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Novel Functional Finishing of Wool Fabric Using Reduced Graphene oxide-zinc oxide Nanocomposite

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Abstract

In this study, a new finishing technique is introduced by treating wool fabrics with reduced graphene oxide/ZnO nanocomposites. Graphene oxide was coated onto wool fabric by dipping the fabric in a graphene oxide solution and drying it in an oven. The nanocomposite was synthesized on wool fabric in a single step by reduction of zinc acetate and graphene oxide with sodium hydroxide in the impregnation bath. The homogenous distribution of the reduced graphene oxide-zinc oxide nanocomposite on the fiber surface was confirmed by field emission scanning electron microscopy (FE-SEM), Energy-dispersive X-ray spectroscopy (EDS), and X-ray mapping. X-ray diffraction patterns proved the presence of zinc oxide nanoparticles on the treated wool fabric. Also, the defect analysis based on X-ray photoelectron spectroscopy (XPS) established the composition of the nanocomposite. Other characteristics of treated fabrics such as antibacterial activity, photo-catalytic self-cleaning, electrical resistance, ultraviolet (UV) blocking activity, and cytotoxicity were also assessed. The treated wool fabrics possess significant antibacterial activity and photo-catalytic self-cleaning properties by degradation of methylene blue under sunlight irradiation. Moreover, this process has no negative effect on the cytotoxicity of the treated fabric even reducing electrical resistance and improving UV blocking activity.

Keywords: Functional finishing, Wool, Reduced graphene oxide-ZnO nanocomposite, photocatalytic, Electrical conductivity.

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1. Introduction

Wool is one of the oldest fibers known to man and is widely used as a high-quality textile material due to its unique natural properties (softness, warmth, lightness, etc.). [1]. The production of wool fabrics with multi-functional properties has become a concern for the textile industry as consumers are constantly looking for improved performance and exciting new innovations. Nanoparticles can now be applied to textile materials to achieve end-use demands for textiles such as self-cleaning, antibacterial, electrical conductivity, and UV blocking. For example, the deposition of zinc oxide nanoparticles on textiles provides new properties such as self-cleaning, UV protection, super hydrophilicity, antibacterial effect, and flame retardancy [2-7]. Wool fabrics loaded with zinc oxide nanoparticles have potential uses in medical and textile applications such as medical devices, healthcare, wound dressing, military, protective clothing, personal care products, and clothing. [8-10]. There is abundant research on the application of ZnO nanoparticles for the functional finishing of textiles. Mohammadi and co-workers prepared photoactive and antibacterial fabric through in situ synthesis of nano ZnO on polyester fabric [11]. Behzadnia et al. produced self-cleaning wool fabric using zinc oxide nanoparticles [12]. Prasad and colleagues obtained durable antibacterial and UV protection fabric based on zinc oxide coatings on cotton fabrics [13]. Zhang et al. applied ZnO nanoparticles for generation of antimicrobial bamboo pulp fabric [14].

Continuous advances in nanotechnology in materials science have opened new pathways for the development of new functional materials. Graphene, a one-atom-thick planar sheet of sp2-bonded carbon atoms tightly packed in a two-dimensional honeycomb crystal lattice, is most attractive due to its unique chemical and physical properties and its diverse potential applications [15]. Some researchers produced electrically conductive fabrics by immobilizing graphene on the surfaces of fabrics [16-18]. Also, ultraviolet blocking and antimicrobial textiles are obtained through applying the graphene [19].

In a mixture of graphene and zinc oxide, graphene would act as an electron acceptor of the photo-generated electrons for ZnO, contributing to the extension of recombination time for electron-hole pairs [20]. Also, the graphene's high surface area would improve the photocatalytic performance of zinc oxide nanoparticles by expanding the photosensitivity under visible light [21]. The application of graphene/zinc oxide nanocomposites for fabric finishing has also been recently reported and has shown its advantages [22].

In this study, nanocomposites of zinc oxide and reduced graphene oxide were synthesized for the first time on wool fabrics. The photo-activity, electrical conductivity, antibacterial activity of the coated fabrics, and the synergism effect of ZnO nanoparticles and graphene on these properties were investigated and discussed in detail.

2. Experimental

2.1. Material

The plain weave structure wool fabric was used with the fabric weight of 170 g/m^2. Graphite powder with particle size less than 20 µm was purchased from Sigma-Aldrich. Zinc acetate dehydrate ($Zn(CH_3COO)_2 \cdot 2H_2O$) as a precursor to synthesis nano ZnO, sodium hydroxide (NaOH), sulfuric acid (H_2SO_4, 98 %), hydrogen peroxide (H_2O_2, 30 %), potassium permanganate ($KMnO_4$) and hydrochloric acid (HCl, 37 %) were prepared from Merck. Methylene blue (CI 52015) was provided by Uhao Co. (China).

2.2. Instrument

FE-SEM and X-ray mapping images and EDS patterns were established by MIRA3-TESCAN, field emission scanning electron microscope (FE-SEM) (Czech Republic). The X-ray diffraction (XRD) analysis was performed using an STOE (model STADI MP) X-ray Diffractometer, made in Germany. The patterns were recorded in the diffraction range of 2Θ from an angle of 10˚ to 90˚ with a scanning speed of 2˚/min at 2Θ step of 0.040˚. Cu Kα radiation (λ= 1.540 Å) with detector scan mode operating at 40 kV and 30 mA was used to investigate changes in crystalline. X-ray photoelectron spectroscopy (XPS) data were recorded using an X-Ray 8025-BesTec XPS system (Germany) with an Al Ka X-ray source (hν = 1,486.6 eV). The electrical surface resistivity of the fabrics was determined based on the AATCC test method 76-2005 using the Hioki Digital HiTester Multimeter, model 3256-50, Japan. UV-blocking activities of the fabrics were recorded using Perkin-Elmer Lambda 35 UV-vis spectrophotometer.

2.3. Methods

2.3.1. Synthesis of graphene oxide

The improved Hummer method to oxidize graphite for the synthesis of graphene oxide was applied [23, 24]. First, 1 g of graphite powder was added to 50 ml of sulfuric acid and stirred for 12 hours at ambient temperature. KMnO4 (3.5 g) was then slowly added in an ice bath below 10°C and then stirred at 50°C for 2 hours. Deionized water (70 mL) and hydrogen peroxide (5 mL) were then added and the solution was stirred for 30 minutes. The resulting product was centrifuged and washed sequentially with deionized water, 5% hydrochloric acid and again with deionized water (three times). The obtained brown paste was mixed with water, and then the dispersion was sonicated in an ultrasonic bath (WiseClean, 50–60 Hz) for 60 min. The final sonicated dispersion was centrifuged at 3000 rpm for 30 min and the nonexfoliated graphite oxide was removed, resulting in a pure graphene oxide aqueous dispersion. Finally, the graphene oxide powder was obtained by freeze-drying.

2.3.2. Preparation of reduced graphene oxide-ZnO nanocomposite onto wool fabric

To coat the wool fabric with graphene oxide, the wool fabric was immersed in an aqueous suspension of graphene oxide (0.5% w/w) and heated at 80 °C for 30 min. Then, the fabric was dried in an oven at 85 ºC for 20 min. Next, zinc acetate with various concentrations (1, 2, 3, 4, and 5 % wt.) was dissolved in 100 mL distilled water at ambient temperature under vigorous stirring. The graphene oxide coated fabrics and raw wool samples were immersed into the solutions. 3 % wt. of sodium hydroxide was added, and the solution was stirred at 85 ºC for 60 min. The treated fabrics were dried at 60 ºC for 30 min followed by curing at 130 ºC for 4 min. Finally, the coated fabrics were washed with distilled water and dried in air.

2.4. Test methods

2.4.1. Discoloration of methylene blue

The photocatalytic activities of treated fabrics were evaluated by decomposition of the methylene blue under sunlight irradiation in water. The dye concentration in the solution was calculated by Varian Cary 300 UV–Vis spectrophotometer using a calibration curve. A computer program determines the absorbance of dye solution at the maximum wavelength of methylene blue - 663 nm. The first step was the preparation of a dye solution with distilled water (10 mg/L). The wool samples (5×5 cm 2) were then added to 100 ml of dye

solution. First, the mixed solution was stirred for 15 minutes without irradiation to equilibrate dye adsorption. The wool samples (5×5 cm 2) were then added to 100 ml of dye solution. First, the mixed solution was stirred for 15 minutes without irradiation to equilibrate dye adsorption. After irradiation, the decolorization and photocatalytic degradation efficiency have been calculated as:

$$\text{Efficiency } (\%) = (C_0 - C_e)/ C_0 \times 100 \quad (1)$$

C0 and Ce correspond to the initial and final dye concentrations before and after sunlight irradiation. In this equation, E% shows the dye photocatalyst degradation percent [25].

2.4.2. Microbiological test

The antimicrobial activity of the samples was evaluated against *Escherichia coli* (*E. coli*, ATCC 25922, Gram-negative bacterium) and *Staphylococcus aureus* (*S. aureus*, ATCC 25923, Gram-positive bacterium) using AATCC 100–2004 test method. This method is specially designed for specimens treated with non-releasing antibacterial agents under dynamic contact conditions. Antimicrobial activity was expressed in terms of the percentage reduction of the microorganisms and calculated as:

$$\text{Percentage reduction of microorganisms } (R)\% = (A\text{-}B)/A \times 100 \quad (2)$$

A and B are the number of microorganisms colonies on untreated and treated fabrics, respectively.

There were 3.4×105 colony forming units (cfu) of bacteria in the primary inoculum. A saline solution of 8.5 g/L sodium chloride to 1000 ml distilled water was used as the neutralizing solution. A serial dilution of 10–10,000 was made for incubation on an agar plate. Tryptic soy agar (Merck, Germany) was applied as the agar.

2.4.3. Cytotoxicity test

The cell toxicity of the treated fabrics was carried out by utilizing an MTT assay. Normal primary human skin fibroblast isolated from the dermis of neonatal *foreskins* was used. Cell culture was performed at 37 ˚C and 5% CO2 condition using Dulbecco's modified Eagles medium (DMEM, Biochrom, Germany) supplemented with 10% fetal calf serum (FCS, Biochrom, Germany). Cells of the third passage were used and seeded in a 96-well microplate at a density of 20000 cells per well and incubated for 48 h. Then, the wool samples (1 inch ×1 inch) were soaked in 2 mL culture medium for 24 h. The cultured medium with leaching substance was added to the cells and incubated for 24 h. The test samples were removed from the cell cultures, and the cells were reincubated in a fresh medium. After incubation for 24 h, the cell viability was determined using the MTT assay [26]. At least three data were averaged.

3. Results and discussion

3.1. FE-SEM images, EDS spectra and X-ray mapping images

In order to study the morphology of treated and untreated wool samples, a field emission scanning electron microscopy (FE-SEM) was used. The FE-SEM images of blank wool (A), wool treated with ZnO nanoparticles (B and C), wool treated with graphene oxide (D), and wool treated with graphene/ZnO nanocomposite (E-G) are illustrated in Figure 1. The wool-treated fabric with graphene oxide surface

displayed some corrugations, while the raw wool fabric surface had been covered with scales, overlapping each other. It reveals the presence of graphene oxide sheets on the wool fabrics. Also, it is possible to recognize the ZnO nanoparticles on the surface of wool fabric treated with nano-zinc oxide *by* comparing Figure 1(A) with Figure 1(B and C). The FE-SEM images of graphene/ZnO-coated wool fabric confirmed the presence of zinc oxide nanoparticles on the graphene sheets (Figure 1(G-I)). They reveal that graphene is decorated with zinc oxide nanoparticles densely. The average sizes of the ZnO nanoparticles are in the range of 11–18 nm. Figure 2A shows the EDS spectrum of treated wool fabric with reduced graphene oxide/ZnO nanocomposite. The results show the presence of C, O, S, and Zn in the sample, which further demonstrated the successful formation of graphene/ZnO nanocomposite on the wool surface. Further, the distribution of C, O, and Zn elements in treated wool samples with reduced graphene oxide/ZnO nanocomposite was studied by elemental mapping. As shown in Figure 2B, the distribution of zinc atoms for coated wool with reduced graphene oxide/ZnO nanocomposite is uniform.

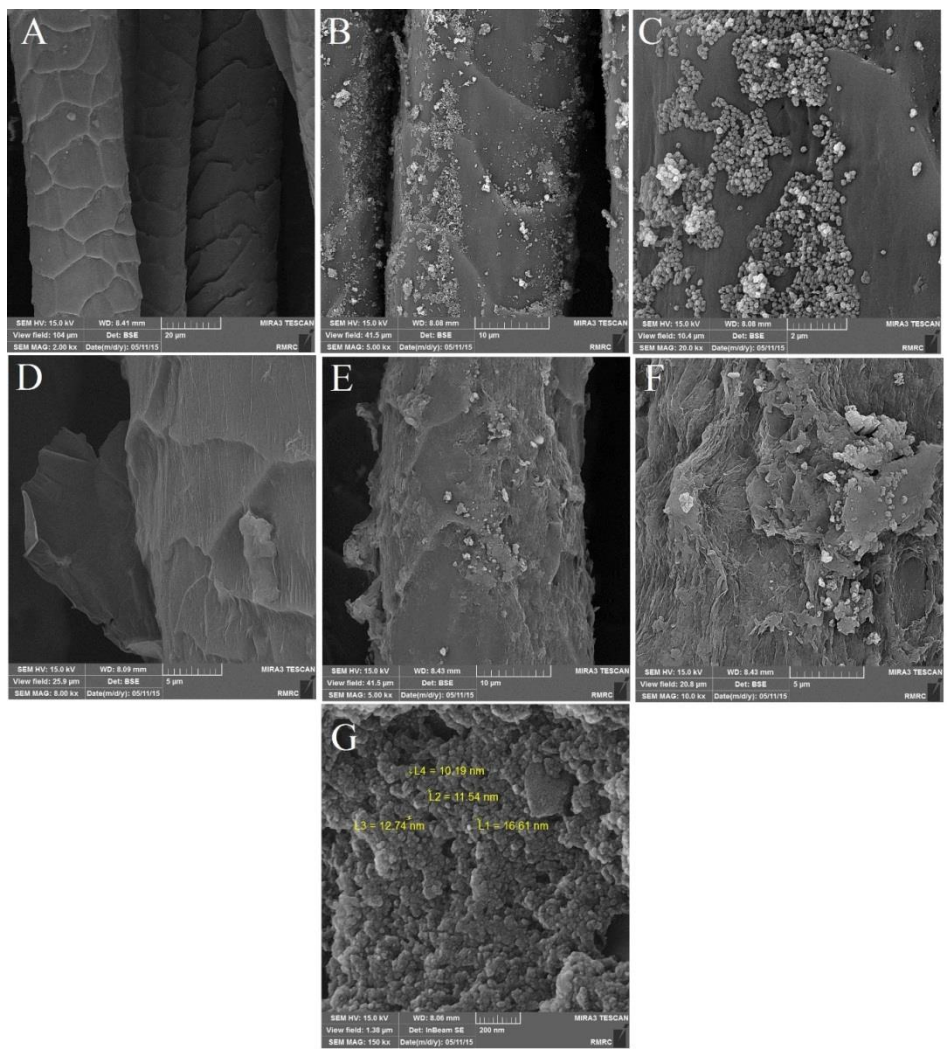

Figure 1. FE-SEM images of various wool samples: (A) raw, (B and C) treated with zinc oxide, (D)

treated with graphene oxide, (E-G) treated with reduced graphene oxide/ZnO nanocomposite>

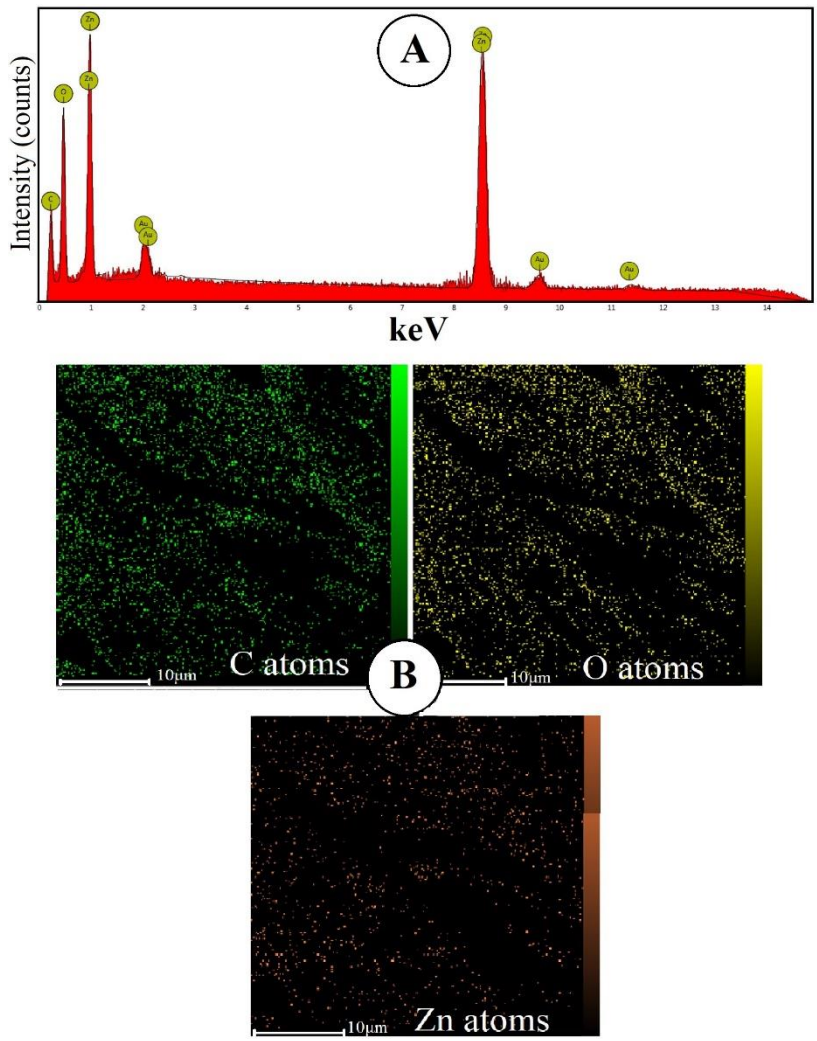

Figure 2. EDS spectrum (A) and X-ray mapping images (B) of treated wool fabric with ZnO/reduced graphene oxide nanocomposite (4 % wt. zinc acetate)>

3.2. XRD pattern

The crystalline status of the reduced graphene oxide/ZnO over wool fabrics was studied by XRD. The XRD pattern of coated wool fabric using reduced graphene oxide/ZnO nanocomposite is shown in Figure 3. Two major peaks can be observed around 2θ=14.4˚ and 23.2˚ related to diffraction peaks of wool. In the case of ZnO/reduced graphene oxide nanocomposite, there are seven peaks at 31.9º, 34.6º, 36.5º, 47.7º, 56.8º, 63.1º, and 67.1º, corresponding to the (100), (002), (101), (102), (110), (103), and (202) planes of quartzite zinc oxide (JCPDS Card No. 36-1451). Also, no obvious diffraction peaks of graphene can be seen, indicating

the absence of layer-stacking regularity or the relatively low content of graphene on the wool surface. In addition, the crystal size was calculated, and for reduced graphene oxide/ZnO nanocomposite treated wool was 117 Å.

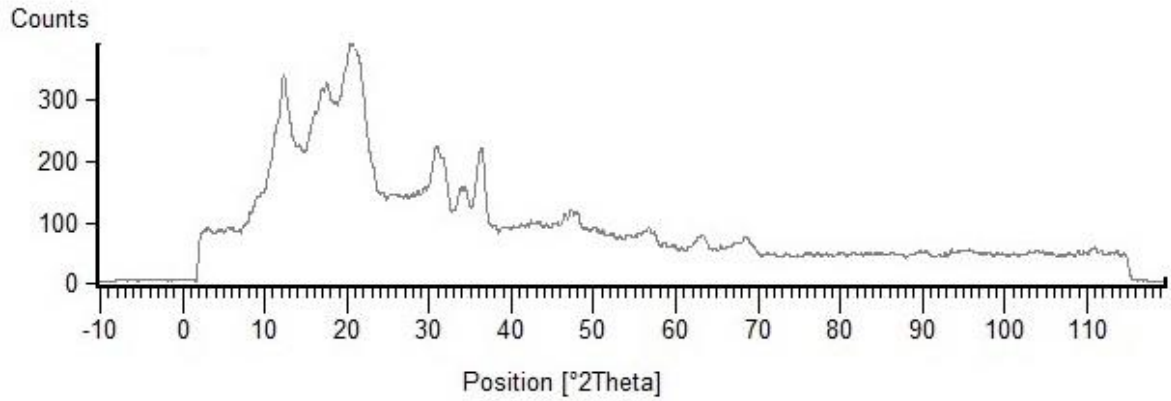

Figure 3. XRD pattern of treated wool fabric with ZnO/reduced graphene oxide nanocomposite.

Table 1. Electrical resistance of various wool fabrics

Sample	zinc acetate (% wt.)	Electrical resistivity (Ω sqr^{-1})
Blank wool	-	1.1×10^{9}
Zinc oxide treated wool	3	1.6×10^{9}
Graphene oxide treated wool	-	2×10^{9}
Reduced graphene oxide treated wool	-	3.4×10^{3}
Nanocomposite treated wool	1	3.6×10^{3}
Nanocomposite treated wool	2	4.1×10^{3}
Nanocomposite treated wool	3	6.4×10^{3}
Nanocomposite treated wool	4	7.9×10^{3}
Nanocomposite treated wool	5	8.2×10^{3}

3.3. Electrical properties

The blank wool, ZnO-treated wood, and graphene oxide-treated wool fabrics were not electroconductive (electrical resistance in the range of 1 × 109 - 2 × 109 ohm/square). After the reduction of graphene oxide (or graphene oxide/ZnO) coated -wool samples by sodium hydroxide, the electrical resistance of the samples was reduced. In other words, its electrical conductivity increased, which indicated the effective reduction of oxygen functional groups from the graphene oxide. The electrical resistance variation of treated wool samples

is presented in Table 1. Based on the obtained results, after the reduction of graphene oxide coated wool fabrics by sodium hydroxide, the insulated fabric turned electrical conductive (electrical resistivity in the range of 3.4×10^{3}-8.2×10^{3} Ω sqr^{-1}), which indicated the effective reduction of oxygen functional groups from the graphene oxide. Also, by increasing the amount of zinc acetate, the electrical conductivity decreases, which is due to the aggregation of zinc oxide nanoparticles on graphene sheets. The condensed deposition of nano-ZnO results in charge transfer to the graphene and a reduction of graphene mobility by charged impurity scattering [27].

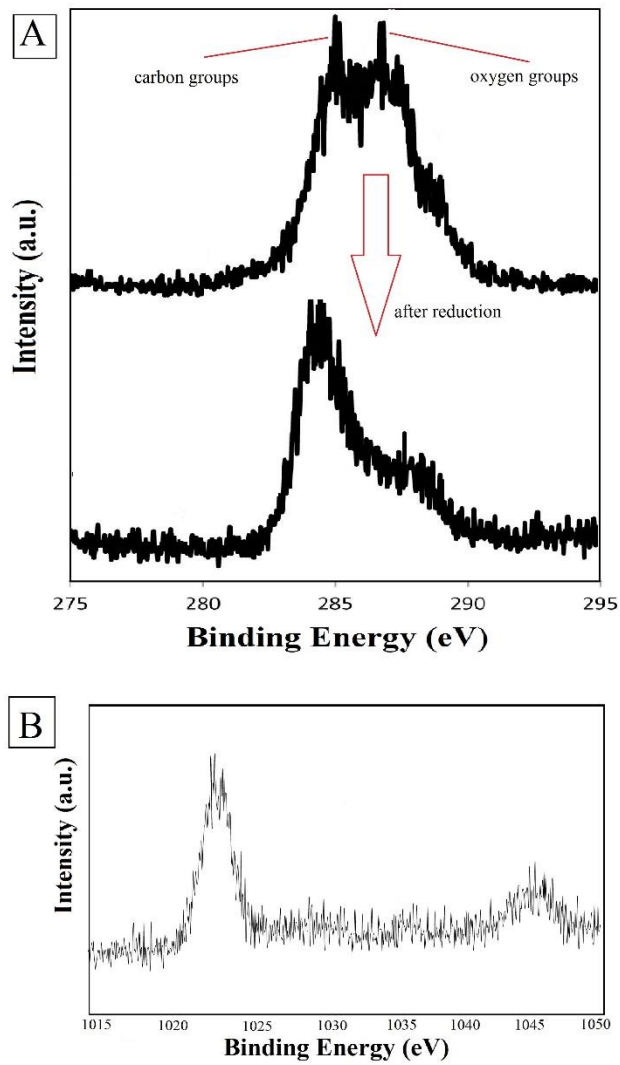

Figure 4. XPS spectra of treated wool fabrics: (A) C 1s core level spectra of treated wool with graphene oxide and ZnO/reduced graphene oxide nanocomposite (B) Zn 2p core level spectrum of treated wool with ZnO/reduced graphene oxide nanocomposite (4 % wt. zinc acetate).

The reduction of the oxygen-containing groups in graphene oxide by sodium hydroxide was studied by X-ray photoelectron spectroscopy (XPS). Figure 4A clearly shows that the O/C ratio in the ZnO/reduced graphene oxide nanocomposite -treated -fabric decreases remarkably compared with that of the graphene oxide- treated- fabric and that most of the oxygen groups were successfully removed. Therefore, we can infer that the graphene oxide was effectively reduced by sodium hydroxide. In the Zn 2p spectrum (Figure 4B), the peaks for Zn 2p3/2 and Zn 2p1/2 were observed at 1022 eV and 1044.9 eV. It also indicates the formation of zinc oxide. Consequently, the results established the successful preparation of the ZnO/reduced graphene oxide nanocomposite on the fabric surface.

3.4. Photocatalytic activity

The photocatalytic activities of wool samples were calculated using the decomposition rate of the methylene blue dye solution under sunlight irradiation. The photo-activity efficiencies of the samples are shown in Figure 5. Figure 5 shows that the methylene blue concentration change was negligible during irradiations in contact with blank wool. Loading of graphene oxide in wool fabric led to a decrease in methylene blue concentration due to the adsorption of methylene blue by graphene sheets. Therefore, the blank wool and the graphene oxide-coated wool fabrics show no photocatalytic activity under sunlight irradiation. The treated wool samples with zinc acetate showed higher dye decomposition ability. Also, more zinc acetate led to better photocatalytic activities (E%), possibly due to more ZnO nanoparticles absorption by the fabric. When the photocatalyst is illuminated by light with energy higher than its bandgap energy, electron-hole pairs diffuse out to the surface of the photocatalyst. The created negative electrons and oxygen combine into $O2^{-}$, and the positive electric holes and water generate hydroxyl radicals. This highly active oxygen species can oxidize organic pollutants. Thus, nano zinc oxide can decompose the available organic matter, dye molecules, bacterial cell membranes, *etc*. [28].

As shown in Figure 5, the nanocomposite-coated samples confirmed more photocatalytic degradation of methylene blue in comparison with nano ZnO-coated wool fabrics. It is because graphene in reduced graphene oxide/ZnO promotes the separation of electron–hole pairs and extends the life of electrons. Also, the adsorption of dye molecules is helpful for the increase of photocatalytic activity. Furthermore, the extended light absorption ability of reduced graphene oxide/ZnO improves photocatalytic performance [29].

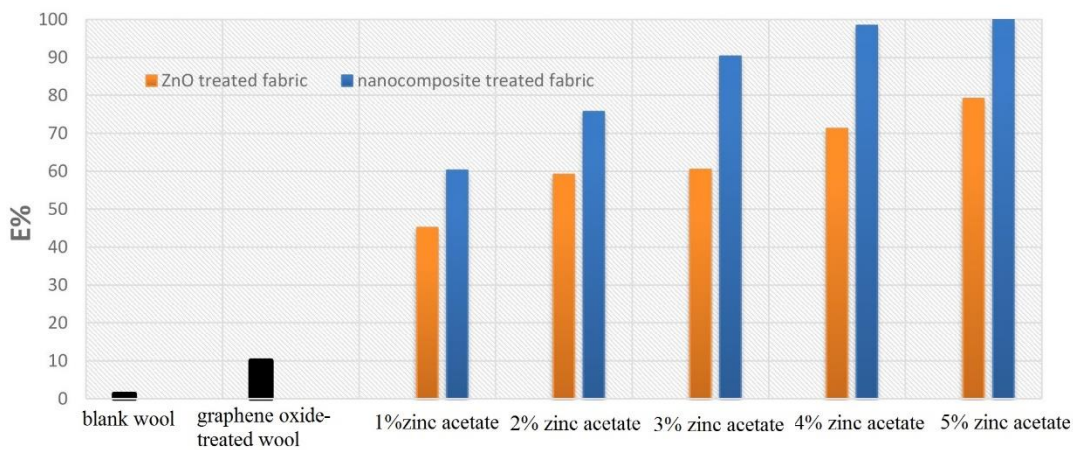

Figure 5. Comparative diagram of photocatalytic performance results of the wool samples.

3.5. Antibacterial property and Cell viability

The antibacterial activities of the samples were evaluated quantitatively by suspension method against both *E. coli* and *S. aureus* bacteria (Figure 6). There were some colonies of viable bacteria seen on agar plates of the blank wool. The nano-ZnO-treated wool sample exhibited 69% reduction for *S. aureus* and 75% for *E. coli*. The ZnO/reduced graphene oxide nanocomposite treated wool indicated higher antibacterial activity than nano-ZnO treated fabrics. The nanocomposite treated sample exhibited 100% reduction for *S. aureus* and *E. coli*. The bacteriostatic activity of reduced graphene oxide is attributed to either physical interaction with the bacteria or oxidative stress of the cell membranes that disrupts the membranes integrity [30, 31]. The ZnO-treated textile can inhibit the growth of bacteria, possibly by two mechanisms. The first important reason is the production and penetration of reactive oxygen species. The hydroxyl radical and hydrogen peroxide can penetrate the cell membranes, which leads to the death of bacteria. The second reason is Zn^{2+} ions in the bacterial culture. These positive ions react with the negatively charged ions on the cell surface of microorganisms, resulting in a variation of cell permeability, transforming the normal metabolism of microorganisms and finally leading to microorganism death [11]. These mechanisms of reduced graphene oxide and ZnO nanoparticles complemented each other and resulted in the significant antibacterial activity of the ZnO/reduced graphene oxide nanocomposite on wool fabric.

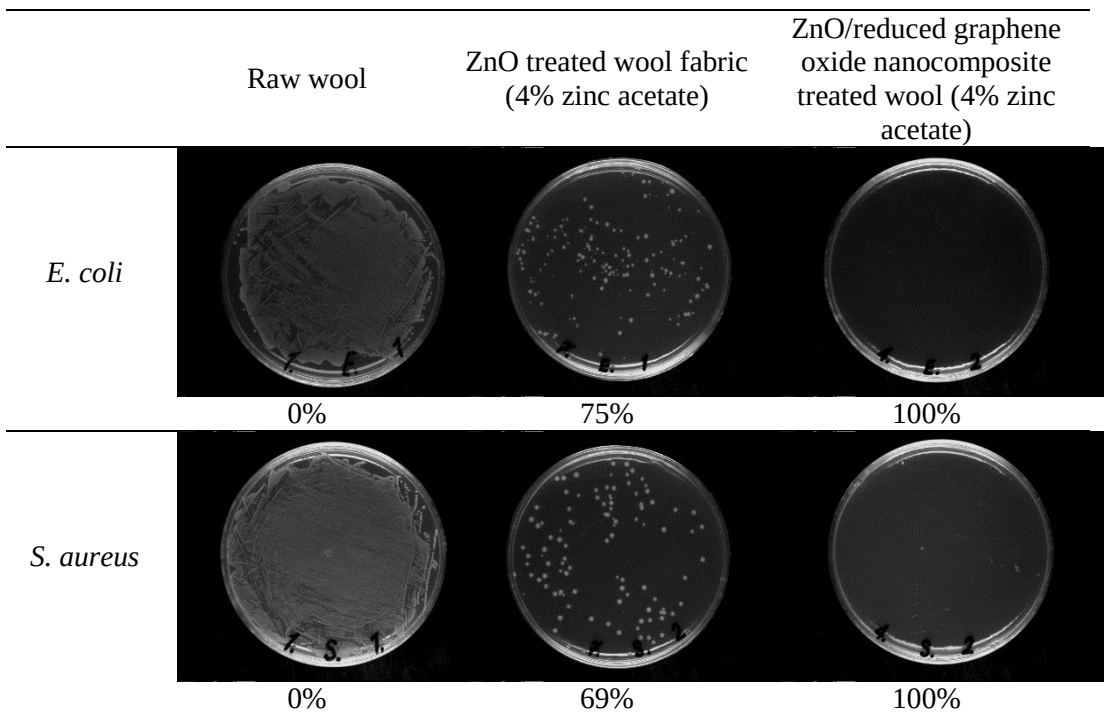

Figure 6. The antibacterial efficiency of the wool samples.

Cytotoxicity test of the ZnO/reduced graphene oxide nanocomposite treated wool fabric and blank wool fabric was examined by MTT assay. The cell viability and morphology of human fibroblast cells are presented in Figure 7. Based on the obtained results, the treated fabrics with ZnO/reduced graphene oxide

nanocomposite have low cytotoxicity, similar to the blank samples. Moreover, the morphology of fibroblast cells in the presence of the treated wool fabric is normal, which is similar to the morphology of the blank wool sample. Therefore, no significant cytotoxicity effect was established by the nanocomposite-treated fabrics on human skin.

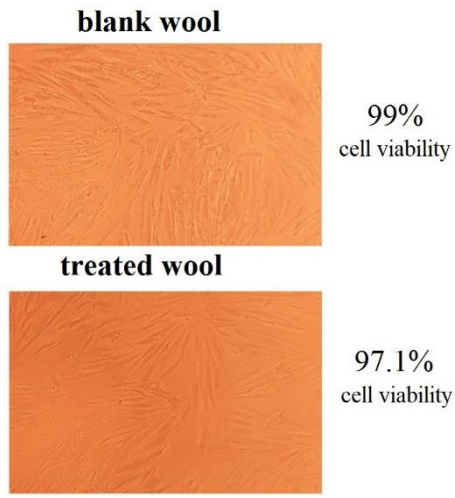

Figure 7. Cell viability properties of the untreated and ZnO/reduced graphene oxide nanocomposite treated wool fabrics.

3.6. UV blocking

The UV transmittance spectra of blank wool fabric and treated wool fabric with ZnO/reduced graphene oxide nanocomposite (in the range of 280–400 nm) were illustrated in Figure 8. The upper line is the UV transmittance spectrum of the raw wool sample, demonstrating that a high percentage of UV light can penetrate the fabric. The ZnO/reduced graphene oxide nanocomposite-treated wool fabric had much lower UV transmittance compared to the raw wool fabric. Consequently, the treated wool fabrics with ZnO/reduced graphene oxide nanocomposite have a UV-blocking appropriate property due to the synergetic UV absorption of zinc oxide and reduced graphene oxide [3].

4. Conclusions

For the first time, we have successfully synthesized ZnO/reduced graphene oxide nanocomposites on wool fabrics as a novel finishing technology. By incorporating ZnO/reduced graphene oxide nanocomposites into wool fabrics, functional properties such as self-cleaning, antibacterial effect, low cytotoxic electrical conductivity, and higher UV radiation were achieved. FE-SEM and X-ray mapping images, XPS spectra, XRD and EDS patterns confirmed the presence of ZnO/reduced graphene oxide nanocomposites on the surface of the treated wool fabrics. All properties of the ZnO/reduced graphene oxide nanocomposite treated wool were superior compared to nano zinc oxide treated wool alone. ZnO/reduced graphene oxide nanocomposites are expected to be used in the fabrication of high-performance fibers and smart textiles.

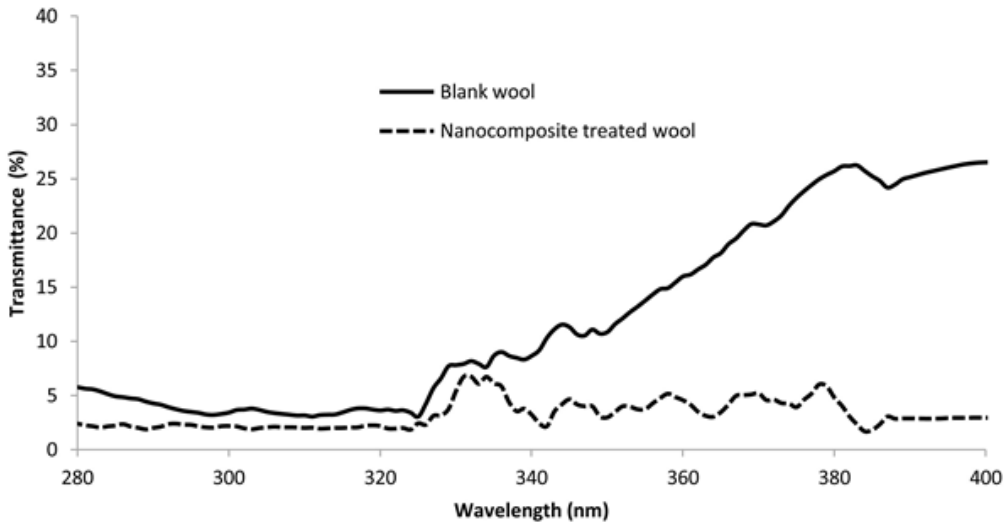

Figure 8. UV transmittance spectra of blank wool fabric and treated wool fabric with ZnO/reduced graphene oxide nanocomposite.

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