



In situ synthesis of nano silver on cotton using Tollens' reagent

Majid Montazer^{a,*}, Farbod Alimohammadi^b, Ali Shamei^b, Mohammad Karim Rahimi^c

^a Textile Department, Amirkabir University of Technology, Tehran, Iran

^b Young Researchers Club, Textile Department, Tehran South Branch, Islamic Azad University, Tehran, Iran

^c Medical Sciences, Tehran North Islamic Azad University, Tehran, Iran

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ABSTRACT

This study introduces a new green method synthesis of silver nanoparticles on the cotton fabric surface through using Tollens' reagent. In this approach, silver nitrate (AgNO_3) was transformed to Ag_2O followed by an aqueous solution with ammonia; subsequently, silver nanoparticles were synthesized on the cotton fabric directly. The main objective of this research was to successfully employ the reducing and stabilizing features of cellulose to synthesize nano silver. Accordingly, the antibacterial efficiency was evaluated against two common pathogenic bacteria: *S. aureus* and *E. coli*. Additionally, the color variation on the cotton fabric and durability of the antibacterial properties on the fabric were assessed and reported. The Raman spectra, CHN elemental analysis, SEM images, XRD patterns, and EDS spectrum were employed to characterize the treated cotton fabrics. The treated fabrics demonstrated an excellent antibacterial activity against the mentioned bacteria. A slight decrease in the antibacterial feature of the cotton fabrics was observed after successive washings. However, an efficient antibacterial activity remained on the fabrics.

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1. Introduction

Applications of the significant features of silver have increased since ancient times and due to the conveniently distributed sizes and shapes as well as a large surface area, silver nanoparticles are known as desirable and effective substances in many fields (Dastjerdi & Montazer, 2010; Sung-Suh, Choi, Hah, Koo, & Bae, 2004). In order to produce highly effective nanoparticles, several synthesis methods have been used. In this regard, some common approaches such as physical processes of atomization or milling, chemical methods of chemical reduction, biological irradiation, water-in-oil microemulsions, and green synthesis methods have been utilized (Capek, 2004; Sanchez, Blanco, & Quintela, 2000; Sharma, Yngard, & Lin, 2009; Son, Youk, & Park, 2006; Vimala, Shivudu, Mohan, Sreedhar, & Raju, 2009; Zhang, Qiao, & Chen, 2007). Courrol et al. employed a photo-induced method to obtain the nanoparticles using UV LED, xenon lamp and sodium lamp. Wang, Qiao, Chena, and Ding (2005) synthesized the silver nanoparticles in a solution containing PVP in which glucose reduced the amount of silver nitrate resulting in a precipitation of nanoparticles of about 20–80 nm. Tollens' procedure, which

belongs to green synthesis methods, is of outstanding importance these days (Sarkar, Jana, Samanta, & Mostafa, 2007; Sharma et al., 2009) synthesized the silver nanoparticles through conventional Tollens' reaction and proved their antibacterial efficiency against pathogenic microorganisms. This method is based on reducing $[\text{Ag}(\text{NH}_3)_2]^+$ in an aqueous solution by aldehydes in the presence of ammonia (Peterson, Bouwman, Chen, & Deutsch, 2007; Sharma et al., 2009). Additionally, in modified reactions the aldehyde is substituted for saccharides, revealing that the smallest particles formed at the lowest ammonia concentration (Sharma et al., 2009). Also, the applications of monosaccharides and disaccharides as reducing agents have been reported (Sharma et al., 2009; Soukupova, Kvitek, Panacek, Nevecna, & Zboril, 2008).

Silver nanoparticles were used to provide anti-bacterial cellulose through immersion in a silver nitrate solution and then reduced to metallic silver nanoparticles with sodium borohydride (Maneerung, Tokura, & Rujiravanit, 2008). Furthermore, cellulose solution was used to synthesize cellulose–silver nanocomposites with AgNO_3 and ascorbic acid in N,N-dimethylacetamide, providing a good dispersion (Li et al., 2010). Also, silver nanoparticles were prepared with the fungal process and applied to the cotton fabric (Duran, Marcato, De Souza, Alves, & Esposito, 2007; El-Rafie, Mohamed, Shaheen, & Hebeish, 2010). In addition, the efficiency of different chemical forms of silver in protecting cellulose fibers against biodegradation were studied and found to provide an excellent protection against fiber biodegradation (Klemencic, Simoncic, Tomsic, & Orel, 2010). Additionally, the influence of dyeing on the

* Corresponding author at: Textile Department, Center of Excellence in Textile, Amirkabir University of Technology, Tehran, Iran. Tel.: +98 21 64542657; fax: +98 21 66400245.

E-mail address: tex5mm@aut.ac.ir (M. Montazer).

antibacterial activity of silver loaded cotton fabrics and the effect of their presence on the color change of the dyed fabrics was evaluated (Ilic et al., 2009).

In this study, the AgNO_3 was transformed to $[\text{Ag}(\text{NH}_3)_2]^+$ complex and then the nano silver particles were loaded directly on the surface of the cotton using the inherently reducing and stabilizing properties of cellulosic chains of the cotton fabric. To the best of our knowledge, this approach has not been reported in other similar articles yet.

2. Materials and methods

2.1. Materials

All chemical substances were of high purity, silver nitrate (AgNO_3), ammonia, sodium hydroxide (NaOH), sodium chloride (NaCl), and Tryptic soy agar were purchased from Merck Co. (Germany). A 100% bleached cotton fabric with a weight of 118 g/m^2 and warp and weft yarn density of 26 and 35 yarns/cm, respectively, was obtained from a local market in Tehran.

2.2. Apparatuses

A Philips X-ray diffractometer, model X Pert MPD was used to assess the crystallinity of the silver nanoparticles on the cotton fabrics. A Spectrophotometer Varian Carry 5000 was employed to obtain the UV–vis absorbance spectrum of the treated fabrics. The SEM images were taken using a scanning electronic microscopy model XL30 made by Philips Co. (Germany). The Raman spectra were employed using an Alpha Thermo Nicolet Dispersive Spectrometer instrument. The CHN elemental composition of the treated fabrics was determined by using a Heraeus CHN-O-Rapid analyzer. The UV–vis absorbance of the liquids was determined using Cecil spectrophotometer, model 9200.

2.3. Color coordinates

The three coordinates (L^* , a^* , and b^*) of CIELAB color system were obtained using a Color eye 7000 colorimeter. The CIELAB color system is widely used in the color measurement of textiles (Hu, 2008). In this system, L^* shows the lightness of the fabric and a^* and b^* indicate red-green (redder if positive; greener if negative) and yellow-blue colors (yellowier if positive; bluer if negative), respectively. The overall color difference between the untreated and the treated cotton samples can be designated by the term ΔE which was calculated based on Eq. (1) (Hu, 2008; Nazari, Montazer, Moghadam, & Anary-Abbasinejad, 2010):

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{0.5} \quad (1)$$

2.4. Synthesizing Tollens' reagent

In this study, the silver nitrate solution was used as a precursor. In addition, all of these investigations were carried out at room temperature and atmospheric pressure. First, the silver nitrate solution (0.5 M) and NaOH (0.5 M) were mixed, resulting in Ag_2O powder precipitation (Eq. (2)):



Subsequently, the precipitated Ag_2O was separated from the solution and then rinsed three times with water which had been distilled twice. The powder was dried in the ambient temperature and Ag_2O powder turned brown (Nersisyan, Lee, Son, Won, & Maeng, 2003).

Then, 2 wt% aqueous ammonium solution was prepared and added to Ag_2O powder slowly, making sure that the molar

concentration ratio of $(\text{NH}_3)_\text{aq}$ to Ag_2O was 4:1 (Guang-nian, Xue-liang, Xiao-lin, & Jian-guo, 2008). Subsequently, The solution was sonicated in an ultrasonic bath for 5 min and the brown color disappeared gradually leading to the preparation of silver–amine complex ion, $[\text{Ag}(\text{NH}_3)_2]^+$ (Tollens' reagent). This is a stable complex ion resulting from strong affinity of ammonia to Ag^+ . The ammonia concentration and the nature of the reductant play a major role in controlling the Ag nanoparticle size (Sharma et al., 2009). In order to prepare the aqueous solution of $[\text{Ag}(\text{NH}_3)_2]^+$, three diverse concentrations of Ag_2O were used including 10 ppm, 25 ppm, and 35 ppm. After the synthesizing was performed, the resulting solution was acidified with dilute acid before disposal. These precautions were made to prevent the formation of the explosive silver nitride (Vogel & Svehla, 1996).

2.5. Scouring

The cotton fabrics were scoured in a bath containing 2 g/l non-ionic detergent with L:G = 50:1 (liquor to good ratio) at 50°C for 15 min and rinsed with distilled water and finally dried at 110°C for 5 min.

2.6. Nano Ag synthesis on the cotton fabric

The cotton fabric was treated with three diverse concentrations of Ag_2O , 10 ppm, 25 ppm, and 35 ppm in order to prepare the aqueous solution of $[\text{Ag}(\text{NH}_3)_2]^+$ for different baths with L:G = 20:1. The temperature of the impregnating bath was increased to the boil at a rate of $4^\circ\text{C}/\text{min}$ for 30 min. Subsequently, the treated fabrics were extracted from the bath, washed and dried at the room temperature. This process led to the formation of different amounts of nano silver on the surface of the fabric.

2.7. Antibacterial test

Two bacteria, *Staphylococcus aureus*, American type culture collection no.6538, as Gram-positive bacteria and *Escherichia coli*, American type culture collection no.11303, as Gram-negative bacteria were used. Some colonies of each bacterium were suspended in a physiologic saline solution (NaCl 0.9% in distilled water at pH 6.5) with concentration of 0.5 McFarland. The vials of bacterial suspensions were then incubated with agitation at $37 \pm 2^\circ\text{C}$, 220 rpm for 2 h. A homogenous suspension of bacteria was prepared. Next, a serial dilution was performed in 5 steps (dilution of 1:100,000) and a concentration of about $(1.5\text{--}2) \times 10^3 \text{ CFU/mL}$ was applied for the antibacterial testing. The bacteriological culture tubes (i.e., $125 \text{ mm} \times 17 \text{ mm}$ glass tubes), containing one piece of cotton fabric ($10 \text{ mm} \times 10 \text{ mm}$) were sterilized by an autoclave device in moisturized heat (121°C , 15 lb) for 15–20 min. Subsequently, an aliquot of 1 mL bacterial suspension and 2 mL of TSB broth was added to every tube and 3 mL in each tube was detected. To ensure that any decrease in bacterial count was due to the exposure to the cotton fabrics, one control of the saline solution with TSB broth, and one control of aliquot with the untreated fabrics including the tubes containing treated cotton fabrics with the bacterial suspensions and the control tubes were incubated at 37°C for 24 h. Next, some samples of $10 \mu\text{L}$ from each tube were taken and counted by pour plate method. In this method, samples are mixed with melted agar (that decreases temperature to 45°C) and poured. The plates were incubated at 37°C for 24 h and the colonies of each plate were counted through colony counted device to compare and determine the bacteria reduction of the suspensions. The results of the numbers before and after the treatment of the cotton fabrics were used

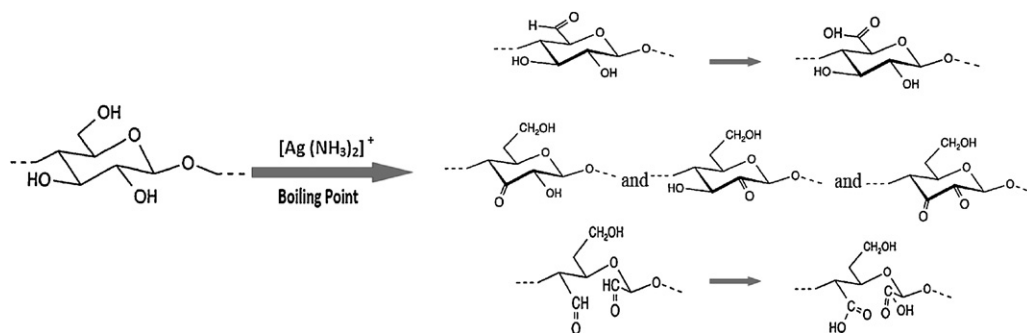


Fig. 1. Schematic structure of oxidation of cellulose by Tollens' reagent.

to determine the bactericidal effect and calculate the percentage of reduction of bacteria using the following equation:

$$\text{Reduction rate \% (R\%)} = 100 \frac{(A - B)}{A} \quad (3)$$

where R is the bacterial reduction ratio, A is the number of bacterial colonies from the untreated fabrics and B is the number of bacterial colonies from the treated fabrics. In order to increase validation of these examinations, every test tube was duplicated. The release of silver ion was determined through soaking pieces of the treated cotton fabric (10 mm × 10 mm) in a tube containing 3 mL of distilled water for 24 h and then removing them from the tube.

2.8. Washing fastness

Washing fastness test on the treated fabrics was conducted according to the AATCC 61(2A)-1996 test method. In this approach, each cycle of the washing process is equivalent to five cycles of home laundries in an ambient condition at $38 \pm 3^\circ\text{C}$. The stability of nanoparticles on the surface of the cotton was studied through antibacterial tests after 10, 20 and 30 washings in the presence of a non-ionic detergent.

3. Results and discussion

3.1. Raman spectra and CHN elemental analysis

Tollens' reagent is a mild oxidizing agent, which reduces silver and oxidizes the cellulose chain to oxycellulose. It is difficult to have both a selective and complete oxidation of a desired position. Cellulose chain has both a reducing and a non-reducing end. Reducing ends are especially reactive, but they are present in such small numbers in cellulose and are often ignored (Gordon & Hsieh, 2007). It is expected that the oxidation process of cellulose occurs on C_2 and C_3 carbons with secondary alcoholic groups, leading to a formation of ketonic groups or with a C–C bond cleavage. Moreover, there was an oxidation occurring on C_6 , as a primary alcohol, resulting in the creation of aldehyde compounds (Fig. 1) (Klemm, Philipp, Heinze, Heinze, & Wagenknecht, 1998; Morrison & Boyd, 1983). Due to the oxidation process, carboxylic groups appear which can react with ammonia. This reaction occurs between $[\text{RCOO}]^-$ and $[\text{NH}_4]^+$ group (Janardhanan, Karuppaiah, Hebalkar, & Rao, 2009; Peterson et al., 2007).

Through the Raman spectra of the treated fabrics the influence of the treatments on the cellulose configuration was investigated with three diverse concentrations (Fig. 2). Based on the results, it was observed that in the region of $3300\text{--}3600\text{ cm}^{-1}$ the main peak belongs to OH groups (Osterberg, Schmidt, & Jaaskelainen, 2006). The treatment of the fabric led to a decrease in the intensity of OH related peak; conversely, the content of the N–H group peak increased in the region of $2900\text{--}3100\text{ cm}^{-1}$ from ammonium

carboxylate (Jenkins, Almond, Atkinson, Hollinsm, & Knowles, 2005). COO^- compounds correspond to a peak near $1360\text{--}1550\text{ cm}^{-1}$ resulting from ν_{asym} and ν_{sym} (Jenkins et al., 2005). In addition, the peak of C=O compounds appeared at $1550\text{--}1650\text{ cm}^{-1}$, overlapping with that of carboxylic acids (Wang & Chen, 2008; Yuen, Choi, Philips, & Ma, 2009). It should be noted that the appeared peak at $755\text{--}800\text{ cm}^{-1}$ was attributed to the ammonium carboxylate (Sanna, Sodob, Laguzzi, Mancini, & Bicchieri, 2009).

CHN elemental analysis was applied to determine the nitrogen content of the fabric. The amounts of nitrogen were 0.2, 0.43, and 0.75 for 10, 25, and 35 ppm of nano silver, respectively. The results of this experiment confirmed the Raman data and indicated the presence of ammonium carboxylate on the surfaces of the fabric.

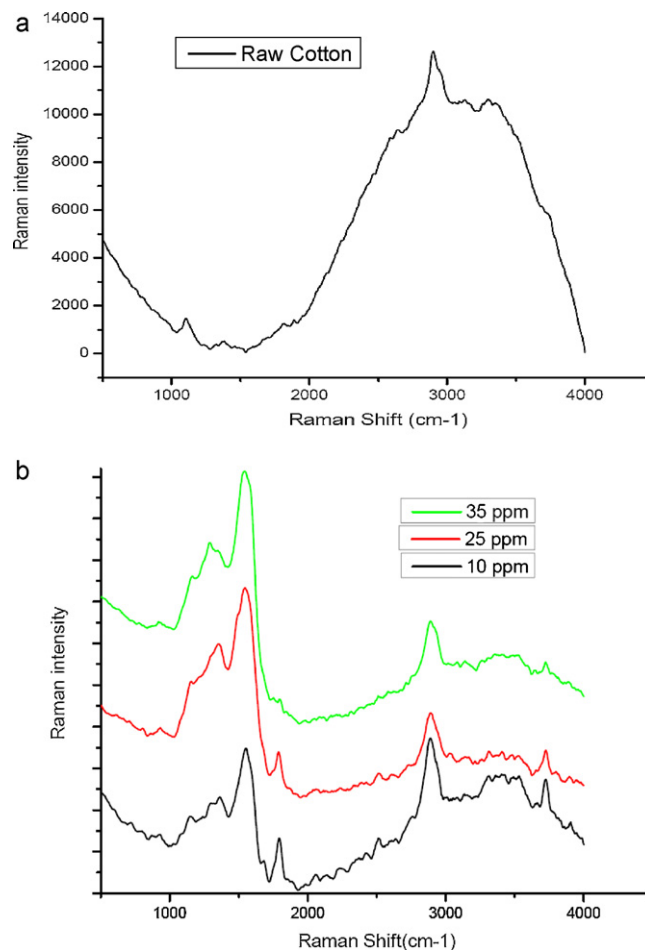


Fig. 2. Raman spectra of cotton fabrics: (a) raw and (b) treated.

Table 1

The color difference of samples containing diverse amounts of silver nanoparticles in comparison with untreated sample based on CIELAB color system.

Samples	L^*	a^*	b^*	ΔE
Untreated sample	82.44	−0.30	0.72	–
Treated with 10 ppm	80.34	−0.09	0.72	2.10
Treated with 25 ppm	77.25	1.04	12.33	12.78
Treated with 35 ppm	64.24	9.28	28.25	34.36

3.2. Color coordinates

The achieved results are proposed in Table 1 with three diverse concentrations of silver nano particles. Through increasing the amount of the silver nanoparticles on the fabrics, the ΔE increased and the color of the fabrics tuned to cream-yellow indicating the formation of the nanoparticles on the fabric surface. A ΔE value of around 2.3 corresponds to a just noticeable difference (JND). This correlation is, however, quite approximate, and there are significant variations in a visual JND over color space (Sharma, 2003). The intensive increase in color change and ΔL with increasing amount of silver nano particles can be related to the oxidation of cellulose and adsorption of amine group to the cellulosic chains.

3.3. UV–vis reflectance spectra

The UV–vis reflectance spectra of the treated cotton fabrics were evaluated using Varian Carry 500. The UV–vis absorbance features

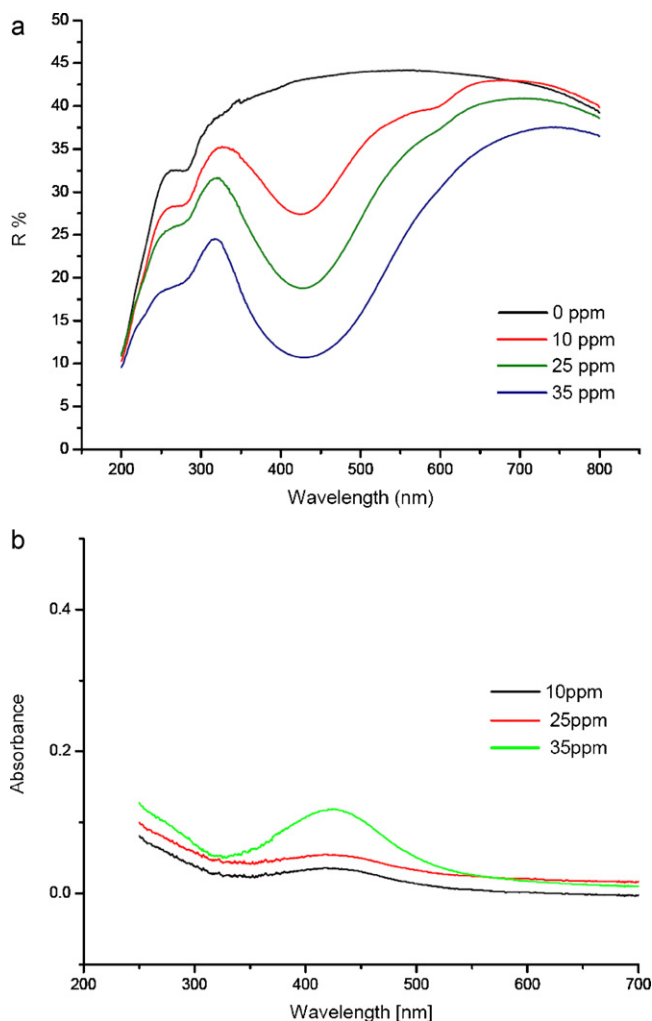


Fig. 3. (a) The UV–vis reflectance spectra of cotton fabrics and (b) the UV–vis absorbance of the solution baths after treating the cotton fabrics.

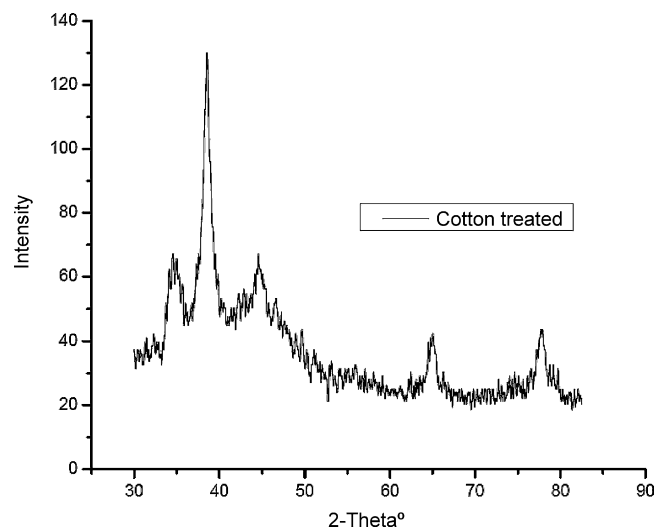


Fig. 4. XRD pattern of cotton sample containing silver nanoparticles.

of the treated fabrics are due to the appearance of a surface plasmon absorption band, and its appearance in the visible region is a characteristic of the noble metal nanoparticles (Luo, Zhang, Zeng, Zeng, & Wang, 2005). Surface plasmon resonance is a collective excitation of the electrons in the conduction band near the nanoparticles' surface (Courrol, Oliveira Silva, & Gomes de, 2007). It was observed that through increasing the amount of nanoparticles on the surface of samples, the average reflectance decreased and the main plasmon peak was observed around 425 nm for three different concentrations of nano silver. These results are consistent with the previous findings (Fig. 3a) (Luo et al., 2005). The maximum peak kept at 425 nm although the absorbance value rises by increasing the concentration of silver. Therefore, the silver particles sizes do not change (Guang-nian et al., 2008). Also, the intensity of the observed plasmon peaks was increased by raising the nano Ag concentration. Conversely, no obvious peak was observed for the untreated cotton fabric. In addition, no tangible shift of absorption band was observed by changing the concentration of nano Ag. Fig. 3b shows the UV–vis absorption wavelength of the remaining solutions after treating the cotton. The absorption peaks are around 425 nm and the intensities increase with an increase the concentration nano silver. The maximum peak is constant corresponding to the UV–vis reflectance spectra of the treated cotton samples.

3.4. XRD pattern

The XRD patterns of the nano silver containing samples in 35 ppm concentration are illustrated in Fig. 4. All the prominent peaks on 2θ scale are about 38.4° , 44.6° , 64.8° , and 77.6° representing the (1, 1, 1), (2, 0, 0), (2, 2, 0), and (3, 1, 1). Bragg's reflections in the face centered cubic (FCC) phase structure of silver nanoparticles. These results are in a good agreement with the early findings (Singh & Khanna, 2007), and indicate the reduction of Tollens' reagent to silver metal. Through full width at half maximum (FWHM) the size of the crystals can be compared. Higher FWHM denotes the smaller crystals; in contrast, a lower amount of FWHM shows bigger ones (Montazer, & Pakdel, 2010). Also the sharp peak at 36° is an indicator of cellulose crystal at (3, 1, 3). The average crystals size based on the Scherrer's equation was somewhere in the region of 88 nm based on a calculation using Eq. (4):

$$\frac{K \times \lambda \times 180}{\text{FWHM} \times \pi \times \cos \theta} = \text{Crystals size (Å)} \quad (4)$$

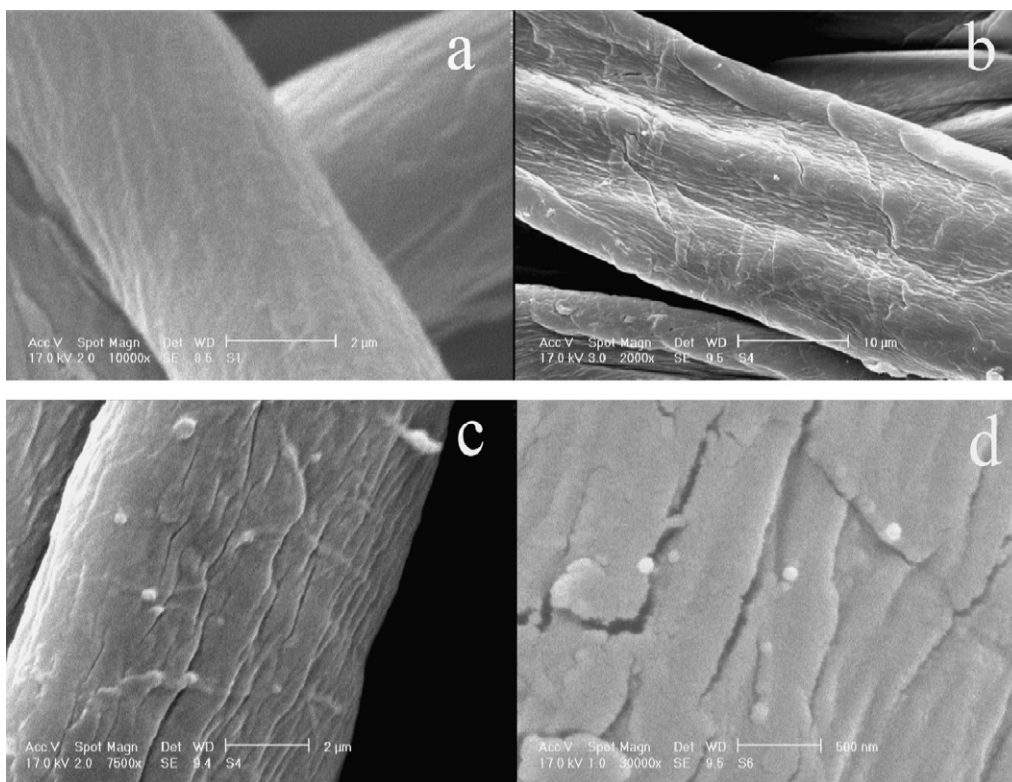


Fig. 5. SEM images of the cotton fabrics: (a) untreated and (b–d) treated with 35 ppm of Ag_2O .

3.5. SEM micrographs

Through the achieved SEM images the presence of silver nanoparticles on the surface of the fabrics treated with a 35 ppm concentration of Ag_2O was confirmed. It was observed that synthesized nanoparticles have a spherical form and the nano particle size of 88 nm can be confirmed as an average particle size. In addition, it was demonstrated that if the spherical precipitations are connected to each other, the autocatalysis mechanism takes place for silver reduction (Fig. 5) (Nersisyan et al., 2003).

3.6. EDS spectrum

Fig. 6 shows the EDS spectrum of a treated sample confirming the existence of Ag element on the surface of the fabric treated with 35 ppm concentration of Ag_2O . The presence of Au element in the EDS spectra is related to a gold layer which covers the outer layer of the sample to prepare it for the SEM images. Fig. 6 confirms the existence of the low amount of nano Ag particles on the surface of the treated fabrics. It was observed that Ag and Au are two major elements on the treated cotton samples. The results were reported

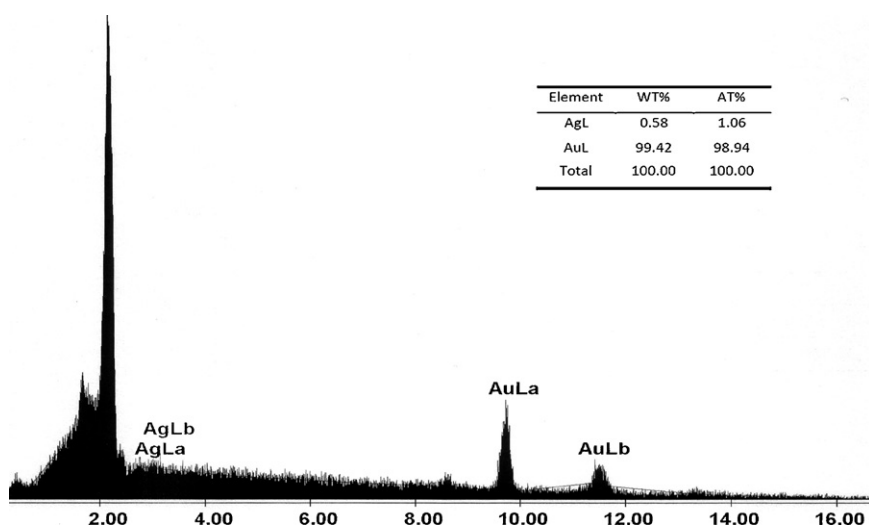


Fig. 6. EDS spectrum of treated fabric.

Table 2

Number of colonies after 24 h and antibacterial reduction percentages of the cotton samples treated with diverse concentrations of nano silver (the numbers of colonies at start was 2×10^3 CFU/mL).

Number of home washing cycles	0		10		20		30	
Bacteria type	<i>E. coli</i>	<i>S. aureus</i>	<i>E. coli</i>	<i>S. aureus</i>	<i>E. coli</i>	<i>S. aureus</i>	<i>E. coli</i>	<i>S. aureus</i>
10 ppm								
Colony No.	<2	14	<2	24	9	40	18	74
Reduction (%)	99.9	99.3	99.9	98.8	99.4	98.0	98.8	96.3
25 ppm								
Colony No.	<2	<2	<2	8	<2	14	<2	28
Reduction (%)	99.9	99.9	99.9	99.6	99.9	99.3	99.9	98.6
35 ppm								
After 24 h	<2	<2	<2	<2	<2	4	<2	16
Reduction (%)	99.9	99.9	99.9	99.9	99.9	99.8	99.9	99.2

based on the both weight percentages (%W) and atomic percentages (%A) of the detected elements.

3.7. Washing fastness of antibacterial property

In this investigation, silver nanoparticles were directly synthesized onto the surface of cotton. In order to evaluate the washing fastness the samples treated with 10 ppm, 25 ppm, and 35 ppm of Ag₂O were washed based on the AATCC 61(2A)-1996 test method. Subsequently, the antibacterial properties of each sample were assessed after each cycle of laundry. An efficient antibacterial property was achieved due to the presence of silver nanoparticles on the surface of fabrics (Table 2).

Concerning the differences between these two bacteria in terms of resistance against antibacterial agents, in the case of *S. aureus* as a Gram-positive, the minimal inhibitory concentration (MIC) is higher than *E. coli*, a Gram-negative, which is in agreement with the knowledge regarding its cell wall that consists of a thick peptidoglycan layer. Overall differences in pathogenicity of *S. aureus* are imputed to genome islands encoding a lot of toxins (Petica, Gavrilu, Lungu, Buruntea, & Panzaru, 2008). Both employed bacteria showed a suitable stability in the laundry even after 30 washing cycles. This means that nano Ag particles placed on the surface of the cotton were stabilized to repeated laundries. There could be two reasons for this phenomenon. On the one hand, the presence of alkali causes the surface of cotton fabric to acquire negative charges, $[\text{Ag}(\text{NH}_3)_2]^+$ has positive charges approaching to the cellulose surface and then reducing to silver metal; therefore, the deposition of nano Ag particles increases on the surface of the fabric. On the other hand, it has been reported that the presence of ammonium has a marked effect on the particle size and it has been revealed that the smallest particles were formed at the lowest ammonia concentration (Pingali et al., 2008; Sharma et al., 2009). In addition, the synthesis process occurred in the boil and led to the swelling of the fabrics. Thus, some of the Ag nano particles were assumed to generate into the intra molecular and produced durable antibacterial properties.

4. Conclusion

This research presented a novel and easy method for a green synthesis of silver nanoparticles on the cotton fabrics. The cellulosic chains of cotton were employed as a reducing agent and a stabilizer for the silver nanoparticles synthesis. The silver nanoparticles imparted an efficient antibacterial property to the cotton fabric with an excellent washing durability. The reduction in antibacterial properties of the nano silver treated cotton fabrics after 30 cycles of washing was negligible. The obtained coordinates of CIELAB color system revealed that through increasing the silver nanoparticles loaded on the surface of the cotton fabrics their color turned to yellow and color changes or ΔE increased. Furthermore, the

mechanism of nano silver synthesis through oxidation of cellulose was proposed and the Raman spectra and CHN elemental analysis were confirmed in this mechanism. The XRD pattern, SEM micrographs and EDS spectrum confirmed the existence of silver nanoparticles on the cotton fabrics. The average crystals size of nano silver based on the Scherrer's equation was in the region of 88 nm.

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