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Synthesis of nano-zinc oxide with different morphologies and its application on fabrics for UV protection and microbe-resistant defense clothing

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Abstract

A liquid precipitation method was used to prepare zinc oxide nanoparticles in three diverse media: water, methanol, and ethylene glycol. The studied materials were examined by scanning electron microscopy, X-ray diffraction, Fourier transform infrared spectroscopy, and ultraviolet-visible spectroscopy. X-ray diffraction patterns showed a hexagonal Wurtzite structure of zinc oxide with a nanocrystalline size. Acquired powders showed different morphologies (rod, star, and spherical structures), which were affected by the nature of the solvent in the reaction. The different zinc oxide powders have varied optical band gaps. Scanning electron microscopy examinations confirmed the arrangement of nano-zinc oxide on the surfaces of the materials. The zinc oxide-covering procedure was carried out on cotton, polyester, and 50/50 wt% polyester/cotton blended fabrics using a simple dip and curing system. The cotton fabric treated with nanorod zinc oxide exhibited the highest ultraviolet protection factor with a value of 247.2. The antimicrobial properties of untreated and treated fabrics with nano-zinc oxide were measured against Gram-negative bacteria *(Escherichia coli)*, Gram-positive bacteria *(Staphylococcus aureus)*, and diploid fungus *(Candida albicans)*. The results showed the antimicrobial action relies on the morphological structure and the particle size of zinc oxide and that it increases with a reduced particle size. The cotton fabric treated with 26 nm nonspherical zinc oxide particles showed the highest antimicrobial efficiency with values of 91.4%, 86.8%, and 84.7% for *Staphylococcus aureus, Escherichia coli*, and *Candida albicans*, respectively. The mechanical properties of treated fabrics were studied. The results confirm that nano-zinc oxide is highly useful for improving the performance of defense textile products because of its biocompatibility, environmental friendliness, and nontoxicity.

Keywords

ZnO nanoparticles, fabrics, UV protection, microbe resistance, defense clothing

Defense textiles have an extraordinary potential for cross-infection because they are often used by groups living in numerous camps and risky climates. The principal challenges faced by the defense clothing industry are not only inventing and improving the current functionalities of textiles,[1–6] but also preservation of the look, flexibility, comfort, washability, and feel of the fabric.[7–11] To protect defense personnel from microbes and cross-infection, a special antimicrobial finish has become a necessity on clothing.[1,2] It is predicted that defense clothes used in military applications will be able to help examine the hazards of the wearer's environment and provide permanent updates on their health status. Cotton is the most commonly used fabric in the manufacture of defense clothing.[12]

The use of nanomaterials in modern textiles lies in areas where innovative principles will be combined into

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long-lasting, multifunctional textile structures without compromising the intrinsic textile properties such as aesthetics, breathability, flexibility, etc.[13] Coating fabrics with nanoparticles is important because they improve durability by penetrating the fabric surface due to their small size and high surface to volume ratio.[14] Incorporating nanomaterials into a textile can affect a host of properties, including shrinkage, strength, antimicrobial qualities, and ultraviolet (UV) protection.[1,2,5,6] The adhesion strength of nano-particles to the fabric they cover depends on the morphological structure and the stabilization of the covered materials in the washing system.

Zinc oxide (ZnO) is a chemical agent used in defense clothing because of its unique chemical and physical characteristics, biocompatibility, environmental friendliness, nontoxicity, and low cost. ZnO can also provide textiles with excellent UV blocking ability owing to its high intrinsic optical band gap energy (3.3 eV), which lies in the UV range.[15–17]

To our knowledge, the dielectric constant of a solvent influences the kinetics of nucleation and the growth of crystallites during their formation. Therefore, the preparation of ZnO in different solvent media with different dielectric constants can produce ZnO with varied morphological structures. Accordingly, the present study is mainly designed to prepare the distinctive morphology of nano-ZnO and investigate the effects on the antimicrobial and UV-protection capacities of particular fabrics (cotton, polyester, and 50/50% cotton/polyester). Three different morphological ZnO nanoparticles have been synthesized in three different media: water, methanol, and ethylene glycol. The UV protective properties of the ZnO-treated textiles are measured by their UV protection factor (UPF). Moreover, we studied the antimicrobial action of the nanoparticles on fabrics against Gram-negative bacteria (*Escherichia coli*), Gram-positive bacteria (*Staphylococcus aureus*), and diploid fungus (*Candida albicans*). The improvement in the physical properties of the fabrics treated with different morphological ZnO nanoparticles opens up the possibility of protecting the human body from disease and solar radiation, and has uses in other technological applications.

Experiments

Materials

All chemicals utilized in the experiments were of analytical reagent grade. Sodium hydroxide (NaOH, 99%) pellets were purchased from Sigma Pharmaceuticals and zinc acetate dihydrate (99%) from El Nasr Pharmaceutical Chemicals (Egypt). Ethylene glycol (90%) was purchased from Nice Chemicals (India), methanol (95%) was purchased from El Nasr Pharmaceutical Chemicals. Oxoid dehydrated agar was used to prepare culture media following the manufacturer's instructions. Preparation of all microbiological media was done using deionized water. Three fabrics (100% cotton, cotton/polyester 50/50 wt%, and polyester) were purchased from the local market as an Egyptian cotton product, with using the technical specifications shown in Table 1.

ZnO synthesis in different media

The ZnO nanoparticles were synthesized in three different media: de-ionized water, methanol, and ethylene glycol. The used preparation method was described by Lee et al.[18] but with some modifications. Different solutions of zinc acetate dihydrate (0.01 mol/50 mL) with different solvents, methanol, water, and ethylene glycol, were kept at temperatures of 50, 90, and 120°C, respectively. Then 20 mL of 1 M NaOH solution (dissolved in the above-mentioned solvents) was added under flow control (4 mL/min) and slow magnetic stirring at the mentioned temperatures.

The reaction mixture was maintained at those temperatures for 1 hour then cooled to room temperature with continuous stirring (700 rpm). The precipitate was immediately filleted by centrifugation then washed with the appropriately mentioned solvents and subsequently dried under an infrared lamp (Infrared Halogen Lamp, 5 cm height, 400 W, for 15 min). The samples prepared were denoted by Z_w, $Z_{m,}$ and Z_e for those in water, methanol, and ethylene glycol, respectively. The synthesis efficiency and yield of ZnO were found to be 1.22, 1.27, and 1.39 g ZnO/100 g of the reaction mixture with high yields of 89, 92, and 98% for $Z_{m,}$ Z_w, and Z_e, respectively.

Fabric treatments

The fabrics under study were first washed with distilled water and detergent (2 g/L of sodium lauryl sulfate) at

Table 1. Technical specifications of the fabrics

Specification	100% cotton	50/50% polyester/cotton	Polyester
Structure	Plain weave	Plain weave	Plain weave
Width (cm)	123	120	143
GSM	150	135	200
Ends/cm	39	37	56
Picks/cm	29	31	34
Warp count	1/40°	1/40°	–
Weft count	1/40°	1/40°	–

GSM: Grams per square meter.

70°C for 30 min to remove lubricants, waxy matter, fats, etc. before being washed a few times with deionized water. ZnO nanoparticles were loaded on the studied fabrics using the pad-dry-cure method. The fabric was cut to a size of 20 × 10 cm and was immersed in the suspension containing ZnO (2 wt%) and Amsterdam Grounds – Acrylic Binder (1 wt%) for 5 min, at a material to liquor ratio of 1:20, then was passed through a padding mangle. The excess suspension was squeezed using a padding mangle, which ran at a speed of 15 m/min and a pressure of 15 kg/cm^2. After padding, the fabric was air-dried and then cured for 3 min at 140°C then immersed for 5 min in 2 g/L of sodium lauryl sulfate to remove unbound nanoparticles. The fabric was then rinsed with distilled water at least 10 times to remove all the soap solution and finally air-dried.[19] Simultaneously, bulk-ZnO coating was carried out for comparison.

Characterization methods

The materials investigated were characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and transmission electron spectroscopy (TEM). XRD patterns were obtained at room temperature using a Philips X'Pert Pro Super diffractometer equipped with a Cu-Kα ($\lambda = 1.54$ Å) source (connected voltage 45 kV, current 40 mA) within the range of 2θ of 10–70°. Infrared absorbance measurements were carried out on a BRUKER FTIR (vector 22), in the range of 400–4000 cm^{-1}. TEM was used to determine the shape and size of the obtained nanoparticles, utilizing a Philips EM201C device working at 80 kV. SEM analyses were done by using a JEOL-JEM100CXII unit.

The amount of ZnO in treated fabrics was determined by complexometric titration with a 0.1 M aqueous solution of ethylenediaminetetraacetic acid disodium salt (Aldrich, Steinheim, Germany). The ZnO on the fabric surface was dissolved in 18 wt% hydrochloric acid before analysis. An average amount of 0.34, 0.30, and 0.23 mg ZnO/cm^2 were obtained for cotton, cotton/polyester, and polyester, respectively.

For the mechanical investigation, two sets of test specimens, one in the weft and other in the warp direction, were tested. Each set consists of at least a four-test specimen; the dimensions of each are 50 mm in width and 150 mm length. The tensile strength of fabric was determined along with standard test procedure ISO 13934-1 (1999).[20]

The UPF was estimated for all textiles tested after exposure to a relative humidity of 65% for 24 hours. Measurements were performed using a Cary 50 UV/Visible spectrophotometer (Varian; Palo Alto, CA). During the estimation, four scans were attained by turning the specimen 90° every time, and the spectral data were recorded as the normal of these four outputs. The UPF values were calculated using the mean rate of transmission in the UVB range (295–315 nm) and the mean rate transmission as part of the UVA locale (315–400 nm) according to the following equation:[21]

$$\mathrm{UPF} = \frac{\sum_{\lambda=290}^{\lambda=400} \mathrm{E}(\lambda) \times \mathrm{S}(\lambda) \times \Delta\lambda}{\sum_{\lambda=290}^{\lambda=400} \mathrm{E}(\lambda)\mathrm{T}(\lambda) \times \mathrm{S}(\lambda) \times \Delta\lambda} \quad (1)$$

Where E(λ) is the solar irradiance (W/m^2) measured, $\varepsilon(\lambda)$ is the erythemal action spectrum, $\Delta\lambda$ is the wavelength interval of the measurements, and T(λ) is the spectral transmittance of the specimen obtained from the spectrophotometric experiments.

The antimicrobial capability of tests were decided subjectively against a Gram-positive organism (*S. aureus*, ATCC 6538), Gram-negative bacteria (*E. coli*, ATCC 8739), and a diploid fungus (*C. albicans*). The bacteria were set in 5 mL supplement stock and incubated at 37°C for 24 hours. Agar Petri dishes were inoculated with the grown developed bacteria. Uncoated and ZnO-coated fabrics were carefully squeezed to the focal point of media culture. The Petri dishes were incubated for 24 hours at 37°C, and the inhibition zone was observed.[22]

In the quantitative test, the circular coated and uncoated fabric samples with a diameter of 4 cm were tested by the reduction of colony-forming units (CFU). Firstly, the control specimen and coated fabrics were sterilized in an autoclave. The diluted bacterial suspension (1.2×10^5 CFU/mL) and the fabrics were added to an Erlenmeyer flask (150 mL). This was then shaken at 300 r/min and 37°C for 1 hour. After that, 0.5 mL of bacteria were collected from each Erlenmeyer flask and diluted 10, 100, and 1000 times, respectively. The diluted bacteria suspensions were plated in the agar medium incubation at 37°C for 48 hours, and the number of CFU then counted. The data obtained are the average values of two parallel runs, and the reduction percentage of bacteria (R) was calculated by Eq. 2.

$$\mathrm{R}(\%) = [(\mathrm{B} - \mathrm{A})/\mathrm{B}] \times 100 \quad (2)$$

Where A is the number of bacterial colonies from the treated specimen after inoculation over a 24-hour contact period and B is the number of bacterial colonies from untreated control specimens after inoculation.

Results and discussion

XRD

The XRD patterns of the synthesized ZnO in diverse media are shown in Figure 1. The diffraction peaks situated at angles $2\theta = 31.8°$, 34.6°, 36.5°, 47.7°, 56.6°, 62.9°, 68.1°, and 69.2°, which correspond to (100), (002), (101), (102), (110), (103), (112) and (201) lattice planes, respectively (given the peak values), revealing the prepared samples have a Wurtzite (hexagonal, space group p63mc and JCPDS no. 36-1451) structure with lattice constants 0.32 nm and 0.52 nm.[23,24] The absence of any additional XRD peaks other than ZnO indicates the prepared samples are free of impurities.

The peak intensity of the (100) lattice plane of the Z_m sample is larger than those obtained in other samples, referring to rod-like or needle-like morphology for the Z_m sample.[25]

The crystallite sizes of the prepared specimens were estimated from line widening as indicated by the Scherrer's equation: $D = 0.9\lambda/\beta \cos \theta$,[26] where λ is the wavelength of the incident X-ray beam (1.54 Å for the Cu Kα), θ is the Bragg diffraction angle, and β the width of the X-ray pattern line at half peak height in radians. The results are listed in Table 2.

In contrast, the diffraction patterns of raw cotton fabrics and polyester in comparison with textiles treated with ZnO are shown in Figure 2. The diffraction patterns of raw cotton fabrics (Figure 2(a)) show four peaks at $2\theta = 14.62°$ (101), 16.29° (101), 22.48° (200), and 44.7° (040), confirming the cellulose structure of cotton.[27–29] The diffraction pattern of polyester (Figure 2(b)) shows a broad diffraction hump at $2\theta = 17–28.9°$, indicating the amorphous nature of the polyester. The diffraction pattern of polyester/cotton fabric shows the main peaks at 2θ angles of 14, 16, 22 (main), 24, 33, and 34°.[30,31] In contrast, the XRD patterns of the treated textiles revealed the presence of peaks at $2\theta = 31.8°$, 34.6°, 36.5°, and 47.7° in comparison to the untreated sample indicative of the presence of the crystalline ZnO on the tested fabrics.

FTIR characterization

The FTIR spectra for synthesized ZnO, control fabrics, and ZnO-coated fabrics recorded in the range of 400–4000 cm^{-1} are shown in Figure 3. The FTIR spectra of

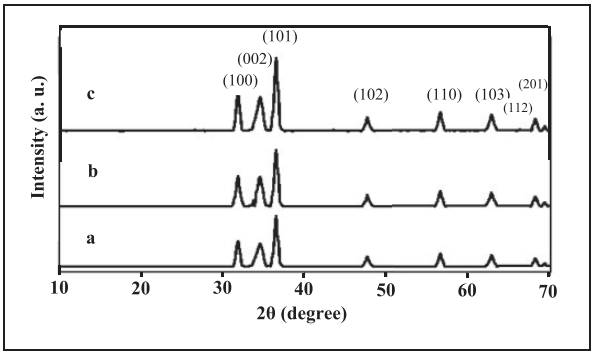

Figure 1. X-ray diffraction (XRD) patterns of zinc oxide (ZnO) samples: ZnO in water (Z_w) (a); ZnO in ethylene glycol (Z_e) (b); and ZnO in methanol (Z_m) (c).

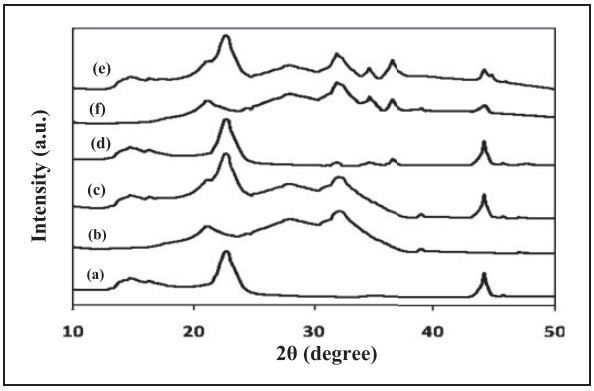

Figure 2. X-ray diffraction (XRD) patterns of treated and untreated textiles: (a) cotton (Co); (b) polyester (PET); (c) ZnO in water (Z_w/Co); (d) Z_w/PET; (e) Co/PET; and (f) Z_w/Co/PET.

Table 2. XRD, TEM, FTIR, and optical data for the ZnO samples synthesized

Sample	XRD (crystallite size), nm	TEM (crystallite size), nm	Morphological structure	FTIR cm^{-1}	Absorption wavelength, nm
Z_w	34	31	Star	470	328
Z_m	28	Diam. 25 nm Long 200 nm	rods	465	318
Z_e	22	26	sphere	460	366

FTIR: Fourier transform infrared spectroscopy; TEM: transmission electron spectroscopy; XRD: X-ray diffraction; ZnO: zinc oxide; Z_w: ZnO in water; Z_m: ZnO in methanol; Z_e: ZnO in ethylene glycol.

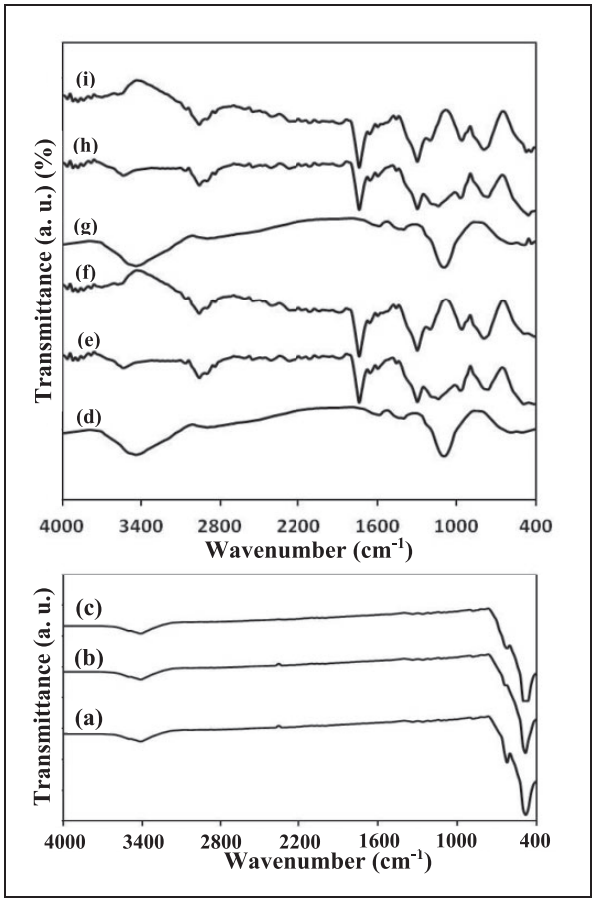

Figure 3. Fourier transform infrared spectroscopy (FTIR) of the investigated zinc oxide (ZnO) samples: (a) ZnO in water (Z_w); (b) ZnO in methanol (Z_m); (c) ZnO in ethylene glycol (Z_e); (d) cotton (Co); (e) polyester (PET); (f) Co/PET; (g) Z_W/Co; (h) Z_W/PET; and (i) Z_W/Co/PET.

ZnO samples exhibit the characteristic vibrational mode of Zn-O at ~465 cm^{-1}.[32,33] The FTIR spectrum of the cotton fabric shows the stretching vibration modes of C-O, and C=O in the region of 1200 to 1700 cm^{-1}, whereas the appearance of a peak at 2920 cm^{-1} is assigned to the C-H stretching peak.[34] The FTIR spectrum of polyester fabric reveals the characteristic peaks in the range of 1750 cm^{-1} due to the ester carbonyl peak.[35] The peaks observed in the range of 1339–1016 cm^{-1} are assigned to the stretching peaks of C-O. The peak at 969 is due to the Trans C-O stretching and vibrations of ester group.[36] The peak observed at ~870 cm^{-1} is assigned to the stretching vibrations of aromatic ring.[36] The Attenuated total reflection- Fourier transform infrared spectroscopy (ATR-FTIR) spectrum for both the uncoated fabrics and ZnO-coated fabrics exhibit almost similar peak positions, except for a slight change in peak observed at 460–470 cm^{-1} corresponding to the Zn-O stretching vibration. The slight change observed in each of the intensity and peak positions of ZnO bands refers to the adhesion occurring between ZnO and the studied fabrics. The main FTIR peaks acquired are listed in Table 2 and Table 3.

Table 3. XRD, FTIR, and adhesion strength data for the treated fabrics

Sample after five washing cycles	XRD (crystallite size) nm	FTIR cm^{-1}	Adhesion weight mg/cm^2	Weight loss (W%)
Zw/Co	36	470	0.35	12.7
Zm/Co	31	466	0.37	11.1
Ze/Co	26	466	0.34	14.5
Zw/Co/PET	38	467	0.29	15.7
Zm/Co/PET	33	468	0.32	14.5
Ze/Co/PET	29	465	0.27	17.7
Zw/PET	29	469	0.23	18.6
Zm/PET	31	467	0.25	17.7
Ze/PET	28	465	0.22	19.5

Co: cotton; FTIR: Fourier transform infrared spectroscopy; PET: polyester; XRD: X-ray diffraction; ZnO: zinc oxide; Z_w: ZnO in water; Z_m: ZnO in methanol; Z_e: ZnO in ethylene glycol.

TEM and SEM

The TEM images of ZnO particles obtained in distinctive media are shown in Figure 4. It can be seen that the morphology and size of the particles were greatly affected by the experimental conditions (Table 2). The ZnO prepared in aqueous medium shows star-shaped particles (Figure 4(c)). However, the sample prepared in methanol exhibits a nanorod shape with a length of 250 nm and an average diameter of 25 nm (Figure 4(b)). The TEM image of the ZnO sample prepared in ethylene glycol demonstrates particles with a spherical shape with an average diameter of 26 nm (Figure 4(a)).

These results reveal the structure of the solvent prompted morphology control contingent on its interaction with the growing crystals. The structure and bulkier functional groups (OH) of ethylene glycol compared with methanol restrict the growth of all crystal planes, thus generate a more uniform dimensional shape, such as a sphere. In contrast, methanol, which has a less complicated structure, permits the growth of some crystal planes, consequently giving a nanorod structure.

Figure 5 demonstrates the SEM micrographs for the untreated and ZnO-treated fabrics individually. It was observed that the uncoated cotton, polyester, and cotton/polyester fabrics have fibrils and smooth surfaces (Figure 5(a), (b), (c)). In contrast, the surface morphology of the treated polyester fabrics was covered with ZnO nanoparticles as shown in Figures 5(e), (h), and (k). It also showed good dispersion of ZnO

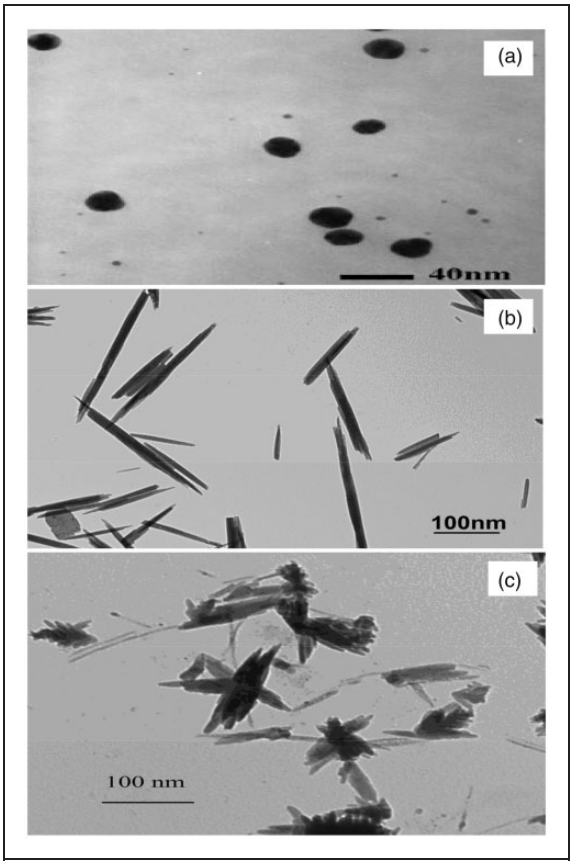

Figure 4. Transmission electron spectroscopy (TEM) of the investigated zinc oxide (ZnO) samples: ZnO in ethylene glycol (Z_e) (a); ZnO in methanol (Z_m) (b); and ZnO in water (Z_w) (c).

nanoparticles on the surface with various morphologies for the different Z_w, Z_m, and Z_e samples. The surface morphology of the treated cotton/polyester fabrics is illustrated in Figures 5(f), (i), and (l).

To evaluate ZnO nanoparticle adhesion to the textile fabrics, a laundering test was done according to the American Association of Textile Chemists and Colorists (AATCC) 124-1996 standard test method. For each treated fabric, 10 washing cycles were performed at 50°C for 30 min. The weight loss percentage (W%) of the fabric was then determined using equation (3):

$$W\% = [(w_1 - w_2)/w_1] \times 100 \quad (3)$$

where W_1 and W_2 are the weights of the fabrics before and after washing, respectively. The results obtained are listed in Table 3, which shows the adhesion between ZnO and the fabrics increase in the order: $Z_m > Z_w > Z_e$.

Optical properties

The optical absorption properties of ZnO depend on its intrinsic optical band gap energy (E_g), which is around 3.3 eV (375 nm) for bulk ZnO.[37] The UV-visible spectra of the prepared ZnO samples are demonstrated in Figure 6. The spectra show a strong absorption band at λ_{max} of 328, 318, and 366 nm for Z_w, Z_m, and Z_e, respectively. The E_g values of the nano-ZnO samples were estimated using the equation $E = 1240/\lambda_{max}$ and were found to be 3.87, 4.0, and 3.38 eV, for Z_w, Z_m, and Z_e, respectively, which are larger than the bulk ZnO (Table 2). This corresponds the fact that the excitonic peak shifts to the blue region results from the diminishing particle size and changing the morphological structure.[38–40] Because ZnO particles can absorb light with the energy of hν that matches or exceeds their band gap energy, ZnO nanoparticles can block UV radiation.[41]

The UPF value shows how effective a fabric is at blocking out solar UV radiation and the higher the UPF value, the more protection the clothing offers. The UPF values in both UVA (315–400 nm) and UVB (315–290 nm) regions for the tested fabrics were calculated from the transmittance data using Eq. (1). The outcomes obtained are recorded in Table 4. From these data, it can be seen the untreated cotton does not absorb UV radiation, whereas untreated polyester/cotton and polyester absorb UV radiation at around 200 and 300 nm. Table 4 demonstrates nano-ZnO-covered fabrics have excellent ability to block UV radiation; any fabric can be considered a UV-defensive item if the UPF quality is more than 40 as listed in the AATCC test method.[42,43] After five washing cycles the UPF values do not significantly decrease (Table 4), revealing there is good adhesion between the nano-ZnO and the fabrics. The results also showed the efficiency of UV blocking for the ZnO-treated textiles increases in the order of cotton > cotton/polyester > polyester. The data also refer to the impact of particle size on the protection of the fabrics against UV radiation. When the particle size is smaller (XRD data), the UPF value is higher. The SEM images presented in Figure 5 show that the covering surface areas of the treated textiles change with the morphological structure, and increases for both cotton/polyester and polyester textiles by ZnO in the order sphere > rod > star, and for cotton in the order of rod > sphere > star. These orders agree with the UPF values.

To our knowledge, there are few studies on the UPF results of coating cotton with ZnO nanorods or nanostars. In general our UV-blocking results for the cotton coated with ZnO nanospherical particles lie in the range of those obtained in the literature. Yadav et al. reported that bleached cotton treated with 2% ZnO with an average size of 40 nm showed it could block 75% of UV radiation.[44] Jazbec et al. reported that cotton treated with low-pressure oxygen plasma for 30 s and

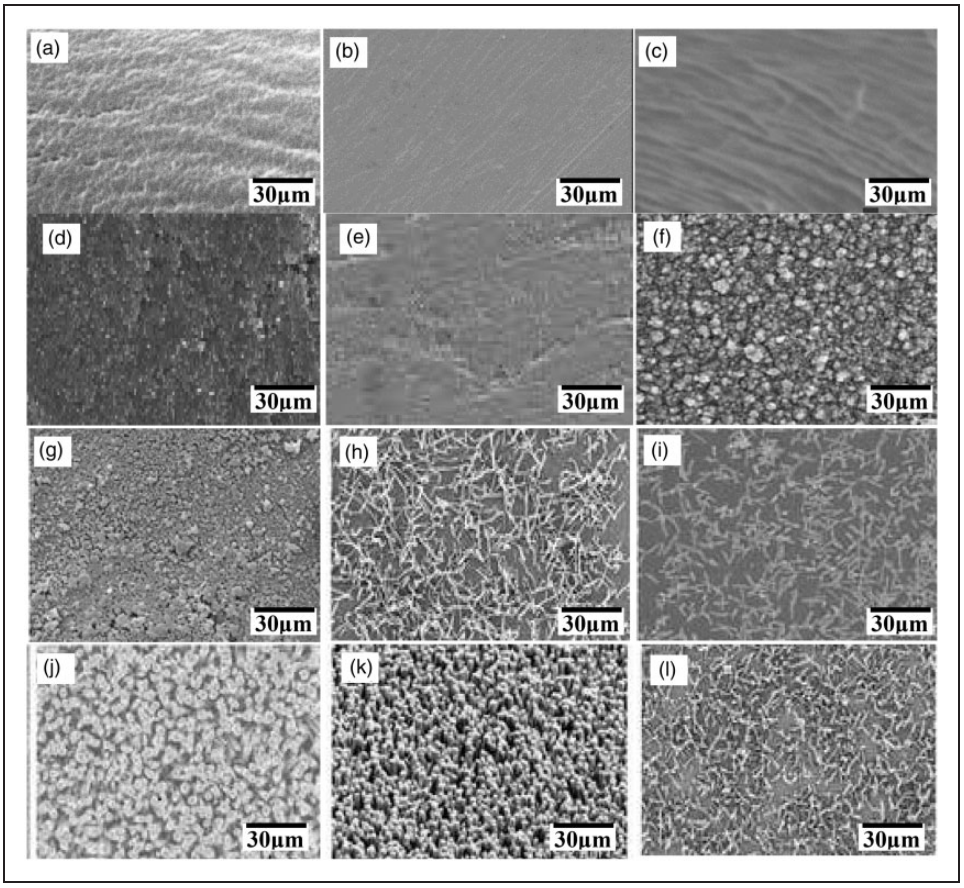

Figure 5. Scanning electron microscopy (SEM) of investigated samples: (a) cotton (Co); (b) polyester (PET); (c) Co/PET; (d) ZnO in ethylene glycol (Z_e/Co); (e) Z_e/PET; (f) Z_e/Co/PET; (g) ZnO in methanol (Z_m/Co); (h) Z_m/PET; (i) Z_m/Co/PET; (j) ZnO in water (Z_w/Co); (k) Z_w/PET; and (l) Z_w/Co/PET.

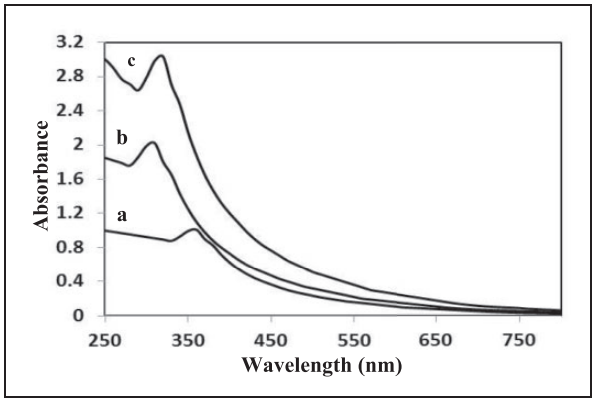

Figure 6. Ultraviolet visible spectra of the investigated zinc oxide (ZnO) samples: ZnO in ethylene glycol (Z_e) (a); ZnO in methanol (Z_m) (b); and ZnO in water (Z_w) (c).

coated with 3% nano-ZnO (40 nm) has a low UPF value of 57.7.[45] In contrast, cotton coated with bundle/flower-like ZnO particles of different sizes showed an excellent ability to block the UV radiation with a UPF value of 105.6.[46] Cotton coated with ZnO nanorods showed a UPF value of 220, which agrees with our results.[47]

Ran, et al. reported a UPF value of 122.5 for nanorod ZnO-coated cotton fabrics.[48]

Mechanical properties of untreated and nano-treated fabrics

Table 5 shows the influence of the nano-ZnO coating on the mechanical properties of untreated and ZnO-treated fabrics. Statistically, a significant difference was not found between the tensile strength and elongation values of the treated and untreated samples. The mechanical properties were also examined after exposure to the fabrics for UV illumination for 100 hrs. The results obtained showed the tensile strength of ZnO-treated fabrics decreases compared with untreated ones.

This may be attributed to damage occurring as a result of the generation of reactive oxygen species during exposure of fabric to UV light.[49]

Table 4. UPF for untreated and treated fabrics

Sample	UVA 315–400 nm	UVB 295–315 nm	UPF value
Cotton	2.02 (2.01)	2.16 (2.12)	8.9 (8.9)
Co/PET	4.2 (4.2)	12.2 (12.1)	22.2 (22.1)
Polyester	6.3 (6.2)	14.2 (14.2)	32.6 (32.6)
Zw/Co	18.2 (18.1)	22.3 (22.0)	139.5 (130.5)
Zm/Co	19.0 (18.7)	23.2 (23.0)	247.2 (240.6)
Ze/Co	25.3 (24.9)	31.2 (30.8)	198.5 (188.5)
Zw/Co/PET	23.1 (22.8)	27.1 (26.5)	100.3 (95.9)
Zm/Co/PET	25.3 (24.7)	31.2 (28.8)	104.2 (100.1)
Ze/Co/PET	28.7 (26.6)	32.7 (30.1)	112.6 (105.7)
Zw/PET	31.1 (29.6)	33.2 (30.7)	61.3 (59.1)
Zm/PET	32.7 (30.1)	36.1 (33.2)	66.3 (62.1)
Ze/PET	33.8 (32.2)	38.1 (35.5)	68.7 (63.9)

Co: cotton; PET: polyester; UPF: ultraviolet protection factor; UV: ultraviolet; Z_w: Zinc oxide (ZnO) in water; Z_m: ZnO in methanol; Z_e: ZnO in ethylene glycol.

Antimicrobial activity

The qualitative antimicrobial properties of uncoated and nano-ZnO-coated fabrics was evaluated utilizing Gram-positive microscopic organisms *(E. coli ATCC 23282)*, Gram-negative microbes *(S. aureus ATCC 29737)*, and diploid fungus (*C. albicans IMRU 3669*). After 24 hours of incubation, the antimicrobial activity of raw and treated specimens against the tested microorganisms was measured. The antimicrobial activity of fabric samples settled as far as the zone of inhibition shaped on the agar medium. The control fabrics (untreated) did not demonstrate any antimicrobial activity (bacterial growth was observed on the surface as shown by the presence of colonies). The nano-ZnO-treated fabrics set on the bacteria-immunized surfaces showed that most of the bacteria were killed under and around the particular zones of restraint (clear zones with no bacterial growth) for the tested microorganisms. High bacterial growth as shown by the bacterial growth lawn (largely indistinguishable collection of colonies) was observed all around. Also, no bacterial growth was seen under or within the fabric. This phenomenon was observed more remarkably for *S. aureus* than for *E. coli* and *C. albicans* for all fabrics treated with ZnO nanoparticles and those that were bulk treated. The diameters of the hindrance zones were measured and recorded in Table 6. The results obtained by the percentage reduction test agree with those of the agar diffusion method (Table 6).

The results reveal antimicrobial activity increases with a decrease in ZnO particle size. This is because the particle size plays a large role in manipulating the

Table 5. Effect of nano-ZnO treatment on mechanical properties of tested fabrics

Sample	Fabric	Tensile strength (kg/cm^2) (standard deviation) Untreated	Tensile strength (kg/cm^2) (standard deviation) Treated	Elongation % (standard deviation) Untreated	Elongation % (standard deviation) Treated
Cotton	Warp	39.0 ± 1.9 (38.0 ± 1.5)	34.5 ± 1.2 (29.2 ± 1.3)	9.0 ± 0.22 (9.1 ± 0.3)	8.7 ± 0.24 (9.2 ± 0.3)
	Weft	33 ± 1.1 (32.8 ± 1.3)	34.1 ± 1.1 (29.1 ± 1.3)	12.4 ± 0.33 (12.7 ± 0.4)	13 ± 0.31 (14.1 ± 0.6)
Co/PET	Warp	30.2 ± 1.4 (29.6 ± 1.3)	31.0 ± 1.5 (26.5 ± 2.0)	19.4 ± 1.2 (19.6 ± 1.3)	19.5 ± 1.5 (20.2 ± 1.3)
	Weft	27.1 ± 1.1 (26.5 ± 1.0)	27.5 ± 1.1 (23.5 ± 0.9)	22.5 ± 1.4 (22.5 ± 1.3)	23.1 ± 1.5 (24.0 ± 1.2)
Polyester	Warp	30.1 ± 1.1 (29.0 ± 0.7)	31.8 ± 1.2 (26.8 ± 1.4)	28.1 ± 1.3 (29 ± 1.5)	28.2 ± 1.2 (29.9 ± 1.3)
	Weft	34.5 ± 1.3 (33.0 ± 1.6)	32.7 ± 1.2 (26.6 ± 1.3)	26.5 ± 1.1 (27.0 ± 1.2)	26.6 ± 1.3 (28.3 ± 1.6)

Co: cotton; PET: polyester; ZnO: zinc oxide.

Table 6. Antimicrobial assessment of fabrics treated with ZnO

	Zone of inhibition in cm			R%		
Sample	*S. aureus*	*E. coli*	*C. albicans*	*S aureus*	*E. coli*	*C. albicans*
Cotton	Nil	Nil	Nil	0	0	0
Co/PET	Nil	Nil	0	0	0	0
Polyester	Nil	Nil	Nil	0	0	0
Zw/Co	4.6 ± 0.5	2.9 ± 0.2	2.5 ± 0.3	82.1	77.8	72.7
Zm/Co	4.8 ± 0.4	3.0 ± 0.2	2.7 ± 0.3	83.4	78.8	74.4
Ze/Co	5.2 ± 0.5	3.3 ± 0.2	3.1 ± 0.3	91.4	86.8	84.7
Zbulk/Co	2.3 ± 0.4	1.5 ± 0.3	1.9 ± 0.4	63.5	55.7	45.7
Zw/Co/PET	3.7 ± 0.3	2.2 ± 0.2	1.9 ± 0.2	72.1	66.1	63.3
Zm/Co/PET	4.0 ± 0.4	2.4 ± 0.2	2.1 ± 0.2	73.2	67.5	65.0
Ze/Co/PET	4.2 ± 0.4	2.6 ± 0.3	2.2 ± 0.3	76.1	71.7	66.3
Zbulk/Co/PET	1.7 ± 0.3	1.5 ± 0.3	1.6 ± 0.2	53.1	47.4	41.2
Zw/PET	2.9 ± 0.4	2.0 ± 0.2	1.8 ± 0.1	63.4	56.8	48.3
Zm/PET	3.0 ± 0.3	2.2 ± 0.3	2.0 ± 0.1	68.2	61.5	50.1
Ze/PET	3.3 ± 0.4	2.5 ± 0.4	2.1 ± 0.3	76.4	66.8	53.3
Zbulk/PET	1.4 ± 0.2	1.1 ± 0.2	0.9 ± 0.2	46.3	41.2	37.6

Co: cotton; PET: polyester; Z_w: Zinc oxide (ZnO) in water; Z_m: ZnO in methanol; Z_e: ZnO in ethylene glycol; *S. aureus: Staphylococcus aureus; E.coli: Escherichia coli; C. albicans: Candida albicans.*

degree of toxicity for ZnO as it disrupts bacterial cell wall integrity and simplifies the penetration of particles through the cell membrane. For all treated textiles, antimicrobial efficiency increases in the morphological order of sphere > rod > star. This finding is in agreement with other research.[50–53] The results also showed nano-ZnO-treated cotton has better antimicrobial activity over the other treated polyester fabrics.

After five washing cycles, the high antibacterial efficiency of the ZnO-coated fabrics was preserved (Table 5), revealing a strong adhesive and durable coating of ZnO nanoparticles on the investigated fabrics.

Our results in Table 5 show that all the nano-ZnO-coated fabrics have higher antibacterial activity on *S. aureus* (Gram-positive bacteria) than on *E. coli* (Gram-negative bacteria). Such susceptibility could be attributed to differences in their cell walls.[54] The cell wall of the Gram-negative bacteria consist of a pair of membranes: an outer membrane made of lipopolysaccharide, lipids, and proteins, and an internal peptidoglycan cytoplasmic membrane that provides effective protection against biocides. However, the walls of the Gram-positive bacteria do not contain the lipopolysaccharide.

Antimicrobial activity was measured in the absence of UV light and carried out in daylight only. The accurate mechanisms of ZnO antibacterial activity in the absence of light are highly debatable and need more understanding. However, the antimicrobial influence of ZnO particles can be interpreted by many mechanisms, involving possible production of free radicals in the absence of light or other toxicity mechanisms besides reactive oxygen species production.[54,55,46] Hence, the reaction of ZnO with H_2O molecules produces H_2O_2, OH ions, and other reactive oxygen species by non-photocatalytic processes. These species can penetrate the cell wall, accumulate there, and generate great oxidative stress inside the cell, damaging the DNA and enzymes of the bacteria, leading to cell death.[56] Another suggested mechanism is dissolution of ZnO NPs into Zn^{2+} ions, which can also penetrate the bacterial cell and stop the action of respiratory enzymes.[57,50] Moreover, it is reported that under daylight conditions, the antimicrobial activity of ZnO is mainly attributed to the attachment of ZnO nanoparticles to the bacterial cell walls, leading to an increase in the concentrations of Zn^{2+} cations in the bacterial cytoplasm.[58,1] Other researchers reported that ZnO surface defects and charges can impact the antibacterial efficiency of ZnO-coated fabrics because the surface contains edges and corners with potential reactive surface sites.[59,60]

It should be noted that our results for the antibacterial activity of cotton treated with different morphological structure of ZnO against *E. coli, S. aureus*, and *C. albicans* (without UV radiation) have values of 77.8–86.8%, 82.1–91.4%, and 72.7–84.7%, respectively, higher than those obtained by other researchers.[61–63] Cotton fabric coated with 3% ZnO nanoparticles

(45 nm) moderately reduced *E. coli* growth.[64] Cotton fabric (honeycomb-weave) coated with 2% ZnO nanoparticles at a size of 50 nm showed high antibacterial efficiency of 80% and >90% against *E. coli* and *S. aureus*, respectively.[65] Cotton coated with bundle/flower-like ZnO particles of different sizes showed good bacteriostatic activity against Gram-negative *Klebsiella pneumoniae* and Gram-positive *S. aureus* bacteria.[46] Polyester fibers coated with 45 nm nano-ZnO particles showed moderate activity against *S. aureus*.[64]

Conclusions

A simple method has been utilized to arrange nano-ZnO particles with a diverse morphological structure and coating them on cotton, cotton/polyester, and polyester fabrics to grant useful properties. Nanoparticles were analyzed by XRD, SEM, and FTIR. The existence of nanoparticles on the fabric surface was tested by SEM and showed a significant change in the UV-absorbing activity of ZnO-treated fabrics. The UPF values of coated fabrics were much higher than 50, revealing excellent protection against UV radiation. The cotton fabric treated with ZnO nanorods exhibited the highest UPF value of 247.2. No significant difference was found between the tensile strength and elongation values of the treated and untreated samples. The antimicrobial activity of the treated textiles was measured qualitatively by agar diffusion methods and quantitatively by a percentage reduction test. The bactericidal activities of samples were tested with *E. coli, S. aureus*, and *C. albicans*. The treated fabrics exhibited notable antimicrobial action against these microorganisms. The cotton fabric treated with 26 nm non-spherical ZnO particles exhibit the highest antimicrobial efficiency values of 91.4%, 86.8%, and 84.7% for *S. aureus, E. coli*, and *C. albicans*, respectively. All the treated fabrics exhibit high antimicrobial and excellent UPF values, even after five washing cycles. These coated antimicrobial textiles are potentially useful in a wide variety of biomedical applications. The results obtained suggest the possibility of using the ZnO-coated fabrics in defense clothing to protect the body against solar radiation and microorganisms and for use in other technological applications.

Declaration of conflicting interests

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