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Comparative study of multifunctional properties of synthesised ZnO and MgO NPs for Textiles applications

Abstract

Purpose

Among various metal oxide nano particles, MgO NPs and ZnO nanoparticles (NPs) in particular are gaining increasing attention due to their multifunctional characteristics, low cost and compatibility with textile materials. Each type of nanoparticle excels over others in certain properties. As such, it is often crucial to carry out comparative studies of NPs in order to identify the one showing higher efficiency/output for particular applications of textile products.

Design/methodology/approach

In the investigation reported in this paper, ZnO NPs and MgO NPs were synthesised via sol-gel technique and characterised. For comparative analysis, the synthesised NPs were evaluated for multiple properties using standard procedures before and after being applied on cotton fabrics by a dip-pad-dry-cure method.

Findings

XRD and FTIR analyses confirmed the successful synthesis of ZnO and MgO nanoparticles. Homogeneous formation of desired nanoparticles and their dense and uniform deposition on the cotton fibre surface were observed using SEM. ZnO NPs and MgO NPs coatings on cotton were observed to significantly enhance self-cleaning/stain removal properties achieving Grade 5 and Grade 4 categories respectively. In terms of UV protection, ZnO or MgO NP coated fabrics showed UPF values of greater than 50 i.e. excellent in blocking UV rays. MgO NPs exhibited 20% cleaning efficiency in treating reactive dye wastewater against ZnO NPs which were 4 % efficient in the same treatment, so MgO was more suitable for such type of treatments at low cost. Both NPs were able to impart multi-functionality to cotton fabrics as per requirement of the end products. However, ZnO NPs were better for stain removal from the fabrics while MgO NPs were appropriate for UV blocking.

Originality/Value

It was therefore clear that multi-functional textile products could be developed by employing a single type of cost effective and efficient nano particles.

Keywords: ZnO NPs; MgO NPs; antibacterial; self-cleaning; UV blocking; effluent treatment

1 Introduction

One of the manufacturing sectors that has drawn tremendous gains from nanotechnology is the textile sector. The application of nanotechnology in textiles has resulted in significant diversification and modification of the intrinsic properties of conventional textiles, for instance in improving the durability, comfort, hygiene related characteristics (Payne, 1997, Lee *et al.*, 2003, Gorenšek and Recelj, 2007, Sun *et al.*, 2009) and self cleaning properties, etc (Haji *et al.*, 2016, Dumitrescu *et al.*, 2020). Furthermore, application of nanotechnology has demonstrated that conventional textile products can be transformed into materials having an entirely new set of properties with an overall enhancement in the functionality too (Yuranova *et al.*, 2006). To achieve the desired functionality, a range of materials from polymeric products (Chirilă *et al.*, 2020, Tadesse *et al.*, 2020) to metals in different forms (Balci *et al.*, 2014) are reportedly used.

In order to optimally fulfil the needs of different industries, including textiles, a large number of natural as well as synthetic materials have been studied by researchers globally for their potential utilisation as nano-materials. Among various classes of materials that are widely researched in the field of nanotechnology, metals in their elemental or other forms such as oxides have drawn considerable interest of the research community (Haji *et al.*, 2013a, Haji *et al.*, 2013b). Metal oxide nanoparticles exhibit a number of functional characteristics such as electrical conductivity, thermal conductivity, antibacterial/bactericidal action, UV protection, self-cleaning, odour and stain prevention, etc. (Anita *et al.*, 2011, Solanki *et al.*, 2011). Owing to their characteristic high surface area to volume ratio, metal oxides nanoparticles are

generally very efficient in delivering the desired functionality. Moreover, these materials are well-known for their ability in general to withstand stringent processing and end-use conditions (Li *et al.*, 2007, Abramov *et al.*, 2009).

Metal oxide nanoparticles such as zinc oxide (ZnO) and magnesium oxide (MgO) are known to have a substantially low cost in comparison to nano-silver, which is arguably the most common nanoparticle that is very widely studied for its applications in the field of functional textiles as well. Furthermore, such metal oxide nanoparticles exhibit multifunctional characteristics which could be considered useful for the textile industry. For instance, owing to their photo-catalytic nature and good adsorption properties, ZnO and MgO nanoparticles can be employed for wastewater treatment of dye effluent from the textile industry (Tan *et al.*, 2015, Tayeb *et al.*, 2019) and for adsorption of harmful materials in gaseous form (Rajagopalan *et al.*, 2002, Li and Klabunde, 1991, Sundarrajan *et al.*, 2010). Other examples of potential applicability of ZnO and MgO nanoparticles in textiles include anti-bacterial, self-cleaning, anti-corrosive, and antifungal characteristics of these materials (Tang and Lv, 2014, Esteban-Tejeda *et al.*, 2015, Saravanan, 2007, Bharathi Yazhini and Gurumalles Prabu, 2015, Saad *et al.*, 2016, Wang *et al.*, 2013). Furthermore, application of these nanoparticles on textile fabrics has been shown to lead to significant improvement in the ultraviolet protection factor of the textile material, which otherwise has a low degree of ultraviolet blocking (Becheri *et al.*, 2008). Such metal oxides can furnish the desired functionality when deposited in their as-produced form, in modified form (Tang *et al.*, 2006b, Bedilo *et al.*, 2002, Fangli *et al.*, 2003) or in combination with other materials such as polymer matrices (Vigneshwaran *et al.*, 2006, Tang *et al.*, 2006a).

A number of studies has reported comparative assessment of the properties of various metallic oxides (Kathirvelu *et al.*, 2008, Sawai, 2003, Sójka-Ledakowicz *et al.*, 2008). For instance, differences in the photo-catalytic activity of zinc oxide (ZnO) prepared by different

methods has been reported in one study (Saravanan *et al.*, 2013a), while in another study ZnO was compared with other metal oxides including copper, nickel and tin for use as a catalyst for degradation of textile dyes (Gnanasekaran *et al.*, 2017). It has been demonstrated that a nano-composite of ZnO with silver and CdO can achieve photo-catalytic activity for textile effluent treatment in visible light (Saravanan *et al.*, 2011). A similar property has been reported for PolyAniline/ ZnO composite (Saravanan *et al.*, 2016) and ZnO/CuO composite (Saravanan *et al.*, 2013b). Similarly, MgO has been studied as an adsorbent for textile dyes on its own (Darvishi Cheshmeh Soltani *et al.*, 2016, Mahmoud *et al.*, 2016, Nassar *et al.*, 2017), as well as in modified form (Asgari *et al.*, 2019), for use in medical textiles (Ponnuvelu *et al.*, 2016) and in smart textiles (Du *et al.*, 2013). These two materials have also been used in combination with each other for various textile-related applications (Raj *et al.*, 2020).

A review of the literature has revealed that both ZnO and MgO in the form of nano-particles could potentially be useful for several applications in the textile sector. These materials are often also compared for differences in their properties. However, most relevant studies evaluate one or two properties only. Thus, in this study, we investigated multiple, textile-relevant properties of ZnO and MgO nano particles that were synthesised using the sol-gel technique. This technique allows fabrication of nano particles having a uniform particle size distribution by synthesis at relatively low temperatures (Mastuli *et al.*, 2012, Epifani *et al.*, 2006). Homogeneity is required for nanoparticles so that when applied on textile materials, uniformity of desired properties throughout the treated article can be achieved. In this study, the synthesised nanoparticles were characterised, formulated into finishing liquor and applied onto cotton fabric through a dip-pad-dry-cure method. The antibacterial activity, stain release, UV-blocking and effluent decolourisation properties were evaluated by standard methods and comparative analyses were conducted.

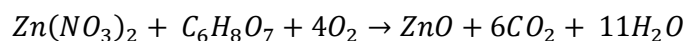
2 Materials and Methods

2.1 Materials

Zinc nitrate hexahydrate (>98.0%), citric acid monohydrate (99.5%), magnesium nitrate hexahydrate (>98.0%), sodium hydroxide (>97.0%) and sodium lauryl sulphate (98.5%) were purchased from Dae Jung Chemicals, Korea. Ethanol ($\geq 99.8\%$, sigma Aldrich) was used as received. Commercially scoured and bleached 100% cotton [115 g/m²; plain weave] fabric was used.

2.2 Synthesis of ZnO NPs

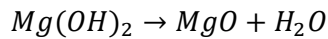
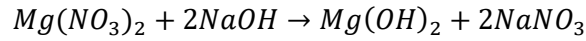
Equal mole (1 M) of zinc nitrate hexahydrate and citric acid monohydrate were dissolved into 100 mL of deionised water and the solution was stirred for 60 min at 90 °C which led the reaction solution to self-combustion. After combustion, the grounded powder was subjected to calcination for 3 hours at 600 °C in a furnace. Scheme 1 depicts the formation of ZnO NPs.



Scheme 1: Synthesis of ZnO NPs by sol-gel method

2.3 Synthesis of MgO NPs

Magnesium nitrate hexahydrate (5.21 g, 0.1 M) was dissolved in 200 mL of distilled water at room temperature. To this, 200 mL of 0.1 M sodium hydroxide solution was added drop wise with a gentle stirring over a period of 30 min. The stirring was continued for another 3 h, and then the reaction mixture was allowed to settle for 6 h. The supernatant solution was discarded, and the remaining suspension was washed with distilled water (50 mL×3). The white precipitates were collected and allowed to dry at 100 °C in a hot air oven for 6 h. The dried precipitates were ground well and calcinated at 400 °C for 4 h. Scheme 2 depicts the formation of MgO NPs.



Scheme 2: Synthesis of MgO NPs by sol-gel method

2.4 Coating of Fabric

Synthesised nanoparticles (ZnO and MgO) were coated on the cotton fabric by dip-pad-dry-cure method. Before application, synthesised nanoparticles were suspended in deionized water and were dispersed using mechanical stirrer. Thus, a sample of 100% cotton fabric was dipped in a suspension containing ZnO NPs (2%) or MgO NPs (2%) and citric acid (3%) for 1 h. The cotton fabric sample was then passed through a padding mangle to maintain a wet pick-up of 100%. After padding, the sample was air-dried and then cured for 3 minutes at 140°C. The coated fabric sample was then immersed in sodium lauryl sulphate (2 g/L) solution for 5 minutes, to remove any unbound nanoparticles. The fabric sample was then rinsed 10 times to washout the soap completely and then air dried. The coated samples were conditioned under standard conditions.

2.5 Characterisation of ZnO and MgO NPs

X-ray diffraction pattern of the synthesised nanoparticles was obtained using the PANalytical X'pert pro X-ray diffractometer using Cu-K α radiation of wavelength $\lambda=0.1541$ nm. In addition, the average crystallite size of the synthesised nanoparticles was calculated using Scherrer's formula.

$$L = \frac{k\lambda}{\beta \cos \vartheta}$$

where L is an average crystallite size, λ is the wavelength of X-ray radiation (0.1541 nm), K is a constant equal to 0.9, β is Full-width half maximum of the peak (in radians) and 2θ is Bragg's angle (degree).

Infrared spectra of synthesised NPs were recorded using Spectrum One spectrophotometer (Perkin Elmer, UK). Each spectrum was acquired using 0.09 cm⁻¹ resolution and 16 scans per spectrum and the vibrational frequencies are reported in wavenumbers (cm⁻¹).

Morphological features of the synthesised nanoparticles, uncoated and coated cotton fabric samples were studied using a JEOL JSM-6380A scanning electron microscope. For surface morphology, the synthesised nanoparticles and the coated cotton fabric samples were coated with up to 300 °Å gold.

2.6 Antibacterial Activity

The antibacterial activity of the synthesised NPs and coated fabric samples were tested according to AATCC TM-147 (the agar diffusion method). A sterile nutrient agar solution was prepared and placed in plates and then left to solidify. Once solidified, a cork broker was used to make holes and then the bacterial culture was spread on the agar with the help of a cotton bud. The coated fabric samples were gently pressed in the centre of the mat culture and the plates are left to be incubated for 24 hours at 37 °C. The zone of inhibition was examined to obtain the antibacterial activity .

2.7 Stain Release Properties

The uncoated and coated cotton fabric samples were stained with drops of ketchup and left under 2 kg weight for 1 min. The residual stain was rated in comparison with an AATCC TM-130 stain-release replica immediately, after 3 h, 5 h and after washing.

2.8 UV Blocking Properties

The UV blocking of the uncoated and coated fabric samples were measured using AATCC TM-183. Thus, two samples of 2×2 inches were taken and placed on the transmission port of the Specord 200 Plus (Analytik Jena, Germany) UV-VIS spectrophotometer and the readings were recorded.

2.9 Decolourisation Efficiency

Decolourisation efficiency of the synthesized nanoparticles was measured by comparing the absorbance of a test dye solution before and after the addition of nanoparticles. For this purpose, a Specord 200 Plus (Analytik Jena, Germany) spectrophotometer was used. The test dye solution containing 10 wt% of the selected reactive dye was prepared and the pH was set at 9.0 by using 0.1N solution of NaOH. 0.05 g of the synthesized nanoparticles was added and the solution was stirred at 450 RPM for 10 minutes followed by stirring at 40 RPM for another 10 minutes. The mixture was then left to rest for 30 minutes and the supernatant samples were collected for the measurement of absorbance.

3 Results and Discussion

3.1 Characterisation of Synthesised ZnO and MgO NPs

X-ray diffraction patterns to characterise the synthesised ZnO and MgO NPs were obtained as described in Section 2.5. As shown in Figure 1(a), the XRD peaks are in good agreement with JCPDF card No. 01-076-0704 (Jamshidi Bandari and Nasirian, 2019). Moreover, no impurities were detected suggesting that high-quality ZnO NPs were synthesised. The average crystallite size of the synthesised ZnO NPs was found to be 20 nm.

Similarly, the XRD peaks shown in Figure 1(b) are in good agreement with JCPDF card No. 01-089-7746 (Imani and Safaei, 2019a, Mantilaka *et al.*, 2014) and no characteristic peaks for any impurities were detected. Thus, the analysis clearly suggests that high-quality MgO NPs were synthesised, for which the average crystallite size was found to be 9 nm.

Insert Figure 1

To further validate the aforementioned analysis, the FTIR spectra of the synthesized nanoparticles were also obtained. Figure 2(a) shows the FTIR spectra of the synthesised ZnO NPs. The vibration bands at 2359.3 cm^{-1} and 2338.2 cm^{-1} can be attributed to atmospheric CO_2 .

(Arshad *et al.*, 2011) The sharp peaks observed in the range of 687.1 to 558.7 cm^{-1} can be attributed to the Zn–O bond (Getie *et al.*, 2017, Shaban *et al.*, 2018, Saoud *et al.*, 2015, Costa *et al.*, 2018).

Similarly, for the synthesised MgO particles, the FTIR spectra is shown in Figure 2(b). The peak at 3721.4 cm^{-1} belong to the O-H group whereas the peaks at 1403.7 cm^{-1} and 654.0 cm^{-1} correspond to Mg-O stretching vibration (Alfaro *et al.*, 2019, Shi *et al.*, 2017, Imani and Safaei, 2019b, Gajengi *et al.*, 2017).

Insert Figure 2

The morphology of the synthesised ZnO and MgO NPs, uncoated and coated cotton fabric samples was observed by SEM and relevant micrographs are shown in Figures 3(a-e). Figure 3a shows the SEM image of the synthesised ZnO NPs in powder form. It clearly shows the spherical nature of the prepared nanoparticles. Furthermore, it can also be observed that the ZnO NPs obtained are homogeneous in nature and agglomerated with an average particle size between 80 – 85 nm.

Figure 3b shows the SEM image of the synthesised MgO NPs in powder form. The image shows a highly agglomerated structure of MgO NPs which are almost spherical in shape with an average particle size between 30 – 40 nm. The differences between particles in SEM and XRD data may be due to agglomeration of particles.

Figure 3(c-e) shows the morphological changes induced on cotton surface coated with the synthesised NPs. In contrast to the SEM micrograph of uncoated cotton fabric (Figure 3c), the micrograph of the cotton fabric coated with ZnO (Figure 3d) and MgO NPs (Figure 3e) shows a dense and uniform deposition of NPs on the cotton fibre surface.

Insert Figure 3

3.2 Antibacterial Activity

The antibacterial property of the fabric coated with the synthesised ZnO and MgO NPs was evaluated against *E. Coli* and *S. Aureus* using the Agar Diffusion method. For this, 15 mm x 15 mm swatches of the coated fabric were laid onto the inoculated agar plates. The images of the agar plates recorded after a 24 hr incubation period at 37 °C are provided in Figure 4. It was observed that both ZnO and MgO NPs, in the form of coating on fabric, exhibited bacteriostatic property against *E. Coli*, as evident from a distinct zone of inhibition that can be seen in Figure 4a. On the other hand, neither the ZnO coated fabric nor the MgO coated fabric showed any activity against *S. Aureus*. Similar results have been reported in other studies as well and can be attributed to structural differences in the cell of gram-positive and gram-negative bacteria (Brayner *et al.*, 2006). Furthermore, the absence of inhibition zone in the case of gram-positive bacteria, as shown in Figure 4b, could be related to low migration rate of the NPs from the coating onto the nutrient agar (D'água *et al.*, 2018)

Insert Figure 4

3.3 Stain Release Properties

To evaluate the stain release properties of the coating, the coated and uncoated samples were stained with drops of ketchup. The residual stain was graded on a scale by comparing it with stain-release replica that is used in AATCC test method 130. According to the stain release replica of AATCC Test Method 130, Grade 1 indicates substantial residual stain (poor stain removal) whereas Grade 5 indicates minimal residual stain (complete removal). A useful fabric in this context would be one that achieves a grade of at least 4. Relevant results of this test that are shown in Table 1 indicate that both ZnO and MgO NPs exhibited acceptable self-cleaning/stain release properties with the ZnO coated fabric showing significantly enhanced self-cleaning/stain removal properties.

Insert Table 1

3.4 UV Blocking Properties

As discussed in the preceding literature review, most metal oxide NPs including ZnO and MgO possess excellent UV absorption ability. Thus, the coated cotton fabrics were expected to exhibit UV blocking property which is often expressed quantitatively as Ultraviolet Protection Factor (UPF). UPF is defined as the ratio of the average effective UV irradiance of an unprotected (uncovered) surface, e.g., skin, to the effective UV irradiance on that surface in the presence of a protective layer which in this case is the fabric. A UPF of greater than 50 is generally regarded as an indicator of excellent UV blocking property of a sheet material such as a woven fabric. The relevant results of this analysis are provided in Table 2 and Figure 5. As shown in Figure 5, the black spectrum obtained for the uncoated cotton fabric shows high transmission of UV through it and the corresponding UPF was found to be 20. In contrast, the UPF values for the ZnO coated fabric and the MgO coated fabric were found to be 58 and 71, respectively. Thus, it can be concluded that coating of cotton fabric with the synthesised ZnO and MgO NPs imparts UV blocking functionality in the fabric.

Insert Table 2

Insert Figure 5

3.5 Decolourisation efficiency

Flocculation experiment for the removal of residual dye was performed. The dye removal efficiency of the synthesised NPs is presented in Figure 6. Almost 4% and 20% of efficiency was achieved at 0.05 g/L of dosage for the synthesised ZnO NPs and MgO NPs respectively. The result indicated that the synthesised MgO NPs was effective for treating reactive dye wastewater as compared to the synthesised ZnO NPs.

Insert Figure 6

4 Conclusion

In this investigation, homogenous, uniform and cost effective ZnO NPs and MgO NPs were synthesised by sol-gel technique. Characterisation and evaluation of relevant properties were conducted on uncoated and coated cotton fabrics for comparative analyses so suitable NPs could be identified for the textile products required to have multifunctional properties as per their end use. It was found that the two types of synthesised NPs did not excel at the same level in all of the properties evaluated in this study. Indeed, although each of the two types of NPs had multiple characteristics, it was not possible to use any one type of NPs to achieve multiple properties on the fabrics at the optimum. Nevertheless, the type of NPs that achieve higher/the highest grade as per standard for the specific properties can be selected for delivering process and products of greater efficiency and cost effectiveness.

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Captions of Figure

Figure 1: XRD Spectra of the synthesised a) ZnO NPs b) MgO NPs

Figure 2: FTIR Spectra of the synthesised a) ZnO NPs; b) MgO NPs

Figure 3: SEM micrographs of a) ZnO NPs Powder; b) MgO NPs Powder; c) uncoated Cotton Fabric; d) ZnO NPs coated Cotton Fabric; and e) MgO NPs coated Cotton Fabric

Figure 4: Agar method results of the synthesised ZnO and MgO NPs against: a) Gram-negative bacterial strains; b) Gram-positive bacterial strains

Figure 5: UV transmittance spectra of uncoated and coated cotton fabrics

Figure 6: UV-VIS spectrum of original and treated effluents

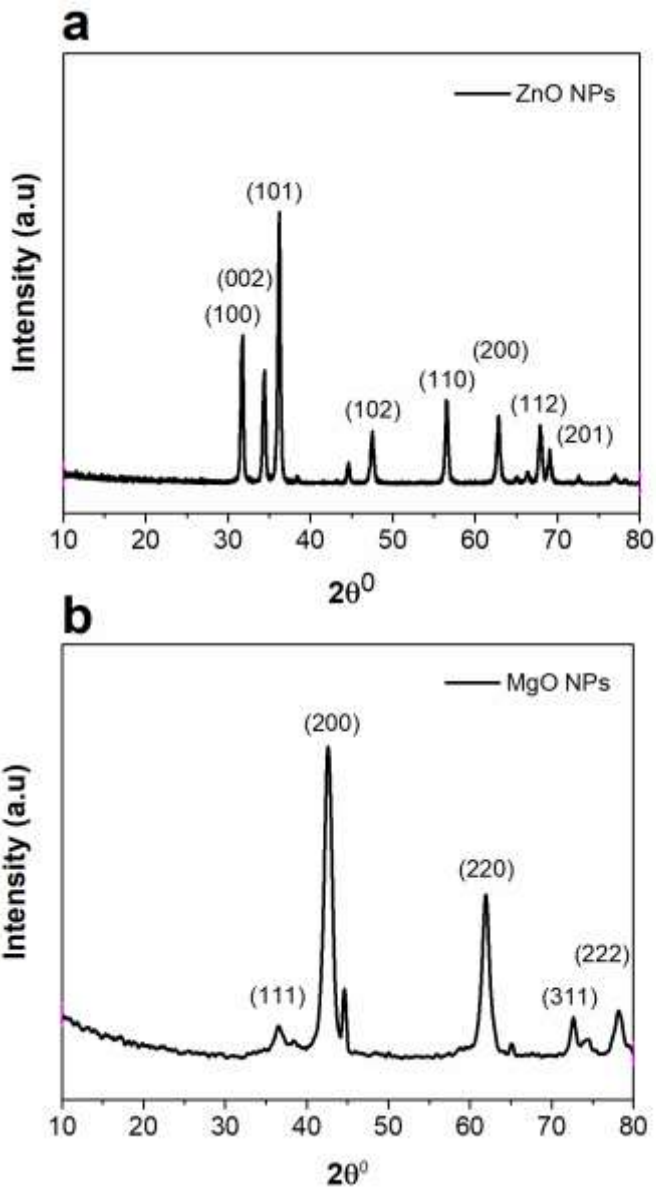


Figure 1

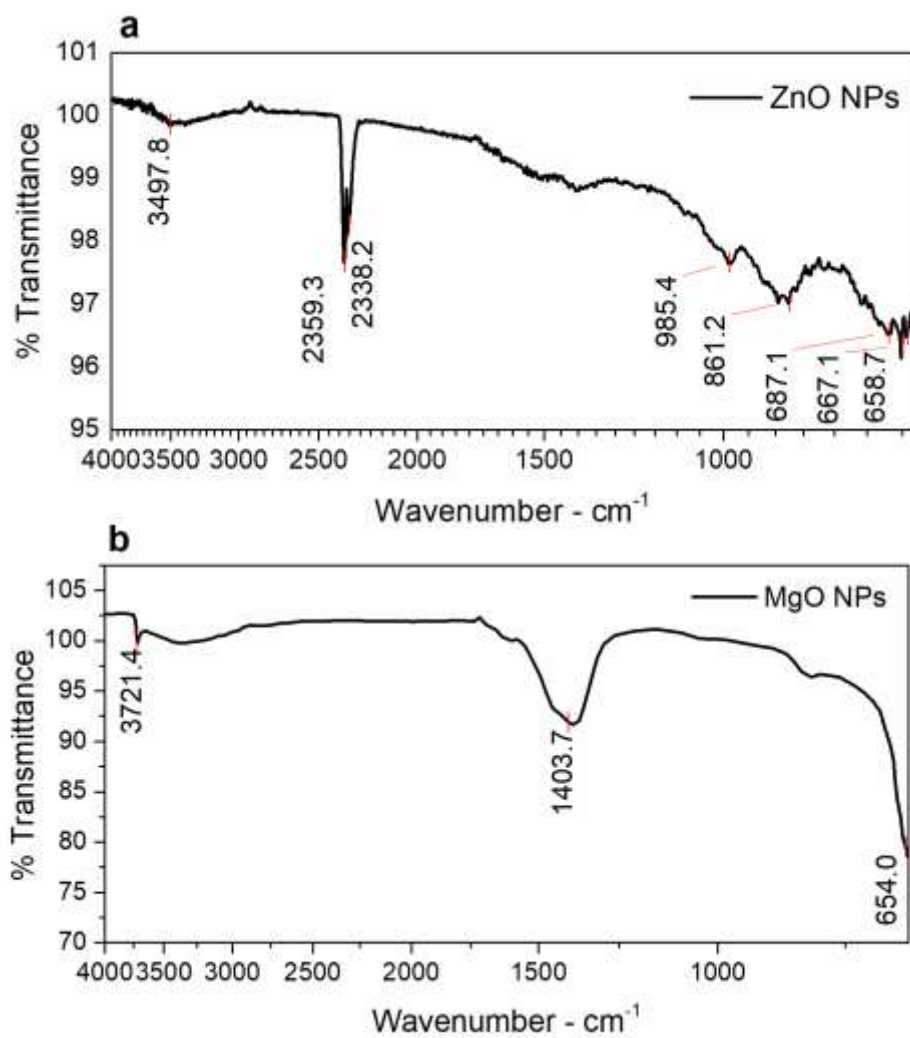


Figure 2

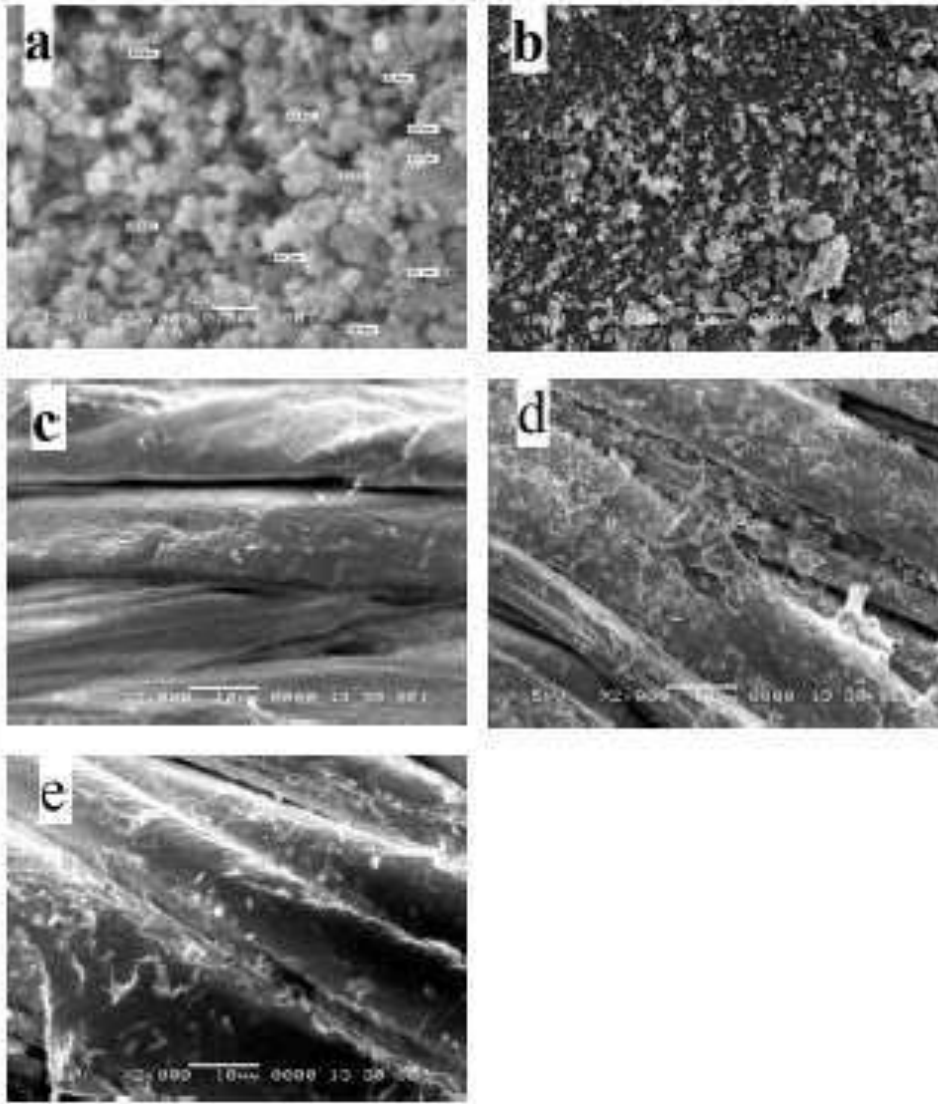


Figure 3

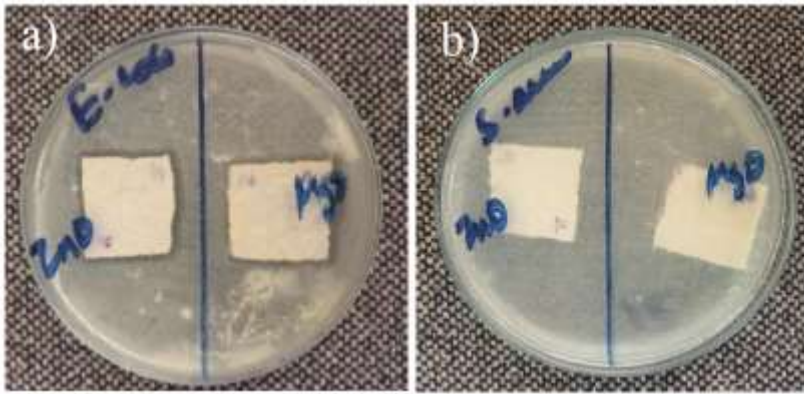


Figure 4

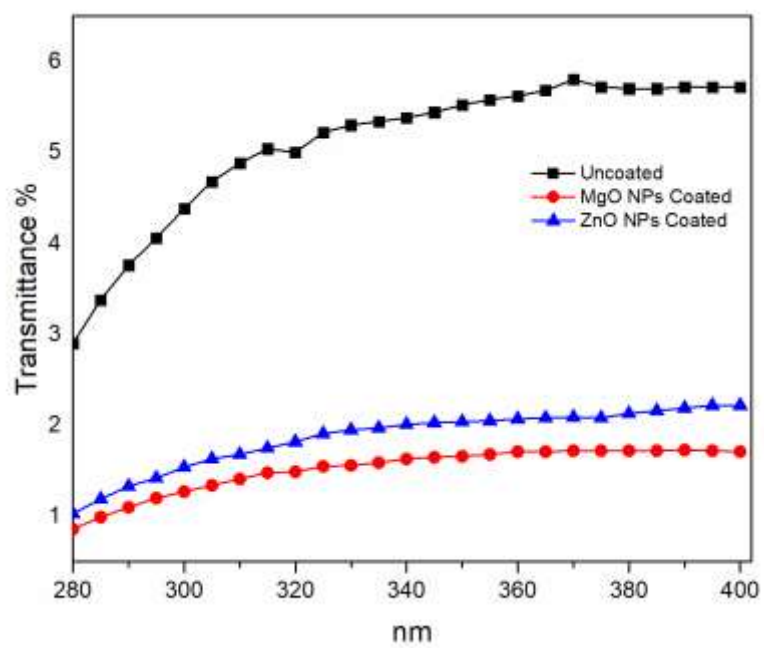


Figure 5

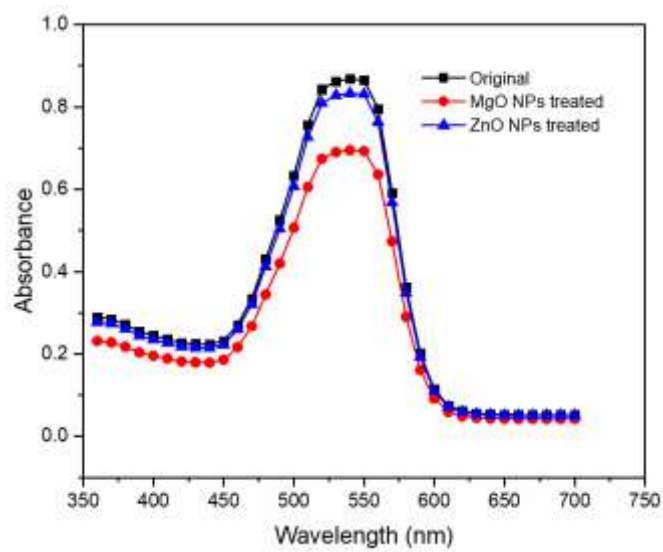


Figure 6

Table 1: Stain Grading of Uncoated and NPs Coated Cotton Fabrics

Time	Uncoated	ZnO NPs coated cotton	MgO NPs coated Cotton
At 0 hours	Grade 1	Grade 2	Grade 2
At 3 hours	Grade 2	Grade 3	Grade 3
At 5 hours	Grade 2	Grade 4	Grade 3
After washing	Grade 3	Grade 5	Grade 4

Table 2. UV (ultraviolet)-blocking properties of the uncoated, ZnO-coated and MgO-coated fabrics

Sample	UPF	UV Protection Category (ASTM D 6603)	UVA (315-400 nm)	UVB (280-315 nm)
Uncoated fabric	21 (20)	Good	5.51	4.14
ZnO NPs coated Fabric	58 (50+)	Excellent	1.65	1.21
MgO NPs coated Fabric	71 (50+)	Excellent	2.04	1.45

Comparative study of multifunctional properties of synthesised ZnO and MgO NPs for Textiles applications

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