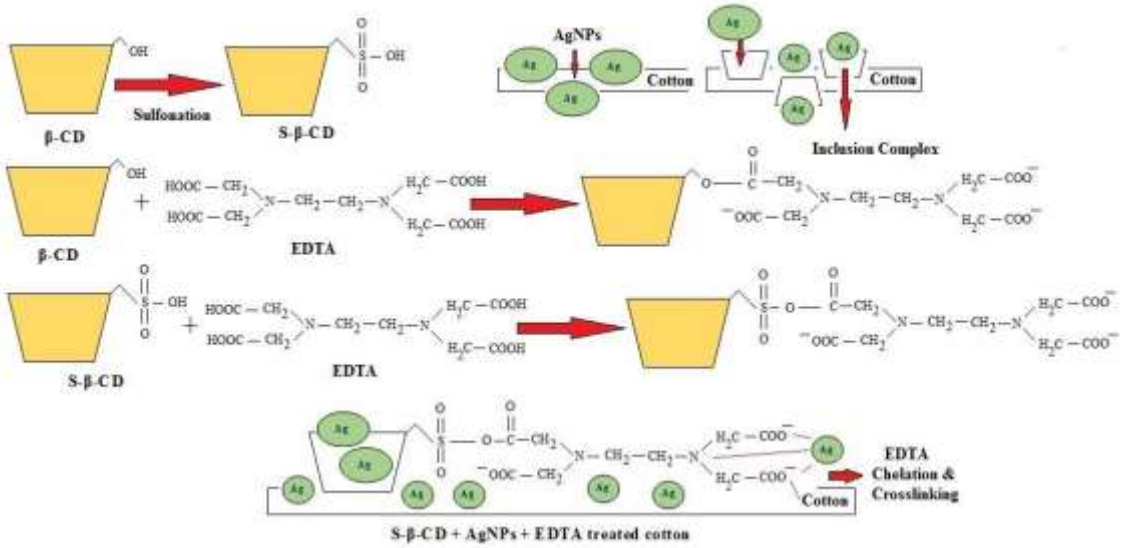

Figure 1. A schematic representation of the applied mechanism

2.2.3 Washing Processes

In order to test the washing stability of the antibacterial treatment, all the samples were washed once with a 5 g/L granular soap at 60°C for 30 min. The washing processes were carried out in accordance with the ISO 105 C03 standard (test method for textile fastness to washing) by SDL ATLAS/M228 machine. Then, the unwashed and washed samples were sent to antibacterial test. From the antibacterial test results, it was understood that these washing conditions were quite harsh compared to household washing conditions. Therefore, the washing standard was changed in the second part of the study and the most antibacterial effective sample was washed at household washing conditions as 40 °C for 1, 5 and 10 times. In accordance with ISO 105 C01 standard (another test method for textile fastness to washing), the washing processes were carried out by SDL ATLAS/M228 machine for 30 min.

2.2.4 Antibacterial Activity

The antibacterial activity tests of the samples against *Staphylococcus aureus* (ATCC 29213, gram-positive bacteria) and *Escherichia coli* (ATCC 25922, gram-negative bacteria) were carried out quantitatively according to the test method for antibacterial finishes on textile materials (AATCC 100). The reduction (%) in the number of living microorganism was calculated from the number of living microorganism present in the medium containing sample according to the Eq. 1.

$$Reduction\ (\%) = \left[\frac{A-B}{A}\right] x\ 100 \quad (1)$$

where A is the number of living microorganism (CFU/mL, colony forming units per milliliter) of control sample after 24h and B is the number of living microorganism (CFU/mL) of treated sample after 24h.

2.2.5 Physical Tests

Add-on calculation

The add-on values of the treated cotton samples were calculated according to Eq. 2.

$$W_{add-on}\ (\%) = \left[\frac{W_2-W_1}{W_1}\right] x\ 100 \quad (2)$$

where W1 is the weight of the untreated fabric sample and W2 is the weight of the treated fabric samples. Each sample was measured three times and average values were calculated.

Tensile strength measurement

The tensile strength and elongation at break values of the samples were measured in SDL Atlas H10KT test machine, according to the strip method of tensile properties of fabrics, TS EN ISO 13934-1. Each sample was measured three times and average values were calculated.

Bending length and bending rigidity measurement

The bending length of the fabric samples were measured by SDL Atlas/M003B in order to determine the handle properties according to the stiffness determination of woven textiles, TS 1409. Each sample was measured three times and average values were calculated. Bending rigidity was calculated according to Eq. 3.

$$G = 0.1\ x\ W\ x\ L^3 \quad (3)$$

where G is the bending rigidity, W is the fabric weight and L is the bending length. General bending rigidity was calculated according to Eq. 4.

$$G_0 = [G_{we}\ x\ G_{wa}]^{1/2} \quad (4)$$

where G0 is the general bending rigidity, Gwe is the average bending rigidity in weft direction and Gwa is the average bending rigidity in warp direction.

Stiffness measurement

The stiffness of the fabric samples were also measured by Prowhite Pneumatic Stiffness Tester in order to determine the handle properties according to the standard test method for stiffness of fabric by the circular bend procedure, ASTM D4032 as an alternative to bending length and bending rigidity measurement. Each sample was measured four times and average values were calculated.

Whiteness and yellowness index measurement

Whiteness and yellowness index values of the fabric samples were measured by DataColor 600TM Spectrophotometer and the results were given as Stensby whiteness index and E313 yellowness index values, respectively. Each sample was measured five times and average values were calculated.

2.2.6 Characterization Analyses

Fourier transform infrared spectroscopy (FT-IR) analysis

Fourier transform infrared spectroscopy (FT-IR) analyses of β-CD and sulfated β-CD powders were carried out by Thermo Nicolet iS50 with a diamond universal ATR (Attenuated Total Reflectance) accessory in ATR mode. The diamond crystal giving an effective depth of penetration of 1 micron and at a resolution of 4 cm^{-1} was used. The measurement was carried out in the region from 4000 to 400 cm^{-1} and recorded spectrum for each sample was the average of 4 scans.

Scanning electron microscopy (SEM) analysis

The surface morphologies of the cotton fabrics were examined by using Scanning Electron Microscope (SEM) in Zeiss Supra 40VP device with x5000 magnification range. Before the analysis, the fabric samples were coated

with gold and palladium (Au + Pd) for 6 min by Quorum Q150R ES device to increase the conductivity of the fabrics.

Energy dispersive X-ray (EDX) analysis

Energy dispersive X-ray analysis (EDX) of treated and untreated fabric samples were also carried out in Zeiss Supra 40VP device and the atomic percentage (%) values of silver nanoparticles deposited on the surface of the samples were detected for each treatment.

X-ray diffraction (XRD) analysis

The X-ray diffraction (XRD) analysis of the silver nanoparticles was recorded using GNR APD PRO 2000 X-ray diffractometer (CuKα) radiation, (λ = 1.54059 A°), the diffraction peaks were obtained in 2θ degree angle, ranging from 30° to 80°. Crystallite size of the nanoparticles was calculated using full width at half maximum (FWHM) of the 100% peak of nanoparticles in Scherrer's equation (Eq. 5),

$$D = \frac{k\lambda}{\beta Cos\,\theta} \quad (5)$$

where D is the mean particle size (nm), λ is X-ray wavelength (λ = 1.54059 A°), β is FWHM of the diffraction line, θ is diffraction angle, and k is constant which assumed as 0.94.

3. RESULTS AND DISCUSSION

3.1 Characterization Analyses Results

3.1.1 Fourier Transform Infrared Spectroscopy (FT-IR) Analysis Results

The spectra of β-cyclodextrin powder and sulfated β-cyclodextrin powder are given in Figure 2.

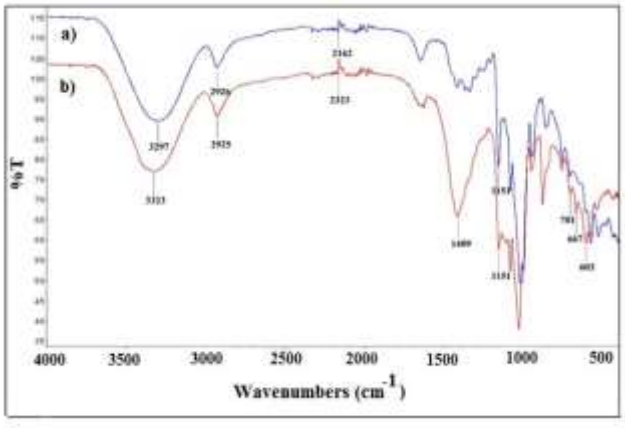

Figure 2. FT-IR spectra of β-CD and S-β-CD powders **a)** β-CD powder, **b)** S-β-CD powder

According to the FTIR spectra, the main groups were the same both for β-CD and S-β-CD powders. These bonds were absorption bands at 3323 and 3297 cm^{-1} (–OH bonds), the bands at 2926, 2925, 2323 and 2162 cm^{-1} (C-H and CH2 aliphatic bonds) and the bands at 1151 cm^{-1} (C-O-C bonds). However, S-O (sulfur-oxygen) bond was observed on the sulfated β-cyclodextrin powder at 667 cm^{-1}. In addition, small changes between 1151 and 1000 cm^{-1} were seen due to the asymmetric and symmetric stretching of SO3 groups. So, these changes indicated the reaction of sulfonic acid on β-CD. These results confirmed the related studies [1, 14, 54, 55] and also revealed the difference between the chemical structures of β-cyclodextrin and sulfated β-cyclodextrin.

3.1.2 Scanning Electron Microscopy (SEM) Analysis Results

SEM images of the treated samples and untreated sample are given in Figure 3.

According to the SEM images, the surface of the untreated cotton sample (Fig. 3a) was smooth and clear as expected while the other surfaces included the residues of the silver nanoparticles. When the SEM image of the sample treated by silver nanoparticles (AgNPs) alone was examined (Fig. 3b), it was found that silver nanoparticles were less adhered to the surface of the samples than the samples treated by β-cyclodextrin and its derivative. When the sample surfaces treated by β-cyclodextrin and silver nanoparticles (AgNPs) (Fig. 3c) were examined, it was observed that silver nanoparticles did not show uniform distribution and formed aggregates in certain areas. When the sample surfaces treated by sulfated β-cyclodextrin and silver nanoparticles (AgNPs) were examined (Fig. 3d), it was observed that silver nanoparticles showed a uniform distribution and the quantity of the inclusion complexes formed on the surface increased. Moreover, the application of EDTA crosslinking agent also ensured a strong adhesion of inclusion complexes to surfaces and supported the increase in the quantity of the inclusion complexes and AgNPs (Fig. 3e and 3f). On the other hand, when Fig. 3e and 3f were compared, it was also clearly seen that the sulfonation of β-CD prevented aggregation.

3.1.3 Energy Dispersive X-Ray (EDX) Analysis Results

By the EDX analysis, the atomic percentage of the elements were determined. As mentioned in the methods part, the samples were coated with a thin gold/palladium film layer to increase the conductivity during SEM and EDX analysis. Therefore, in order to calculate the accurate atomic percentages of the elements, the atomic percentages of gold and palladium elements related to the coating in the analysis were eliminated and were remained as unidentified peaks in the images in Figure 4.

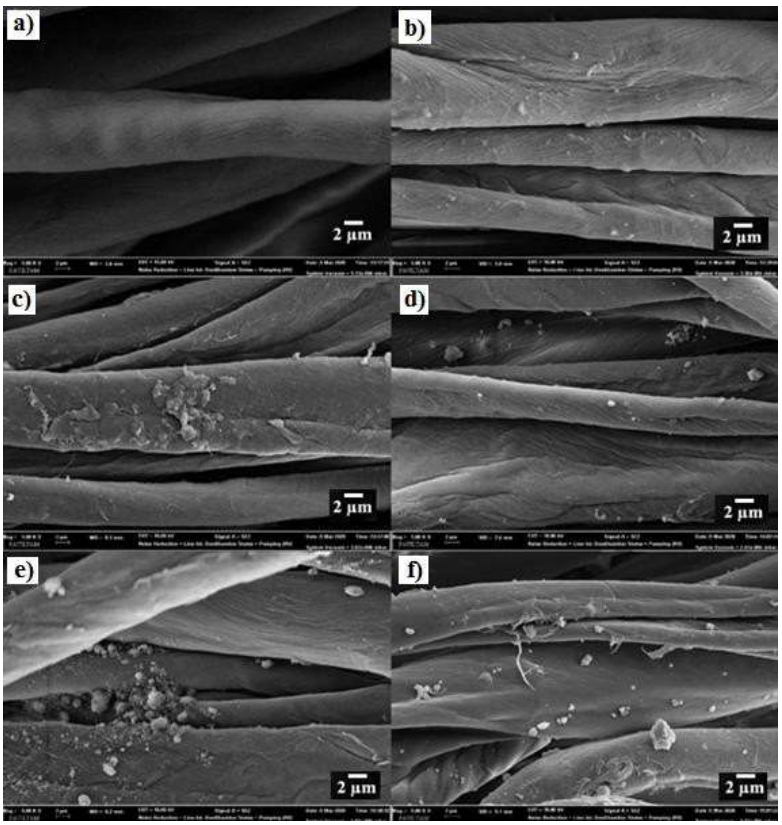

Figure 3. SEM images of the samples **a)** untreated sample **b)** sample treated by Treatment 1 (AgNPs) **c)** sample treated by Treatment 2 (β-CD + AgNPs) **d)** sample treated by Treatment 3 (S-β-CD + AgNPs) **e)** sample treated by Treatment 4 (β-CD + AgNPs + EDTA) **f)** sample treated by Treatment 5 (S-β-CD + AgNPs + EDTA)

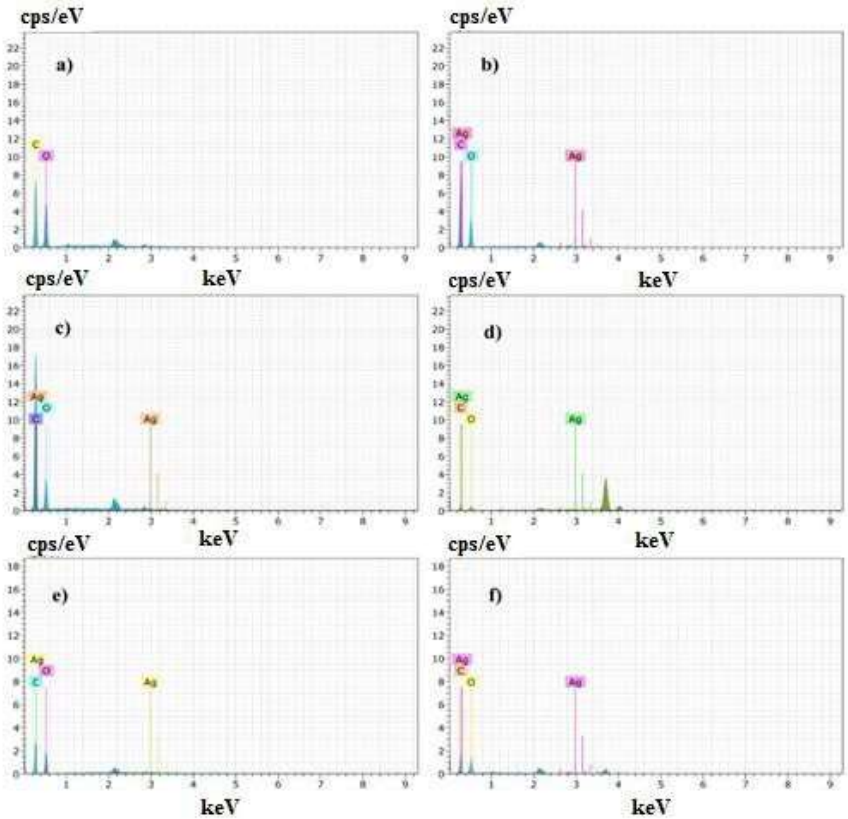

Figure 4. EDX spectra of the samples **a)** Untreated sample **b)** sample treated by Treatment 1 (AgNPs) **c)** sample treated by Treatment 2 (β-CD + AgNPs) **d)** sample treated by Treatment 3 (S-β-CD + AgNPs) **e)** sample treated by Treatment 4 (β-CD + AgNPs + EDTA) **f)** sample treated by Treatment 5 (S-β-CD + AgNPs + EDTA)

In untreated sample, only C and O elements were detected as a characteristic elements of cotton. After treatments, the atomic percentage of C element decreased and the atomic percentage of O element increased. In addition, the atomic percentage of Ag element were also detected depending on the treatment formulations. The atomic percentage values of the Ag element in the treated samples were found as 0.38, 0.63, 4.42, 1.01 and 8.67 respectively. So, the amount of AgNPs on the sample treated by S-β-CD was much higher than the sample treated by β-CD. It was also observed that the samples treated by EDTA crosslinking agent possessed higher Ag amount than the others. It was observed that EDTA bound more silver nanoparticles (AgNPs) and inclusion complexes on the surface. According to these results, it was concluded that the sample with the highest quantity of silver nanoparticles was the sample treated by Treatment 5, in other words by S-β-CD + AgNPs + EDTA.

3.1.4 X-Ray Diffraction (XRD) Analysis Results

XRD spectra of the silver nanoparticles are given in Figure 5.

XRD analysis was used to characterize the crystalline nature of the silver nanoparticles. Silver nanoparticles showed diffraction peaks at 2θ in the planes of (111), (200), (220) and (311) respectively. These planes were characteristics of the face-centered cubic silver crystals. The planes were indexed with lattice constants a=4.077 Å and matched to the ICDD (International Centre for Diffraction Data) card no. 01-087-0720. In addition, similar Bragg's reflections confirmed the silver nanoparticles as reported in the related studies [4, 10, 14, 24, 27, 33]. Then, the mean particle size (D) of the silver nanoparticles was calculated with Scherrer equation and found as 31 nm.

3.2 Antibacterial Activity Test Results

The antibacterial activity results of the treated fabrics against *S. aureus* are shown in Table 3.

The antibacterial activity results of the treated fabrics against *E. coli* bacteria are shown in Table 4.

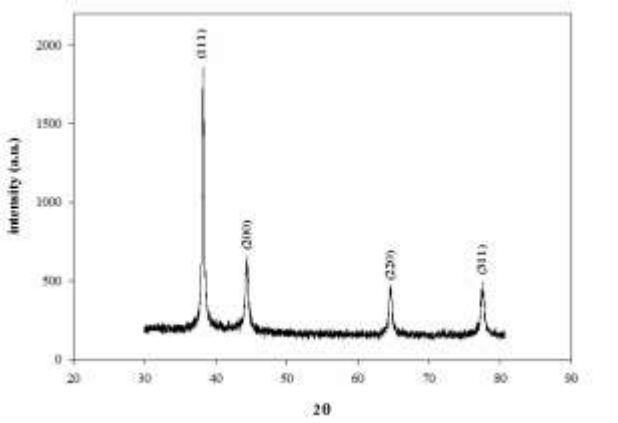

Figure 5 XRD spectra of the silver nanoparticles

Table 3 The antibacterial activity results of the treated fabrics against *S. aureus*

Treatment	Bacterial count before washing		Bacterial count after washing at 60 °C		Bacterial reduction (%)	
	Control sample after 24h	Treated sample after 24h	Control sample after 24h	Treated sample after 24h	Before washing	After washing
Treatment 1 (AgNPs)	560.000	290.000	560.000	400.000	**48.21**	**28.57**
Treatment 2 (β-CD + AgNPs)	560.000	260.000	560.000	310.000	**53.57**	**44.64**
Treatment 3 (S-β-CD + AgNPs)	560.000	38.000	560.000	280.000	**93.21**	**50.00**
Treatment 4 (β-CD + AgNPs + EDTA)	560.000	188.000	560.000	263.000	**66.43**	**53.04**
Treatment 5 (S-β-CD + AgNPs + EDTA)	560.000	28.000	560.000	220.000	**95.00**	**60.71**

Table 4 The antibacterial activity results of the treated fabrics against *E. coli*

Treatment	Bacterial count before washing		Bacterial count after washing at 60 °C		Bacterial reduction (%)	
	Control sample after 24h	Treated sample after 24h	Control sample after 24h	Treated sample after 24h	Before washing	After washing
Treatment 1 (AgNPs)	590.000	390.000	590.000	560.000	**33.90**	**5.08**
Treatment 2 (β-CD + AgNPs)	590.000	250.000	590.000	530.000	**57.63**	**10.17**
Treatment 3 (S-β-CD + AgNPs)	590.000	102.000	590.000	430.000	**82.71**	**27.12**
Treatment 4 (β-CD + AgNPs + EDTA)	590.000	225.000	590.000	360.000	**61.86**	**38.98**
Treatment 5 (S-β-CD + AgNPs + EDTA)	590.000	28.000	590.000	280.000	**95.25**	**52.54**

According to the antibacterial test results, in the unwashed samples, the most effective samples were the samples treated by cyclodextrin derivative (sulfated β-CD), while in the washed samples, the most effective samples were the samples treated by crosslinking agent (EDTA). Considering these results, it was understood that S-β-CD showed better antibacterial activity than β-CD via sulfur-oxygen bonds. In other words, the modified structure of β-CD by sulfonation reaction improved the expansion on polymeric materials and the ability to form inclusion complexes with AgNPs that kill bacteria. So, since the number of inclusion complexes formed by S-β-CD was higher than the number of inclusion complexes formed by β-CD, the antibacterial activity was found to be better after derivatization. Moreover, Selvam et al. [54] reported sulfur units of S-β-CD also acted as an antibacterial agent that can positively affect the antibacterial activity. In case of EDTA, EDTA crosslinking agent ensured that the inclusion complexes and AgNPs remained attached to the fabric surface even after washing. This fact was attributed to the chelation towards AgNPs and improved crosslinking between cotton and inclusion complexes thanks to EDTA. In terms of microorganism type, the antibacterial activity against gram positive bacteria (*S. aureus*) was seen to be better than gram negative bacteria (*E. coli*). This result was attributed to the fact that gram positive bacteria have one cytoplasmic cell membrane however gram negative bacteria have two cell membranes (cytoplasmic membrane and outer membrane) as reported in the related studies [9, 26]. Zhang et al. [21]; Hebeish et al. [22]; Ru et al. [27]; and Sharma et al. [33] also reported the antibacterial activity of nanosilver-based antibacterial textiles was better against *S. aureus* than *E. coli* as similar to the results of this study. Zhang et al. [21] introduced carboxyl groups into cotton fiber by three methods, namely selective oxidation, butane tetracarboxylic acid (BTCA) grafting and polyacrylic acid (PAA) adsorption and loaded AgNPs on these cotton fibers. They reported the fabric nanocomposites prepared by selective oxidation and BTCA grafting exhibited excellent antibacterial activity (100%) even after 80 washing cycles. Hebeish et al. [22] synthesized cationized cotton, treated this cationized cotton with reactive cyclodextrin graft copolymerized with poly-acrylic acid (MCT-βCD-g-PAA) and then applied nanosilver colloids to this modified cotton. They used disc diffusion method to evaluate the antibacterial activity and they obtained inhibition zone diameters between 14 and 21 mm depending on the content of silver nanoparticles in the samples. Ru et al. [27] studied the antibacterial finishing of cotton fabric with silver nanoparticles stabilized by nanoliposomes and they reported the sterilizing rates all were above 99.34%. Sharma et al. [33] produced silver nano-coated cotton fabric and evaluated the antimicrobial activity. They reported that the antibacterial efficiency of this fabric was more than 90% after 50 laundry cycles.

In the first part of the study, the washing process was carried out at 60 °C. But in the second part of the study, the milder and widely used household type temperature (40 °C) was preferred as washing condition for repeated washings. The repeated washing cycles (1, 5 and 10) were applied to the sample treated by AgNPs, sulfated β-CD and EDTA in other words Treatment 5.

The antibacterial activity test results of the sample treated with AgNPs, sulfated β-CD and EDTA against *S. aureus* and *E. coli* before washing and after 1, 5 and 10 washing cycles at 40 °C are shown in Tables 5 and 6 respectively.

According to the results, it was seen that the sample treated by AgNPs, sulfated β-CD and EDTA could maintain its antibacterial activity as 79% against *S. aureus* and 77% against *E. coli* bacteria even after 10 washing cycles.

3.3 Physical Test Results

3.3.1 Add-on Results

The add-on results are given in Table 7.

The add-on percentage values of the samples were found to be between 0.8% and 3.7% and this difference was attributed to the concentration variation of the chemicals. So, the add-on values increased due to the increase in the total mass of the chemicals.

3.3.2 Tensile Strength Results

The tensile strength and elongation at break values in the weft and warp direction of fabrics treated with β-cyclodextrin, sulfated β-cyclodextrin, EDTA and silver nanoparticles and untreated fabrics are given in Table 8.

Table 5 The antibacterial activity of the sample treated by Treatment 5 before and after washing at 40 °C against *S. aureus*

Treatment 5 (S-β-CD + AgNPs + EDTA)	Bacterial count for control sample after 24h	Bacterial count for treated sample after 24h	Bacterial reduction (%)
Before washing	360.000	18.000	95.00
After 1 washing at 40 °C	360.000	42.000	88.33
After 5 washing at 40 °C	360.000	58.000	83.88
After 10 washing at 40 °C	360.000	74.000	79.44

Table 6 The antibacterial activity of the sample treated by Treatment 5 before and after washing at 40 °C against *E. coli*

Treatment 5 (S-β-CD + AgNPs + EDTA)	Bacterial count for control sample after 24h	Bacterial count for treated sample after 24h	Bacterial reduction (%)
Before washing	375.000	18.000	95.25
After 1 washing at 40 °C	375.000	58.000	84.53
After 5 washing at 40 °C	375.000	68.000	81.87
After 10 washing at 40 °C	375.000	85.000	77.33

Table 7 Add-on results

Treatment	Approx. weight of the sample (g)	Add-on (%)
Untreated	0.917	-
Treatment 1 AgNPs	0.925	0.80
Treatment 2 β-cyclodextrin + AgNPs	0.935	1.96
Treatment 3 S-β-cyclodextrin + AgNPs	0.937	2.18
Treatment 4 β-cyclodextrin + AgNPs + EDTA	0.940	2.50
Treatment 5 S-β-cyclodextrin + AgNPs + EDTA	0.951	3.70

Table 8 Tensile strength and elongation at break results

Treatment	Elongation at break		Tensile strength		Tensile strength increase	
	Weft (%)	Warp (%)	Weft (N)	Warp (N)	Weft (%)	Warp (%)
Untreated	23.35	19.14	591	1014	-	-
Treatment 1 AgNPs	15.77	15.94	625	1035	5.54	2.03
Treatment 2 β-cyclodextrin + AgNPs	14.67	14.97	725	1126	18.56	9.97
Treatment 3 S-β-cyclodextrin + AgNPs	15.03	15.47	720	1112	17.93	8.84
Treatment 4 β-cyclodextrin + AgNPs + EDTA	14.26	14.40	735	1159	19.67	12.51
Treatment 5 S-β-cyclodextrin + AgNPs + EDTA	14.76	15.30	728	1144	18.86	11.33

It was observed that the finishing treatments increased the tensile strength values of the fabrics. The tensile strength of the cotton fabric significantly increased in fabrics treated by β-CD and S-β-CD with AgNPs in comparison to the fabric treated by AgNPs alone. The increase in the strength of cotton fabric with β-CD and S-β-CD with AgNPs can be explained by the plasticizing effect of cyclodextrins on the cotton fiber structure. By disrupting the intermolecular and/or intramolecular H bond between cellulose chains, β-CD/S-β-CD decreased the restricted movement of cellulose chains and thus increased the tensile strength of the cotton samples as reported in the related study of Setthayanond et al. [40]. When the effects of β-CD and S-β-CD on the tensile strength were compared to each other, a slight decrease was observed in the samples treated by S-β-CD due to the sulfonation reaction. EDTA was seen to support the increase in tensile strength positively. On the other hand, a slight increase in the tensile strength was also observed in the cotton sample treated by AgNPs alone. Therefore, it was thought that nanosilver application was also effective in mechanical properties of the cotton samples. Taking the related studies into consideration, it was understood that the improved mechanical properties could be attributed to the binding of AgNPs onto the hydroxyl groups of the cellulose chains of the cotton fibers [13, 19]. When the elongation at break values were examined, it was observed that the elongation at break values of the treated fabrics were lower than the values of the untreated fabric. This was attributed to the increase in tensile strength and the decrease in the elongation at break values was found to be in line with the increase in tensile strength values.

3.3.3 Bending Length, Bending Rigidity and Stiffness Results

The bending length, bending rigidity and stiffness values of the samples are given in Table 9.

After the finishing treatments, the bending length and bending rigidity of the fabrics increased. This fact showed that the finishing processes caused stiffness in the fabric handle. In terms of the application of β-CD or sulfated β-CD, it was found that sulfated β-CD caused

more stiffness and EDTA led the stiffness to increase. However, there was no significant difference between the untreated fabric and the fabric to which AgNPs alone was applied. This fact was attributed to the variation in the inclusion complex formation on the cotton surface as in the add-on results. The stiffness values were found to be in accordance with add-on and bending rigidity results, namely the more inclusion complex formation gave rise to more stiffness. Similarly, stiffer handle in comparison to untreated cotton after MCT-β-cyclodextrin treatment was also reported by Setthayanond et al. [40].

3.3.4 Whiteness and Yellowness Index Results

The whiteness and yellowness index values are given in Table 10.

Table 9 Bending length, bending rigidity and stiffness results

Treatment	Weft		Warp		General bending rigidity (mg.cm)	Stiffness
	Bending length (cm)	Bending rigidity (mg.cm)	Bending length (cm)	Bending rigidity (mg.cm)		
Untreated	1.66	112.01	2.18	253.82	168.62	352.25
Treatment 1 / AgNPs	1.72	124.66	2.20	260.87	180.34	356.00
Treatment 2 / β-cyclodextrin + AgNPs	1.81	145.28	2.31	302.00	209.46	431.00
Treatment 3 / S-β-cyclodextrin + AgNPs	1.86	157.65	2.38	330.30	228.20	434.25
Treatment 4 / β-cyclodextrin + AgNPs + EDTA	1.90	168.05	2.46	364.73	247.57	438.00
Treatment 5 / S-β-cyclodextrin + AgNPs + EDTA	1.93	176.13	2.52	392.07	262.80	441.25

Table 10 Whiteness and yellowness index results

Treatment	Whiteness index (Stensby)	Yellowness index (E 313)
Untreated	86.09	4.59
Treatment 1 / AgNPs	85.50	5.19
Treatment 2 / β-cyclodextrin + AgNPs	84.91	5.15
Treatment 3 / S-β-cyclodextrin + AgNPs	76.69	8.86
Treatment 4 / β-cyclodextrin + AgNPs + EDTA	83.54	5.36
Treatment 5 / S-β-cyclodextrin + AgNPs + EDTA	75.13	9.75

After the finishing treatments, it was observed that the degree of whiteness decreased in all samples compared to the untreated sample. On the other hand, the degree of yellowness of all samples increased significantly especially in the samples treated by sulfated β-cyclodextrin compared to the untreated sample. This color change in the samples treated by sulfated β-cyclodextrin can be explained by the combined effect of nanosilver application, sulfonation reactions of the cyclodextrin and the curing conditions. At the curing conditions, it was thought that degradation of cyclodextrin in the presence of AgNPs, that catalyze the reaction, occurred to some extent and caused the color change. The color change (from colorless to cream yellow or to dark brown) of the cotton fabrics after nanosilver application that is related to the oxidation of cellulose and adsorption of amine group to the cellulosic chains was also reported by other studies [15, 19, 21, 27, 56]. When these studies were considered, it can be said that the values of CIE L* and WI under 60 showed brownish color. Therefore, the results of this study were in acceptable limits since the final color of the sample whose color change was maximum was cream yellow and WI value was 75.13.

4. CONCLUSION

In this study, the effects of β-cyclodextrin (β-CD) complex and derivative (S-β-CD) complex with silver nanoparticles on cotton fabric were investigated and compared. Five treatments were applied as AgNPs alone; β-CD + AgNPs; S-β-CD + AgNPs; β-CD + AgNPs + EDTA and S-β-CD + AgNPs + EDTA to cotton samples. Antibacterial test results showed that the most effective treatment was S-β-CD + AgNPs + EDTA and this treatment could be stable to 10 washing cycles as 79% against *S. aureus* bacteria and 77% against *E. coli* bacteria.

Physical test results showed that the treatments caused an increase in weight, stiffness, tensile strength and yellowness values of the samples. In terms of physical test results, relatively more changes were obtained in the samples treated by S-β-CD than the others.

FTIR analysis verified the difference in the chemical structure of the β-CD and S-β-CD. SEM analysis showed the inclusion complexes between AgNPs and β-CD or S-β-CD on the cotton surface and the fact that EDTA supported the increase in the quantity of the inclusion complexes and

AgNPs. EDX analysis detected the atomic percentages of silver nanoparticles on the samples and determined the highest quantity of silver nanoparticles was in the sample treated by S-β-CD + AgNPs + EDTA. XRD analysis confirmed the silver nanoparticles were in the cubic structure and in the average particle size of 31 nm. As a result, the treatment of S-β-CD complex with AgNPs and crosslinking this complex to cotton sample by means of EDTA was found to be the most favorable method in terms of biomedical applications.

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