

Use of zinc oxide nano particles for production of antimicrobial textiles

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Abstract

The application of nanoscale materials and structures, usually ranging from 1 to 100 nanometers (nm), is an emerging area of nanoscience and nanotechnology. Synthesis of noble metal nanoparticles for applications such as catalysis, electronics, textiles, environmental protection, and biotechnology is an area of constant interest. Recently, an awareness of general sanitation, contact disease transmission, and personal protection has led to the development of antimicrobial textiles. The development of antimicrobial cotton fabrics using Zinc oxide nanoparticles has been investigated in this present work. The ZnO nanoparticles were prepared by wet chemical method and were directly applied on to the 100% cotton woven fabric using pad-dry-cure method. The antibacterial activity of the finished fabrics was assessed qualitatively by agar diffusion and parallel streak method, quantitatively by percentage reduction test. The topographical analysis of the treated fabric and untreated fabric were studied and compared. The results show that the finished fabric demonstrated significant antibacterial activity against *S. aureus* in both qualitative and quantitative tests. The SEM analysis revealed the embedding of ZnO nanoparticles in treated fabrics. The wash durability study of the treated fabric was also carried out and found to withstand up to 25 wash cycles.

Keywords: Nanoparticles, zinc oxide, antimicrobial finish, wash durability

1. Introduction

Nanoscale science and technology have emerged over the past decade as the forefront of science and technologies. The intersecting fields of study that create this domain of science and engineering perfectly typify the rapid, multidisciplinary advancement of contemporary science and technology. Inorganic materials such as metal and metal oxides have attracted lots of attention over the past decade due to their ability to withstand harsh process conditions (Fu *et al.*, 2005; Makhluaf *et al.*, 2005). Of the inorganic materials, metal oxides such as TiO₂, ZnO, MgO and CaO are of particular interest as they are not only stable under harsh process conditions but also generally regarded as safe materials to human beings and animals (Stoimenov *et al.*, 2002; Fu *et al.*, 2005). The use of nanoparticles of silver and zinc oxide has been seen as a viable solution to stop infectious diseases due to the antimicrobial properties of these nanoparticles. The intrinsic properties of a metal nanoparticle are mainly determined by size, shape, composition, crystallinity and morphology (Dickson and Lyon 2000).

In view of the textile industry's innovative history, it is no wonder that nanotechnology has found its way into this sector so quickly. Nanotechnology is forecasted as the second industrial evolution in the world. The novel properties and low material consumption amount has attracted global interest across disciplines and industries. The textile sector is no exception. As stated by the "European Technological Platform for Textiles and Fashion", the textile industry to thrive must improve and reduce the costs of the processes, offer innovative products for traditional markets, develop new products for new markets. Nanotechnology can have an important role to achieve these goals and, in effect, all over the world public and private research institutions and private enterprises are actively engaged in nanotechnology research aimed at applications in the textiles sector. The competition is growing and technological innovation is crucial to keep pace with it. Health concerns along with customer satisfaction have made functionally finished textiles a fast-paced and fast growing industry.

With growth in world population and the spread of disease, the number of antibiotic resistant microorganisms is rising along with the occurrence of infections from these microorganisms. With this increase in health awareness, many people focused their attention on educating and protecting themselves against harmful pathogens. It soon became more important for antimicrobially finished textiles to protect the wearer from bacteria than it was to simply protect the garment from fiber degradation (Ye *et al.*, 2006). The need for antimicrobial textiles goes hand-in-hand with the rise in resistant strains of microorganisms. Functional textiles include everything from antimicrobial finished textiles, to durable, or permanent press finished garments, to textiles with self-cleaning properties, and also textiles with nanotechnology. With the above back ground information the present study was carried out with the main objective of evolving a simple method for the synthesis of ZnO nanoparticles, design a method to finish ZnO nanoparticles onto cotton fabrics to confer antimicrobial function and finally evaluate the finished fabrics in terms of antibacterial, wash durability and topographical function.

2. Materials and methods

2.1 Nanoparticle preparation

The zinc oxide nanoparticles were prepared by wet chemical method (Yadav *et al.*, 2006) using zinc nitrate and sodium hydroxide as precursors and soluble starch as stabilizing agent. Different concentrations of soluble starch (0.1%, 0.5% and 1.0%) were dissolved in 500 ml of distilled water by using microwave oven. Zinc nitrate, 14.874 g (0.1 M) was added in the above solution. Then the solution was kept under constant stirring using magnetic stirrer to completely dissolve the zinc nitrate.. After complete dissolution of zinc nitrate, 0.2 M of sodium hydroxide solution (20 ml was used in our study) was added under constant stirring, drop by drop touching the walls of the vessel. The reaction was allowed to proceed for 2 Hours after complete addition of sodium hydroxide. After the completion of reaction, the solution was allowed to settle for overnight and the supernatant solution was then discarded carefully. The remaining solution was centrifuged at 10, 000 X g for 10 mins and the supernatant was discarded. Thus obtained nanoparticles were washed three times using distilled water. Washing was carried out to remove the byproducts and the excessive starch that were bound with the nanoparticles. After washing, the nanoparticles were dried at 80 degree C for overnight. During drying, complete conversion of zinc hydroxide into zinc oxide takes place.

2.2 Application onto fabrics

A fine-medium weight 100% cotton woven fabric (plain weave, 75.30 g/m²; ends, 75/inch; picks, 60/inch) was used for the application purpose. ZnO nanoparticles were applied on cotton using pad-dry-cure method. The cotton fabric cut to the size of 30 ×30 cm was immersed in the solution containing ZnO (2%) and acrylic binder (1%) for 5 min and then it was passed through a padding mangle. A 100% wet pick-up was maintained for all of the treatments. After padding, the fabric was air-dried and then cured for 3 min at 140°C. The fabric was then immersed for 5 min in 2 g/l of sodium lauryl sulfate to remove unbound nanoparticles. Then the fabric was rinsed at least 10 times to completely take out all the soap solution. The fabric thus washed was air-dried. Simultaneously, bulk-ZnO coating was carried out for comparison.

2.3 Assessment of antibacterial activity

2.3.1 Qualitative tests

2.3.1.1 Agar diffusion method (Mucha *et al.*, 2002)

Bacteriostasis agar was dispensed in sterile petriplates. 24 hours broth cultures of the test organisms (*E.coli* and *S.aureus*) were used as inoculums. Using sterile cotton swab the test organisms were swabbed over the surface of the agar plates. The test fabrics (fabrics treated ZnO nanoparticles) & Control (fabrics treated with ZnO bulk) was gently pressed in the center of the mat culture. The plates were incubated at 37°C for 18-24 hours.

2.3.1.2 Parallel streak method: (AATCC Test method 147-1992)

Sterile bacteriostasis agar was dispensed in petriplates. 24 hours broth cultures of the test organisms (*E.coli* and *S.aureus*) were used as inoculums. Using 2 mm inoculation loop, 1 loop full of culture was loaded and transferred to the surface of the agar plate by making 7.5cm long parallel streaks 1cm apart in the center of the plate, without refilling the loop. The test specimen (fabrics treated ZnO nanoparticles & Control i.e. fabrics treated with ZnO bulk) was gently pressed transversely, across the five inoculums of streaks to ensure intimate contact with the agar surface. The plates were incubated at 37°C for 18-24 hours.

2.3.2 Quantitative tests

2.3.2.1 Percentage reduction test (Mucha *et al.*, 2002)

Specimens of the test material were shaken in a known concentration of bacterial suspension and the reduction in bacterial activity in standard time was measured. The efficiency of the antimicrobial treatment is determined by comparing the reduction in bacterial concentration of the treated sample with that of control sample expressed as a percentage reduction in standard time. The evaluation of modified Hohenstein test was made on the basis of the percentage reduction of bacteria by the sample. Percentage reduction was calculated using the following formula.

$$R = (A-B) / A$$

Where R is percentage reduction, A is the number of bacteria in the broth inoculated with treated test fabric sample immediately after inoculation i.e., at zero contact time and B is the number of bacteria recovered from the broth inoculated with treated test fabric sample after the desired contact period (18 hours).

2.4 Topographical analysis by SEM:

The topographical analysis of the test fabrics (finished with ZnO nanoparticles) and the fabrics finished with ZnO bulk were studied comparatively based on the Scanning Electron Microscopic analysis.

2.5 Wash durability of the finished fabric :(Sarkar et al., 2003)

The wash durability testing of the finished fabrics was carried out using a neutral soap at 40° C (+/- 2° C) for 30 minutes, keeping the material : liquor ratio at 1: 50, followed by rinsing washing and drying. After drying the test fabrics and the control were assessed for antimicrobial activity by the methods as described earlier (Sec. 2.4.3)

3. Results and discussion

The results of the qualitative antibacterial assessment by agar diffusion show that the fabric sample treated with ZnO nanoparticles showed a maximum inhibitory effect against *S.aureus* and is shown (Table 1).

Table 1. Antibacterial assessment by agar diffusion method

Fabric treated	Organism	Zone of Inhibition (in cm)				
		Trial-1	Trial-2	Trial-3	Mean	Range
Fabrics treated with ZnO nanoparticles	<i>S.aureus</i>	4.9	5.3	5.4	5.2	0.5
	<i>E.coli</i>	3.4	3.2	3.3	3.3	0.2
Fabrics treated with ZnO Bulk	<i>S.aureus</i>	2.3	2.8	2.4	2.5	0.5
	<i>E.coli</i>	1.9	1.5	1.7	1.7	0.4
Fabrics without ZnO nanoparticles (Control)	<i>S.aureus</i>	0	0	0	0	0
	<i>E.coli</i>	0	0	0	0	0

It is evident that the ZnO nanoparticles treated fabric showed higher antibacterial activity when compared with ZnO bulk treated fabrics whereas the untreated fabrics showed no antibacterial activity. In general the antibacterial activity was higher against *S.aureus* than *E.coli* in both ZnO nanoparticles and bulk treated fabrics. The comparative antibacterial activity of ZnO nanoparticles, ZnO bulk and untreated fabric (control) against *S.aureus* were shown in the Figure 2.

The antibacterial activity of the ZnO particles were studied by Zhang *et al.*, 2007. It seems that active oxygen species generated by ZnO particles could be a mechanism although there is no direct evidence from the results of this study. The presence of active oxygen species has been detected by Yamamoto *et al.*, (2000). It has already been proved that both nano-sized and micron-sized ZnO suspensions are active in inhibiting the bacteria growth; the nano-sized ZnO suspension clearly has a much higher activity than the micron-sized ZnO suspension (Zhang *et al.*, 2009). These results corresponds with the results of our study as ZnO nanoparticles treated fabrics and also the ZnO bulk treated fabrics showed antibacterial activity but the activity in the ZnO nanoparticles treated fabrics was much higher.

When assessed for antimicrobial activity by parallel streak method the ZnO nanoparticles treated fabric sample showed a maximum inhibitory effect against *S.aureus* with a zone of inhibition of 5.8 cm followed by *E.coli* with a zone of inhibition of 3.7 cm and is shown in the Figure 1.

The quantitative bacterial reduction was studied by percentage reduction test and the results were shown in the Table 2. The results of this percentage reduction test correspond with that of the agar diffusion and parallel streak method. The ZnO nanoparticles treated fabrics showed maximum percentage of reduction with a reduction percentage of 94.16% for *S.aureus* followed by 86.5% for *E.coli*. The ZnO bulk treated fabrics expressed comparatively a lower percentage of reduction. The fabrics without any treatment (control) has negative values for the percentage reduction test because the final number of cells will be much higher than the initial number of cells as it have no bactericidal activity and the results were found to be zero.

The enhanced bioactivity of ZnO nano particles was studied by Nagarajan Padmavathy *et al* 2008. Scientists (Jin *et al.*,2009) working at the US Agriculture Department's Food Safety Intervention Technologies Research Unit evaluated the antimicrobial activity of zinc oxide quantum dots (ZnO QDs), nanoparticles of purified powdered ZnO, against these pathogens and found that the ZnO nano particles have antibacterial activity.

The ZnO particles produced by wet chemical method, when observed by Scanning Electron Microscope (Figure 2) revealed that the particles are more or less spherical and the size of the particles ranges from 60-75 nm. The SEM analysis of the treated fabrics showed Zinc oxide nano particles embedded on to the fabrics (Fig. 3.1 &3.2), which is absent in case of the control fabrics i.e.

fabrics treated with ZnO bulk. ZnO of Nanosize was also produced by Professor Yong-Chien Ling and his research team and assessed its antimicrobial activity.

Figure 1. Antimicrobial assessment by Parallel Streak Method (AATCC147)

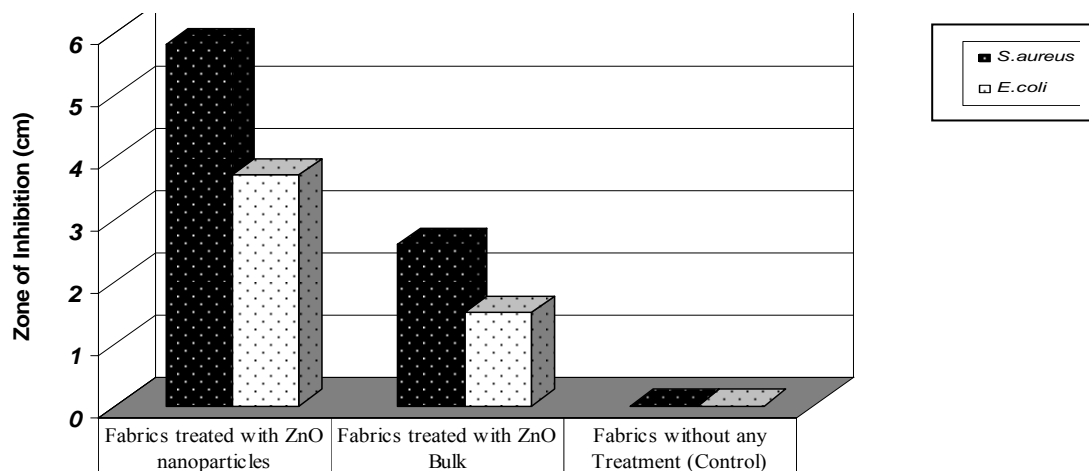


Table-2: Antibacterial Assessment by Percentage Reduction Test (AATCC 100)

Fabric tested	Organism	Initial number of bacterial cells/ml	Final number of bacterial cells/ml	% Reduction
Fabrics treated with ZnO nanoparticles	<i>S. aureus</i>	6×10^6	0.35×10^6	94.16
	<i>E. coli</i>	6×10^6	0.81×10^6	86.5
Fabrics treated with ZnO bulk	<i>S. aureus</i>	6×10^6	3.1×10^6	48.33
	<i>E. coli</i>	6×10^6	3.5×10^6	41.66
Fabrics without any treatment (control)	<i>S. aureus</i>	0	0	0
	<i>E. coli</i>	0	0	0

3.1 Wash durability

Wash durability test carried out with the test fabrics showed that the significant antimicrobial activity was actively retained in the ZnO nanoparticles treated fabrics upto 10 washes (Table-3) even after repeated wash cycles. After 10 washes the % bacterial reduction was very low and there was no activity found in the fabrics after 20 washes. Whereas the ZnO bulk treated fabrics retained the antimicrobial activity only upto 5 repeated wash cycles. The untreated control fabrics were not subjected to any wash durability test as it has no antibacterial activity.

Table-3. Wash durability testing

No. of Washing cycles	Fabrics treated with ZnO nanoparticles		Fabrics treated with ZnO bulk	
	% Bacterial Reduction		% Bacterial Reduction	
	<i>S. aureus</i>	<i>E. coli</i>	<i>S. aureus</i>	<i>E. coli</i>
1	94.05	86.28	47.27	40.22
2	93.62	85.94	37.59	32.52
5	89.42	81.38	19.49	11.24
10	74.36	69.54	0	0
15	40.25	34.96	0	0
20	12.05	9.85	0	0
25	0	0	0	0
30	0	0	0	0

There are several methods for preparing nanosized ZnO powders such as spray pyrolysis (Liu *et al.*, 1986), precipitation (Trindade *et al.*, 1994), thermal decomposition (Verges *et al.*, 1992), hydrothermal synthesis (Chen *et al.*, 2000) and electrochemical growth (Mahamuni *et al.*, 1999). Different methods yield different particle sizes of ZnO, depending on the type of precursor, the solvent, the pH and the temperature of the reacting solution. The choice of method depends on the final application. From the above results we clearly came to know about the enhanced bioactivity of ZnO nanoparticles by studying the antimicrobial activity of ZnO nanoparticles treated fabrics. The enhanced bioactivity of smaller particles is attributed to the higher surface area to volume ratio. For smaller ZnO nanoparticles, more particles are needed to cover a bacterial colony (2µm) which results in the generation of a larger number of active oxygen species (released from ZnO on the surface of the colony), which kill bacteria more effectively. ZnO nanoparticles were found to be more abrasive than bulk ZnO, and thus contribute to the greater mechanical damage of the cell membrane and the enhanced bactericidal effect of ZnO nanoparticles.

Figure 2. Antibacterial activity of (a) ZnO nanoparticle treated fabric, (b) ZnO bulk treated fabric and (c) untreated fabric (control) against *s.aureus* by Disc diffusion method

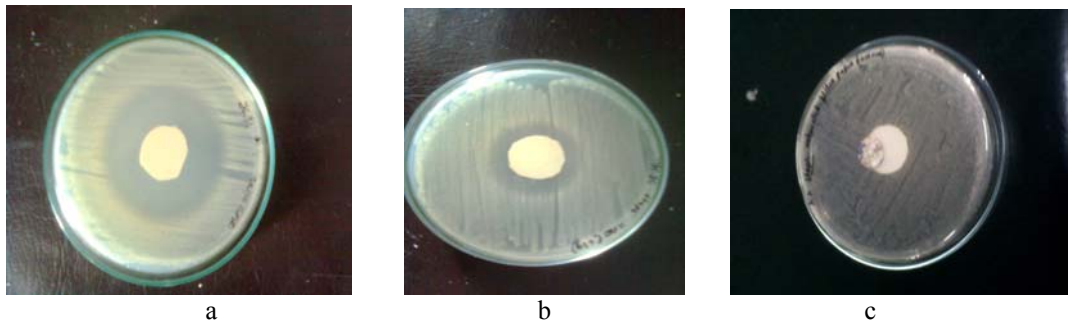


Figure 3.1. SEM images showing ZnO Nanoparticles

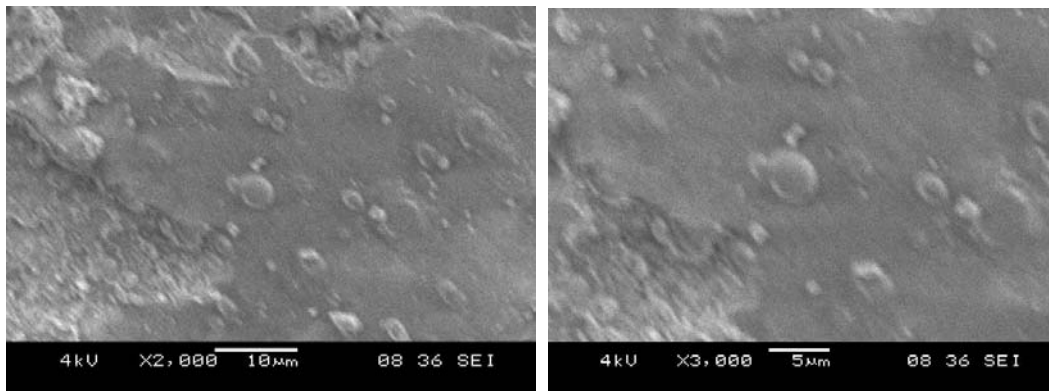
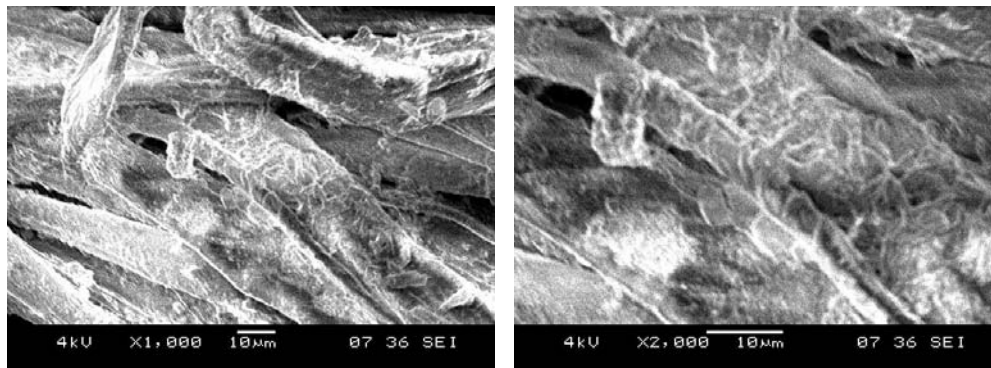


Figure 3.2. SEM images showing ZnO Nanoparticles embedded onto the fabrics



4. Conclusion

In conclusion, a simple method has been developed to prepare nano-ZnO and coat the same on cotton fabrics to impart functional properties. The nano-ZnO coated cotton fabric is found to have the antimicrobial property. It also clearly demonstrated that the ZnO nanoparticle treated fabrics showed increased antibacterial effect than the ZnO bulk treated fabrics in comparison with the untreated fabric. The results also demonstrated that higher antibacterial activity was observed against *S.aureus* than *E.coli* both in qualitative and quantitative tests. The wash durability can be enhanced by manipulating the particle concentration and size. The SEM analysis of the ZnO nanoparticles treated fabric proves the entrapment of ZnO nanoparticles in the treated fabrics. Further the padding conditions and particle size of the ZnO has to be optimised for enhanced antimicrobial effect in cotton fabrics. The technology can be further extended to polyester, silk and other fabrics. Such type of antimicrobial finish can find wide application in the health and hygiene textile sector. The result of this paper raises other areas to be concentrated for further research to answer a number of questions before a concrete conclusion could be drawn.

Acknowledgement

The authors greatly acknowledge the financial support from DRDO, Ministry of Defence, Govt. of India and the support from Principal and Secretary, PSG College of Arts & Science, Coimbatore, TamilNadu, India.

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Received December 2009

Accepted January 2010

Final acceptance in revised form March 2010