Novel Feature of Nano-Titanium Dioxide on Textiles: Antifelting and Antibacterial Wool

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ABSTRACT: In this study, the antifelting and antibacterial features of wool samples treated with nanoparticles of titanium dioxide (TiO_2) were evaluated. To examine the antifelting properties of the treated samples, the fabric shrinkage after washing was determined. The antimicrobial activity was assessed through the calculation of bacterial reduction against *Escherichia coli* (Gram-negative) and *Staphylococcus aureus* (Gram-positive) bacteria. TiO_2 was stabilized on the wool fabric surface by means of carboxylic acids, including citric acid (CA) and butane tetracarboxylic acid (BTCA). Both oxidized samples with potassium permanganate and nonoxidized wool fabrics were used in this study. The relations between both the TiO_2 and carboxylic acid concentrations in the impregnated bath and the antifelting and antibacterial properties are discussed. With increasing concentration in the impregnated bath, the amount of TiO_2 nanoparticles on the surface of the wool increased; subsequently, lower shrinkage and higher antibacterial properties were obtained. The existence of TiO_2 nanoparticles on the surface of the treated samples was proven with scanning electron microscopy images and energy-dispersive spectrometry. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 121: 3407–3413, 2011

Key words: coatings; colloids; crosslinking; esterification; fibers

INTRODUCTION

Wool is a high-quality protein fiber and is widely used as a high-quality textile material. There has also been considerable interest in finding new applications for wool.[1] Wool fibers are known as a superior textile material because of their lightness, warmth, softness, and smoothness.[2] They consist of keratin proteins, which are made of 21 amino acids, and these amino acids bring both free amino and carboxylic acid groups into wool.[3] The scales of the outer layer of the wool surface play an important role in many of wool's properties, including the felting, appearance, soiling, dyeing, and adhesion of polymers, such as in shrink-resist treatments.[4] Shrink-proofing processes are one of the most common finishes that are used in the wool industry.[5] Almost all wool garments shrink during laundering, but their propensity differs.[5] The scales of wool overlap with each other.[6] These surface scales cause a frictional difference when they are rubbed from tip to root than when they are rubbed from root to tip, and this difference is one of the undesirable features of wool fibers.[6] The fibers in the fleece on a sheep all grow out of the follicles in the same direction, and they generally grow at a similar rate.[5] After the fleece washing process, the unit direction of the fibers will not be the same as they used to be; therefore, fibers with different directions are placed next to each other, shrinking the produced fabric as a result.[5] Chlorination, oxidation with potassium permanganate, treatment with permonosulfuric acid, polymer deposition processes for wool tops, and fabric treatment with polymers are some common approaches that are used in the wool industry to reduce the shrinkage of wool.[5] In several studies, the positive impacts of $KMnO_4$ treatment on reducing the shrinkage of wool have been demonstrated.[6] It has been reported that through oxidation processes, the surface morphology of wool changes. Oxidation processes affect the dyeability, water absorption, and other features of wool.[6,7] Normally, $KMnO_4$ would diffuse right through wool fibers and readily oxidize all of the accessible cystine amino acids in the epicuticle.[5]

Through oxidation processes, higher amounts of carboxylic acids can be absorbed on the surface of wool, and this was demonstrated quantitatively by Montazer and coworkers.[8,9] Nanoparticles of TiO_2 have a high affinity towards carboxyl and hydroxyl groups; therefore, introducing higher amounts of these groups can lead to a higher absorption rate of TiO_2 on the surface of wool in comparison with nonoxidized samples.[10–15] The amount of $KMnO_4$

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consumed by the wool increased with time, temperature, and concentration of $KMnO_4$ in the treatment bath.[16] Because of the suitable properties of nano-titanium dioxide (nano-TiO_2), such as a high photoactivity, thermal and chemical stability, low cost, and nontoxicity, this semiconductor is widely used in practical applications. For instance, in different industries, such as water purification and air purification, it has been used.[11,17,18] In this study these nano-TiO_2 particles were linked on the wool surface with two different crosslinking agents, butane tetracarboxylic acid (BTCA) and citric acid (CA).[19] One of the most important features of nanoparticles is that they generally demonstrate a significant surface area because the ratio of the surface to the volume of the materials grows with a reduction in the particle size; hence, novel functional properties can be observed.[5]

With the advent of the antimicrobial finishing methods, the growth of microbes and fungus on textile surfaces during use and storage has been significantly reduced; meanwhile, scientists continue to search for new ways to decrease this unsuitable phenomenon.[20] These organisms can be found everywhere in the environment and places in which necessary requirements, such as nutrients, temperature, and moisture, exist; they can be multiplied quickly and affect textiles negatively.[20] The generation of an unpleasant odor and unwanted stains and the reduction of the mechanical strength of the fabrics are examples of the demerits of growing microorganisms on the surface of textiles.[20] The prevention of microbial attacks on textiles has become most important because of its hygienic features on treated textiles, and therefore, interests in antimicrobial fabric finishing have increased over the last few years.[20,21] This tendency among consumers that most of the time prefer to use antimicrobial garments is also noteworthy.[22] The large surface area and ability to retain moisture make textile fabrics more prone to bacterial growth, and they offer an ideal environment for microbial growth, providing oxygen, water, and warmth.[23]

For antimicrobial treatments, positive impacts of reagents such as organic silicon quaternary ammonium compounds, halogenated diphenyl ether derivatives nitrofurantoin, and organic nitrogen compounds have been reported.[24] The application of natural antimicrobial agents on textiles goes back to antiquity, when ancient Egyptians used spices and herbs to preserve mummy wraps;[25] moreover, there have been several studies on natural antimicrobial agents, one of which was written by Han and Yang,[22] about the effects of curcumin on the antimicrobial features of woolen fabrics.[22] Singh et al.[26] showed that some natural dyes also play a prominent role in producing antimicrobial features on textiles. It has been shown that metal ions can react with keratin fibers and effectively lead to better antimicrobial

properties.[27] The application of nanoparticles on textiles has been known as a state-of-the-art approach in this field in recent years.[28] In fact, general methods of preparing antimicrobial fabrics or fibers are the adsorption or grafting of some compounds on fiber surfaces.[28] Wang demonstrated that antibacterial woolen fabrics can be achieved by means of nano-SiO_2.[28] In another study done by Fu et al.,[29] the antimicrobial features of TiO_2 nanoparticles were assessed.[29] Other approaches that have been carried out in this field include the coating of wool with resin-bonded copper-8-quinolinolate, chlorinated phenol and its datives, sodium dichloroisocyanurate, and organic compounds in finishing processes.[30] Numerous pieces of research have been conducted regarding the other various aspects of TiO_2 and novel features that can be obtained. For instance, self-cleaning,[10–12] UV protection,[9,31] and hydrophilic or hydrophobic properties of TiO_2[32–35] have been discussed extensively in related articles.

The main purpose of this study was to evaluate the antifelting and antimicrobial properties of wool through treatment with nano-TiO_2. In this study, the relations among TiO_2, crosslinking agent types, and concentration with these novel properties were investigated.

EXPERIMENTAL

Materials

The characteristics of the 100% pure wool fabric used were as follows: gram mass $350 g/m^2$, yarn fineness 30Nm, and warp and weft yarn densities $16.2 cm^{-1}$ and $15.1 cm^{-1}$, respectively. Nanopowder of titanium dioxide (TiO_2; Degussa-p-25, Dusseldorf, Germany) was used, and its average particle size was around 21 nm. Among other qualities, nano TiO_2 has two prominent properties, i.e. its large surface area and comprising a mixture of two anatase and rutile phases. BTCA ($C_6H_{10}O_8$) and citric acid monohydrate ($C_6H_8O_7 \cdot H_2O$) from Merck (Darmstadt, Germany) were used as crosslinking agents, and sodium hypophosphite (SHP; $NaH_2PO_2 \cdot H_2O$) from Riedl-de-haen (Sleeze, Germany) was used as a catalyst. Components of the impregnating bath were mixed by means of ultrasonic waves. Moreover; the treated samples were cured in an oven. Scanning electron microscopy (SEM) images and energy-dispersive spectrometry (EDS) spectrum were prepared with a SERON AIS 2100 scanning electron microscope (South Korea).

$KMnO_4$ treatment

The scoured wool samples were treated with 3% $KMnO_4$ in acidic solution (pH = 4) in the presence

TABLE I
Ingredients of the Impregnated Bath

TiO$_2$ (g/100 mL)	Crosslinking agent (CA or BTCA; %)	SHP (%)
0.01	10.5	6.3
0.75	10.5	6.3
0.38	1.25	0.75
0.38	10.5	6.3
0.38	19.75	11.85

of 10 g/L NaCl. To adjust the pH, $4N$ sulfuric acid was used. The treatment was conducted for 20 min in L:G → 40 : 1 (liquid to good ratio). The MnO$_2$ ions remained on the surface of the wool, resulting in an undesirable brown color. Both 3% sodium bisulfite along with 0.5 mL of $4N$ H$_2$SO$_4$ were used to remove the brown color from the samples. The color removing process took 15 min. Afterwards, the oxidized samples were rinsed with warm (40°C) and cold water (22°C) carefully and then were dried at room temperature for 24 h. Additionally, all of the treatments were carried out in an ultrasonic bath.

Nano-TiO$_2$ treatment

The wool samples were treated with diverse concentrations of either TiO$_2$ or crosslinking agents for 32 min according to Table I. The finishing bath was composed of cross-linking agent (either BTCA or CA), SHP, and TiO$_2$. Through sonicating, the nanoparticles were dispersed, prior to inserting the wool samples. The main crosslinking reactions between the wool fabric and carboxylic acid compounds were carried out in curing.[24] The treated samples were cured at 120°C for 2 min.

Antifelting testing

The treated samples were immersed in a solution containing 1% nonionic detergent at 50°C in L:G → 40 : 1 and were agitated for 45 min, according to ISIRI 1454 standard. Before and after this process, the surface dimensions of the fabrics were measured, and the results were reported as percentages on the basis of eq. (1):

$$\text{Area shrinkage } (\%) = (P - S) \times 100/P \qquad (1)$$

where P is the primary surface area of the fabric and S is the surface area of the samples after shrinkage.

Antimicrobial testing

The antibacterial properties of the treated samples were quantitatively evaluated by the AATCC 100-1999 test method against *Escherichia coli* and *Staphylo-*

coccus aureus, which were pathogenic Gram-negative and Gram-positive bacteria, respectively. Nano-treated samples were inserted into the germ-containing agar plates, inoculated with *E. coli* and *S. aureus*, and then incubated in agar media. To evaluate the TiO$_2$ antimicrobial activity on the wool fabric, the colony reduction on various kinds of fabrics were compared. The results were expressed as percentages, which were achieved from eq. (2):

$$\text{Reduction } (\%) = (A - B) \times 100/A \qquad (2)$$

where A and B are the number of bacterial colonies of the untreated and TiO$_2$-treated samples, respectively.

RESULTS AND DISCUSSION

Antifelting

Wool is one of a large group of animal fibers that is almost entirely composed of a family of proteins generally known as *a-keratins*.[5] In this investigation, the wool samples were oxidized with potassium permanganate in the presence of NaCl. Numerous studies have been carried out to better understand the impacts of diverse surface modification methods on wool shrinkage.[36] McPhee has reported that reducing the oxidized samples by sodium bisulfite has two distinct effects: first, precipitated MnO$_2$ is removed from the wool, and second, the reduction of the oxidized wool causes an increased shrink resistance. In addition, it has been demonstrated that through the oxidation process, the sharp edges of wool scales are fully removed.[36] Kantouch et al.[16] illustrated that the oxidation process in an acidic solution gave better antishrink properties in comparison with those of in alkali solutions. Also, they declared that the amino acids cystine, tyrosine, and tryptophan were greatly affected by the KMnO$_4$ treatment. The nitrogen and sulfur contents decreased with increasing amount of KMnO$_4$.[16] This reduction in the amount of sulfur indicated that the amino acid cystine was affected during this process.[16] The mechanism of the degradation of the amino acid cystine is proposed in Scheme 1. Montazer and Pakdel[9] proved that through oxidation process, oxidized wool seemed whiter than the raw one. The shrinkage rate reduced from 9% in raw wool to 6.8% in an oxidized one, and this result was consistent with previous findings (Fig. 1).[16,37] This could have been due to the pivotal role of scales in the shrinkage of wool; after the oxidation process, these

$$Cys-S-S-Cys + NaHSO_3 \leftrightarrow Cys-S-SO_3Na + Cys-SH$$
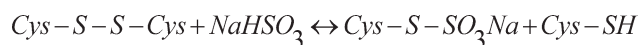
Scheme 1 Degradation of the amino acid cystine (Cys) in the oxidation process.
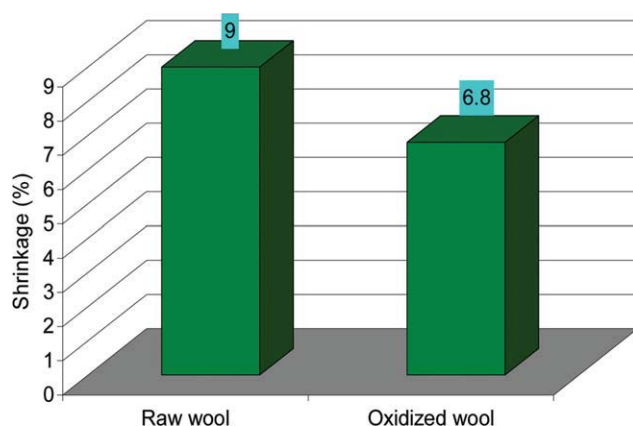
Figure 1 Effect of the oxidation process on the wool shrinkage. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

scales were removed relatively, and therefore, their role was reduced.[16,24,37] The carboxylic acid groups on the surface of wool can react with many cationic compounds, for instance, metal ions, such as Ag^+ and Ti^+.[30]

The results in Figures 2 and 3 show that with increasing amount of TiO_2 and crosslinking agents, the rate of shrinkage decreased; this can be accounted for in these two conditions: higher amounts of TiO_2 absorbed on the surface of wool and, consequently, the ups and downs of the surface of wool covered completely. Subsequently, the roughness of wool decreased; as a result, a lower rate of shrinkage was observed. TiO_2 showed photoinduced hydrophilicity, especially under UV rays. This property of TiO_2 was evident because of its porous structure, which enabled fast water absorption. It is also believed that the hydrophilicity is related to the density of the surface
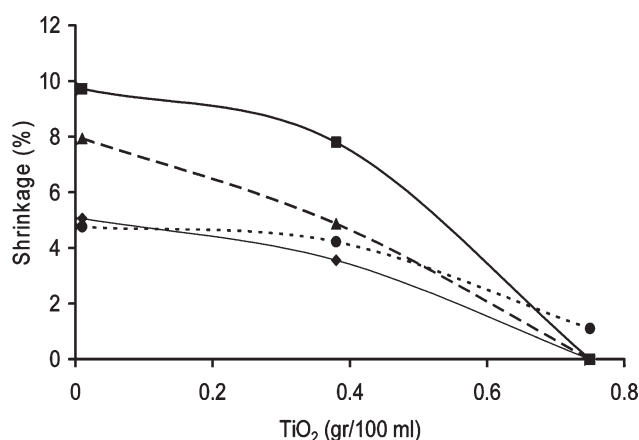

Figure 2 Effect of the TiO_2 concentration on the wool shrinkage: (◆) oxidized sample treated with TiO_2, 10.5% CA, and 6% SHP; (■) nonoxidized sample treated with TiO_2, 10.5% CA, and 6% SHP; (▲) nonoxidized sample treated with TiO_2, 10.5% BTCA, and 6% SHP; and (●) oxidized sample treated with TiO_2, 10.5% BTCA, and 6% SHP.
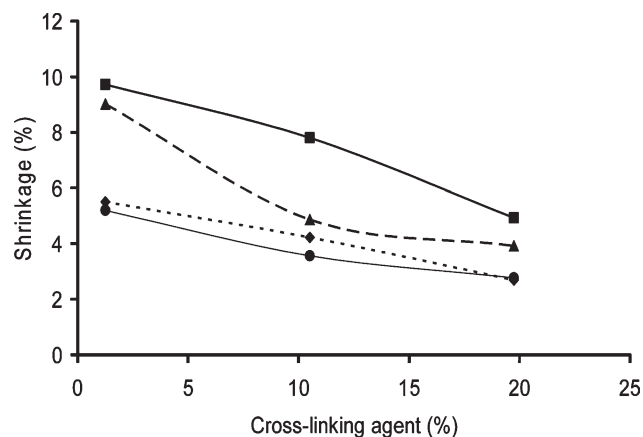

Figure 3 Effect of the crosslinking agent concentration on the shrinkage: (■) nonoxidized sample treated with 0.38 g/100 mL TiO_2, CA, and 6% SHP; (▲) nonoxidized sample treated with 0.38 g/100 mL TiO_2, BTCA, and 6% SHP; (◆) oxidized sample treated with 0.38 g/100 mL TiO_2, BTCA, and 6% SHP; and (●) oxidized sample treated with 0.38 g/100 mL TiO_2, CA, and 6% SHP.

hydroxyl of TiO_2.[32–35,38] A way to justify the relation between the hydrophilicity of the treated wool and the decrease in the shrinkage would be that the absorbed water could play a role as a plasticizer among the surface scales of the wool; therefore, shrinkage decreased. In oxidized samples, the surface shrinkage was lower than the nonoxidized samples, and in those with higher nano-TiO_2 contents (0.75 g/100 mL), no obvious shrinkage was observed. Wool fabrics treated with CA and BTCA had diverse shrinkages. In previous articles, we confirmed that through the oxidation process, higher amounts of crosslinking agents could react with the functional groups of wool. As a result, a higher amount of TiO_2 could be absorbed on the surface of the fabric.[9,12,31] BTCA absorbed a greater amount of TiO_2 in comparison with CA; therefore, it could add better antishrinkage properties to wool, although it is an expensive substance. Thus, most scientists prefer the utilization of CA as a crosslinking agent because it is more economical. In those oxidized samples that were treated with high concentrations of crosslinking agent (BTCA or CA), no obvious shrinkage was observed. In the sample just treated with TiO_2, the shrinkage was about 7%. These results confirm that with the presence of adequate amounts of either TiO_2 or crosslinking agent in the impregnated bath, antifelting wool was obtained.

Antimicrobial properties

The wool samples were treated with diverse concentrations of TiO_2 and crosslinking agents to assess their impacts. It was proven that through an increase in the amount of nano-TiO_2 particles in the impregnated bath, the absorption of nanoparticles on the surface of

the wool increased. Consequently, the proposed features could be achieved in higher quality.[9,12] The self-cleaning and UV-absorption properties of TiO_2 have been evaluated by Montazer and Pakdel.[9,12,31] With regard to the comparison between crosslinking agents, Wang and Chen[39] showed that TiO_2 surface deposition of treated fabrics for BTCA, which contains four carboxylic acid groups, was higher than that of CA, which contains two carboxylic groups.[39] Acid crosslinking agents have attracted a great deal of scientific attention in recent years.[39,40] Wang and Chen, through SEM images, showed that the surface of a BTCA-treated fiber had a higher deposition of crosslinking agent, but CA-treated and raw fibers had lower and no deposition of crosslinking agent, respectively.[39] It follows that higher amounts of TiO_2 could be absorbed on the surface of the wool. Through oxidation processes, the binding between wool and carboxylic acids occurs better in the treated wool in comparison with the raw wool. In the other words, through these treatments, not only is wool allowed to obtain a higher level of crosslinking agents, but a higher amounts of nanoparticles can also be absorbed on the surface of the wool, through which higher proposed features can be achieved (Fig. 4).

The microorganisms chosen for this study were *E. coli* and *S. aureus*, which are the most frequently evaluated species.[23] *E. coli* resist common antimicrobial agents and causes urinary tract as well as wound infections, and *S. aureus* is the major cause of cross-infection in hospitals.[23] The relations of antimicrobial activity to the TiO_2 and crosslinking agent concentrations are shown in Figures 5 and 6, respec-

Figure 5 Effect of the TiO_2 concentration in the impregnated bath on the antimicrobial activity against *E. coli* bacterium: (▲) oxidized samples treated with TiO_2, 10.5% BTCA, and 6% SHP; (■) nonoxidized samples treated with TiO_2, 10.5% BTCA, and 6% SHP; (◆) oxidized samples treated with TiO_2, 10.5% CA, and 6% SHP; and (●) nonoxidized samples treated with TiO_2, 10.5% CA, and 6% SHP.

tively. No obvious microbial reduction was observed for *S. aureus* on the treated samples.

Figure 5 shows that a higher amount of TiO_2 on the surface of the wool caused better antimicrobial properties in the treated samples, and those oxidized fabrics that were treated with BTCA as a crosslinking agent and TiO_2 showed better microbial reduction against *E. coli*. Figure 6 depicts the prominent role of the crosslinking agents in this phenomenon. BTCA-treated samples had a higher antimicrobial
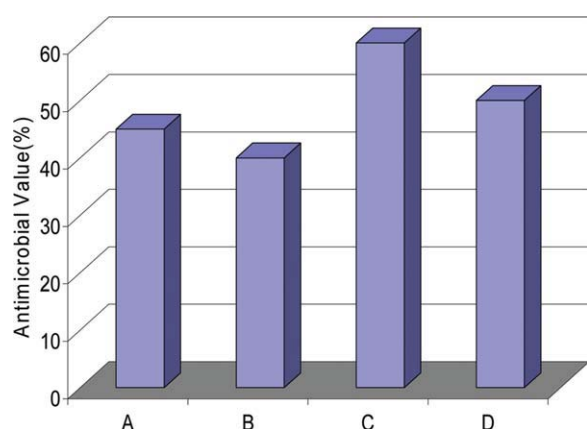
Figure 4 Effect of the oxidation process on the antimicrobial activity against *E. coli* bacterium: (A) oxidized sample treated with 0.75 g/100 mL TiO_2, 10.5% CA, and 6% SHP; (B) nonoxidized sample treated with 0.75 g/100 mL TiO_2, 10.5% CA, and 6% SHP; (C) oxidized sample treated with 0.75 g/100 mL TiO_2, 10.5% BTCA, and 6% SHP; and (D) nonoxidized sample treated with 0.75 g/100 mL TiO_2, 10.5% BTCA, and 6% SHP. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary. com.]
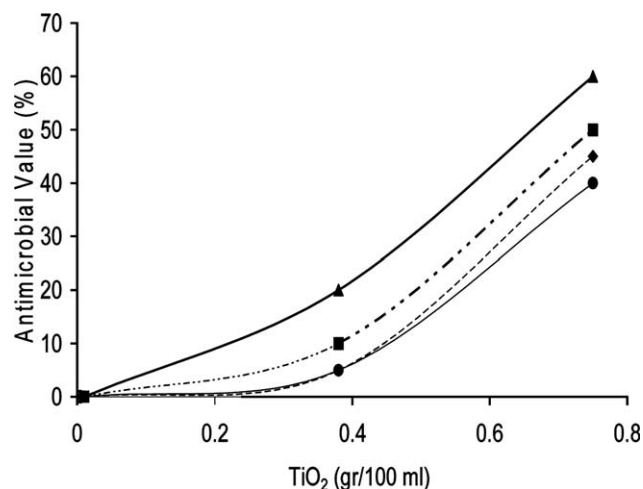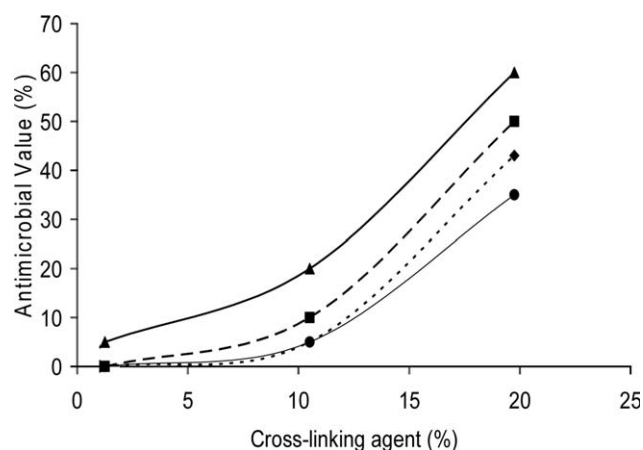
Figure 6 Effect of the crosslinking agent concentration in the impregnated bath on the antimicrobial activity against *E. coli* bacterium: (▲) oxidized samples treated with 0.38 g/100 mL, BTCA, and 6% SHP; (■) nonoxidized samples treated with 0.38 g/100 mL TiO_2, BTCA, and 6% SHP; (◆) oxidized samples treated with 0.38 g/100 mL TiO_2, CA, and 6% SHP; and (●) nonoxidized samples treated with 0.38 g/100 mL TiO_2, CA, and 6% SHP.
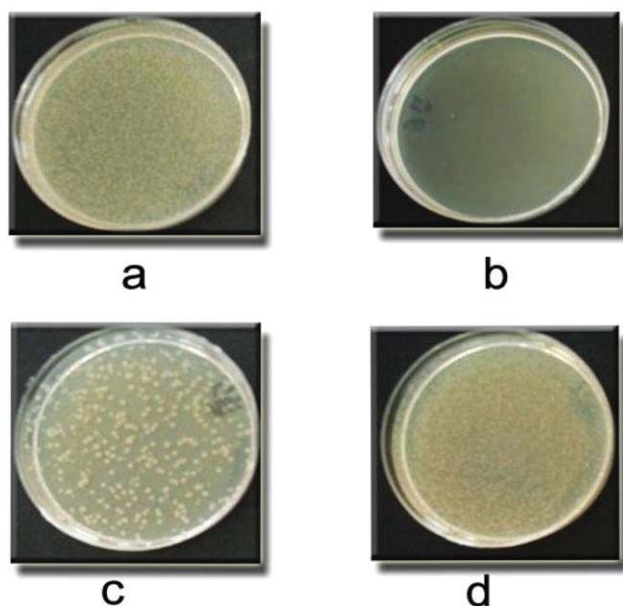
Figure 7 Antimicrobial efficiency of the wool fabric under different conditions: (a) raw wool against *E. coli* bacterium; (b) wool treated with TiO_2, BTCA, and SHP against *E. coli* bacterium; (c) raw wool against *S. aureus* bacterium; and (d) wool treated with TiO_2, BTCA, and SHP against *S. aureus* bacterium. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

percentage. The main accepted reasons for the antimicrobial activity of TiO_2 are positive species. The positive species are produced by TiO_2, especially under UV rays, whose energy level is higher than that of its band gap. This, in turn, gives birth to the photocatalytic nature of TiO_2, which reacts with negatively charged residues at the cell surface of bacteria and leads to the leakage of intercellular substance. As a result, the normal metabolism of microorganisms is inhibited; this leads to the death of bacteria (Fig. 7).[23] The cell wall of the *S. aureus* bacterium is thicker than that of *E. coli*; therefore, TiO_2 was not powerful enough to destroy it. Hence, antimicrobial activity was not observed (Fig. 7).[23] The acidic situation was under the isoelectric point of the wool. In this state, the wool surface carried negative charges; therefore, higher amounts of TiO_2 particles, whose charges were positive in acidic solutions, could be absorbed on the surface of the fabric.[3] The foregoing results suggest that an adequate amount of antibacterial and also crosslinking agents are necessary for the inhibition of the microorganisms.

SEM images and EDS spectrum

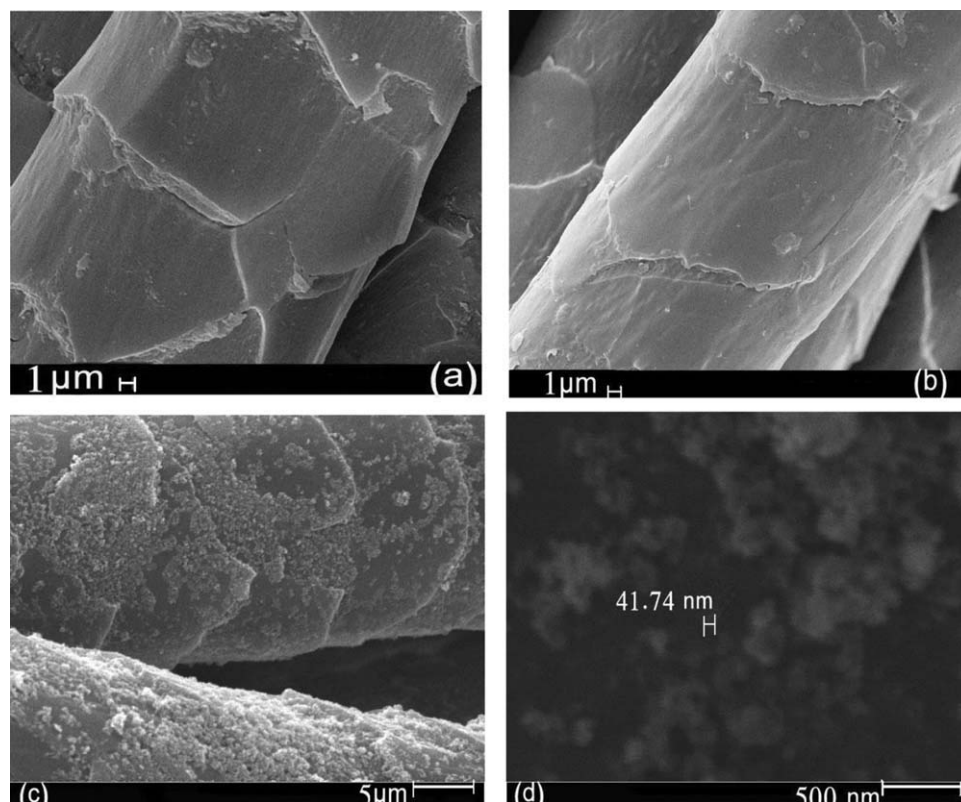The surface morphology of raw and oxidized wool samples were also evaluated by SEM images, which

Figure 8 SEM images of the wool fabrics: (a) raw wool (3000×); (b) oxidized wool (3000×); (c) wool treated with TiO_2, BTCA, and SHP (6000×); and (d) wool treated with TiO_2, BTCA, and SHP (80,000×).
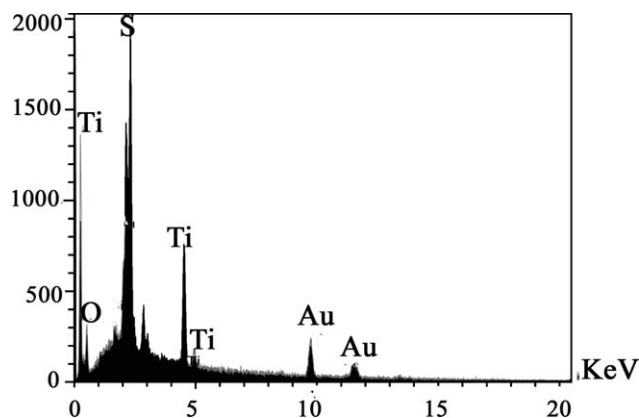
Figure 9 EDS spectrum of treated wool.

were taken by a SERON AIS 2100 (Fig. 8). It was observed that through the oxidation process, the sharp edges of the wool scales were removed; this led to less shrinkage than before. On the surface of the treated samples, the diameter of a TiO_2 particle was measured and was around 40 nm. The photograph [Fig. 8(c)] reveals the deposition of nano-TiO_2 particles on the treated fabrics.

In this research, the EDS spectrum was used to analyze the absorbed particles, which existed on the surface of the treated wool (Fig. 9). In addition, through this test, the purity of the nano-TiO_2 particles that we used was determined. Other elements, such as S and O, were also detected. The appearance of Au in the EDS spectrum is due to the gold layer, which was used to cover the sample surface to obtain the SEM images.

CONCLUSIONS

In this study, the details of the antifelting and antimicrobial features imparted onto wool samples were examined. It was shown that through the treatment of oxidized and nonoxidized wool samples with nano-TiO_2, stabilized by crosslinking agents (CA and BTCA) and catalyst (SHP), antimicrobial and antishrinkage properties were obtainable on the wool fabrics. We observed that between two distinct crosslinking agents, BTCA-treated samples turned out to be better because of the higher TiO_2 absorption on the surface of the wool. We demonstrated that the oxidation process with potassium permanganate played a prominent role in raising the rate of carboxylic acid absorption and, hence, that of the TiO_2 nanoparticles. With increasing amount of TiO_2 in the impregnated bath, the antimicrobial and antifelting features of the wool were enhanced, and with the rise of crosslinking agents, such features could be improved. SEM images and EDS were used to confirm the existence of nanoparticles on the surface of the wool.

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