

Preparation, Characterization and Functional Analysis of Zinc Oxide Nanoparticles Coated Single Jersey Cotton Fabric

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Abstract

Among the diverse fields of application of technical textiles, which are poised for tremendous growth in fast-developing economies like those of India and China, non-implantable healthcare and hygiene products are assuming significant importance because of specificity of their end uses. Protective textiles offer protection from hazardous chemicals, microbes, heat, extreme cold and radiation and have special application potential in today's technologically advanced world. In addition to this, the advent of nanotechnology has opened innumerable new avenues giving rise to high-performance textiles and apparel. Today, the healthcare industry is increasingly concerned with the exposure and transference of various microorganisms that are commonly present in the atmosphere. Studies have been carried out in the present work to fine-tune the properties of zinc oxide nano-particles for special applications. In the current work, soluble starch (stabilizing agent), zinc oxide nitrate and sodium hydroxide (precursors) were used for the preparation of zinc oxide nanoparticles by wet chemical method. The zinc oxide nano-particles were microencapsulated and applied to single jersey cotton fabric. The antibacterial property of the coated fabric was determined quantitatively and qualitatively. Techniques such as Scanning Electron Microscopy (SEM) and physical and chemical characterisation were employed to study the phase and morphology of the nanoparticles. The results indicate that the coated fabric have high antibacterial efficiency. The nanoparticles synthesized in this work have an average size of 50 nm and the physical and chemical properties of the treated fabric are markedly different from those of the untreated fabric. Insights into zinc oxide encapsulated coated fabric prove their efficacy against microbes and hence can be used for medical apparel.

Keywords: Nanotechnology; Finishing; Nano particles; Medical apparel

Introduction

Nanotechnology is basically the engineering of functional systems at the molecular scale. In nanotechnology, the design, characterization, production, and application of structures, devices, and systems are controlled by manipulation of size and shape at the nanometer scale which enhances their characters with at least one superior characteristic or property. 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 [1].

In technical terms, the word "nano" means 10^{-9} , or one billionth of something. For comparison, a virus is roughly 100 nano meters (nm) in size. The word nanotechnology is generally used when referring to materials with the size of 0.1 to 100 nano meters; however it is also inherent that these materials should display different properties from bulk (or micrometric and larger) materials as a result of their size. These differences include physical strength, chemical reactivity, electrical conductance, magnetism, and optical effects [2].

Nanotechnology has tremendous potential to contribute to human, flourishing in socially just and environmentally sustainable ways [3]. Most of the exciting inventions in the field of nanotechnology are synthesis of nanoparticles. Nanoparticles are often defined as particles of less than 100nm in diameter that exhibit new or enhanced size-related properties compared with larger particles of the same material. Nanoparticles exist widely in the natural world and recently they are being deliberately manufactured from metal oxides.

Today, the health care industry is increasingly concerned over the exposure and transference of various micro organisms that are commonly present in the atmosphere. The increase in the presence of resistant pathogens that can be transmitted is also major concern which has led to the production and development of innovative new products in the textile industry. With the advancements in science and technology, the textile industry has produced fabric with wrinkle-free, anti felting, water repellent, stain resistant, flame resistant, anti-microbial finish developed by the incorporation of nanoparticles on them. The most necessary finish required for any textile material is the finish against micro-organism. The inherent properties of the textile fibres provide room for the growth of micro organisms and cross infections.

Besides the structure of the substrate, the chemical processes may induce the growth of microbes intensively. Antimicrobial finish to fabric has become a necessity to protect the wearer and the textile substrate itself. Under suitable conditions various types of micro-organisms like bacteria, fungi and virus deposit themselves and multiply in contact with the human bodies. These types of multiplication can cause staining, discoloration, degradation on the fabric surface and generates

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foul odor. Some type of finish is required to protect the textile surface from the growth of micro-organisms. Microbes can be categorised into three types –bacteria, fungi and algae. The first two groups are generally applicable to textiles. The below outlines show the main difference and the problems they can cause in textiles.

Why anti microbial finish?

- ❖ To protect the textile user against pathogenic organisms.
- ❖ To protect wearer against odor causing microorganisms.
- ❖ To withstand the laundry process.
- ❖ To safe guard the textiles from damage caused by mould, mildew or rot producing micro-organisms.
- ❖ To protect the textile products from deterioration, discoloration and staining.
- ❖ Helps maintain a safer, more sanitary environment.

Antimicrobial finishes and treatments within textiles can help to avoid or control cross infection, and by stopping microbial growth can extend the life time of the product.

Antimicrobial treatments

Antimicrobial treatments can be added in a number of ways including as coating to the finished fabric or fibre or by incorporating the antimicrobial agents into the fibre during the spinning process. Antimicrobial fabrics can deter microbial growth in two ways. Fabric such as linen/lamb's wool can be classified as 'Passive' as their inherent surface structure inhibits the growth of micro-organisms without the use of agents. While cotton/denim fabric are 'Active' where the active microbial agent are used to either kill or inhibit their growth (Table 1).

The antimicrobial agents used to treat the textiles falls into two categories known as Static and Cidal. Many traditional treatments fall into the 'Cidal' category and the new developments tend to favour the 'Static' type agents as these are of lower risk.

Nanofinishes fall under the 'Static' type where the finishing techniques use nanosize particles which can impart various kinds of properties for all varieties of textile materials. The study of Nanoparticles has created much interest, because of the new properties that they exhibit at the nanoscale size. First, nano materials have a relatively larger surface area when compared to the same mass of material produced in a larger form. This can make materials more chemically reactive and affect their strength or electrical properties. Second, materials can be produced as nanoscale in one dimension (for example, very thin surface coatings), in two dimensions (for example, nano wires and nano tubes) or in all three dimensions (for example, nano particles). Recent study shows that nano particles can improve

moisture absorption, protect the product from, withstand more than 50 home launderings and impart antimicrobial character to fabric.

The antibacterial activity of silver has long been known for its strong inhibitory and bactericidal effects as well as broad spectrum of antimicrobial activity. But recently scientists at Bar-Ilan University in Israel have discovered the size-dependent antibacterial effect of nano scale zinc oxide. The surface of the zinc oxide nanoparticles reacts with water to produce reactive oxygen species that destroy the bacteria cell membranes. And, if the nanoparticles are made small enough, the bacteria will actually internalize the nanoparticles, resulting in even higher killing efficiency as the cells are attacked from the inside too.

Materials and Methods

Materials required

Fabric sample with the following specifications were used for the analysis. A fine-medium weight single jersey cotton fabric was used for the application purpose.

Preparation of nanoparticles

Wet chemical method was followed for the preparation of the nano particles. 0.1% using zinc oxide nitrate as precursor and sodium hydroxide as stabilizing agent. Soluble starch in different concentration was mixed with 500 ml distilled water and was kept in the microwave oven for melting. The mixture becomes hot and is liquefied. After cooling, 0.1 M zinc oxide nitrate (14.874 g) was added to the above solution. When zinc oxide nitrate was completely dissolved, 0.2 M Sodium hydroxide (20 ml) was added in drops to this mixture along the sides of the container with constant stirring using magnetic stirrer and it was left overnight. This makes precursor and sodium hydroxide to bind together. Later the supernatant was discarded carefully using a pipette. Excess starch and zinc oxide nitrate are removed by centrifuging the remaining solution at 3000 rpm for 10 minutes. The supernatant formed again was discarded and the palette was washed three times with distilled water. The mixture was left overnight in hot air oven at 80°C. The next day the nanoparticles were scrapped. 2% Solution was prepared using distilled water.

Microencapsulation of zinc oxide nanoparticles

Microencapsulation is a process, where active biological such as enzymes, cells or therapeutics such as antibiotics or vitamins, are entrapped within a semi-permeable matrix. The resulting capsules or particles generally range from micrometers to millimeters in size [4]. Applications of microencapsulation include controlled release of the active components, particle coating, flavor stabilization, taste masking, physical/chemical stabilization, and improvement of shelf life and prevention of exposure of the active material to the surroundings [5].

There are numerous reasons for microencapsulation. In some cases, the core must be isolated from its surroundings, as in isolating vitamins from the deteriorating effects of oxygen, retarding evaporation of a volatile core, improving the handling properties of a sticky material, or isolating a reactive core from chemical attack. In other cases, the objective is not to isolate the core but to release it in a controlled manner; the rate at which it leaves the microcapsule is controlled, as in the controlled release of drugs or pesticides.

Microencapsualtion by ionic gelation method

Microcapsules containing nano-particles were prepared employing sodium alginate, the nano-particles being the core material and sodium

Microbe type	Description	Causes	Treated with
Bacteria	Simple structure Fast growing in warm and wet conditions	Unpleasant odours	Antimicrobial agents
Fungi	Complex structure Slow growing	Staining and loss of performance.	Anti mycotic agent

Table 1: Antimicrobial activity of cotton/denim fabrics using active anti microbial agents.

alginate the wall material; the microencapsulation was carried out by the ionic-gelation method.

A 3% solution of sodium alginate was prepared. Then 30 ml of lemon grass extract and 10 ml of tween were added to the polymer solution and mixed thoroughly to form smooth viscous dispersion. This was sprayed into calcium chloride solution by means of a sprayer. The droplets were retained in calcium chloride for 15 minutes. The microcapsules were obtained by decantation and repeated washing with iso-propyl alcohol followed by drying at 45°C for 12 hours. The microcapsules were then used for finishing selected fabrics.

Application of microencapsulated zinc oxide nano oxide nanoparticles of textile fabric

Single jersey cotton fabric was finished with a dispersion of the microcapsules by the exhaustion method, the solution having the following composition:

Peptone	10 g
Beef extract	5 g
Sodium chloride	5 g
Agar	1.5%
Distilled water	1000 ml

8% of citric acid was used as cross linking agent. The fabric was kept immersed in the solution at 50°C for 30 minutes. The fabric was then removed, squeezed and dried at 80°C in an oven for 5 minutes and finally cured at 120°C for 2 minutes (Figure 1).

Assesment of Antimicrobial Property of Treated Fabric

Antibacterial tests -agar diffusion method (AATCC 147)

Specimens of the test material including corresponding untreated controls of the same material were placed in intimate contact with nutrient agar, which had been previously streaked with inoculum of a test bacterium. After incubation, a clear area of interrupted growth underneath and along the sides of the test material indicated antibacterial activity of the specimen. A standard strain of bacteria, specific to the requirements of the materials under test, was used. A surface coating capable of releasing metal species to a broth of living organisms in a controlled manner is an extremely interesting material for a number of biotechnological applications.

Staphylococcus aureus (ATCC 6538) and *Escherichia coli* (ATCC 11230) were used as standard Gram positive and Gram negative

organisms respectively. AATCC bacteriostasis broth / agar medium were used as a growth medium for evaluation.

Heating to boiling was done to dispense the ingredients. A pH of 7.0 – 7.2 was used by means of 1.0 N sodium hydroxide solution. A quantity of 10.0 ± 0.5 ml of the bacteriostasis broth was dispensed in conventional bacteriological culture tubes (125 x 17 mm) and sterilised at 103 k Pa (15 psi) for 15 minutes.

Maintenance of culture of test organisms

Using a 4-mm inoculating loop, the culture was transferred daily to nutrient / bacteriostasis broth for not more than two weeks. At the conclusion of two weeks, a fresh transplant was made from stock culture. The culture was incubated at 37 ± 2°C. The stock culture maintained on nutrient agar slants was stored at 5 ± 1°C and transferred once a month to fresh agar. The purity of the culture was checked by making streak plates periodically and observed for single species-characteristic type of colonies.

Test specimens (Enzyme and Antimicrobial treated) and the untreated fabric samples (control) were cut into circular pieces of convenient size (15 mm radius).

Sterilized nutrient / bacteriostasis agar medium previously sterilized and cooled to 47 ± 2°C was dispensed by pouring 15 ± 2 ml into each of standard (15 x 100 ml) flat-bottomed petri-dishes. The agar was allowed to solidify and inoculated with a day culture (slant cultures) of the test organisms. These were placed on the petri-dishes and allowed to harden. The textile test specimen was placed on solid agar and attached to it. For conditioning, the test dish was stored for 24 hours at 5°C and then placed in an incubator. If the fabric curled, preventing intimate contact with the inoculated surface, small sterile glass plates were placed on the ends of the fabric to hold it in place. The plates were then incubated at 37°C for 18-24 hours (Figure 2).

Evaluation

At the end of the incubation time, the test specimens were observed. The agar under the sample was also evaluated. This was important if no zones of inhibition existed. This assessment was made by visual examination as well as under a microscope with 40X magnification. The evaluation was made on the basis of absence or presence of bacterial effect in the contact zone under the specimen and the possible formation of a zone of inhibition around the test specimen; the zones of bacteriostasis were measured in mm.

A clear zone of inhibition was noticed in the control or unwashed sample which was later evaluated for its anti-microbial efficacy after 5 washes and 10 washes respectively.

Wash durability

To evaluate the durability of antibacterial effect after washing, test-specimens of the treated fabric of size of 7.5 x 13.5 cm were washed according to AATCC 61(1A)-2001 test method using AATCC standards specified detergent WOB. One cycle of laundering by this method is equivalent to five typical careful hand laundings at temperature of 40 ± 3°C. The treated samples were subjected to two cycles of consecutive laundering, the first for five cycles and the second for ten cycles. The washed specimens were then tested for its antimicrobial activity at the end of the two stages of laundering.

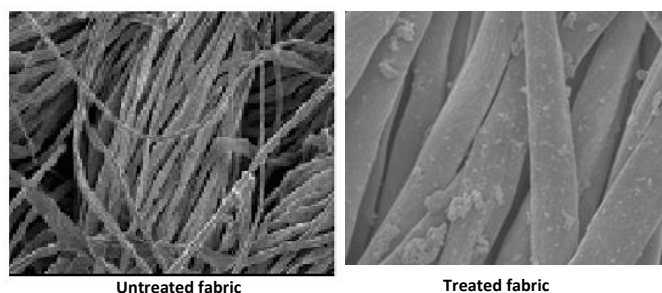


Figure 1: SEM micrograph of zinc oxide encapsulated nano particles on single jersey cotton fabric.

Antibacterial Test (Quantitative Method –Shake Flask Test - AATCC 100)

Swatches of test and control fabric already tested for antibacterial activity qualitatively were evaluated for their bacterial resistance by means of a quantitative test. Test and control swatches were inoculated with the test organisms. After incubation the bacteria were eluted from the swatches by shaking them in known amounts of neutralising solution. The number of bacteria present in the liquid was determined and the percentage of reduction in bacterial population as a result of the finish on the treated specimen was calculated.

Both *S. aureus* (ATCC 6538) and *E. coli* (ATCC 11230) were used for commercial textile samples and only *S. aureus* was used for all other treated fabrics. AATCC bacteriostasis broth / agar medium were used as a growth medium for evaluation (Table 2).

Maintenance of the culture of test organism

Using a 4 mm inoculating loop, the culture was transferred daily to the nutrient / bacteriostasis broth for two weeks. At the end of this period, a fresh transplant was made from the stock culture. The culture was incubated at $37 \pm 2^\circ\text{C}$. The stock cultures maintained on nutrient agar slants was stored at $5 \pm 1^\circ\text{C}$ and transferred once a month to fresh agar. The purity of the culture was checked by making streak plates periodically and observing for single species characteristic type of colonies. Circular swatches of 4.8 ± 0.1 cm diameter were cut out from the test fabric. The swatches were stacked in a 250 ml wide-mouth glass jar with screw cap. A swatch of the same fibre type and fabric construction as the test sample, but containing no antimicrobial finish, was used as the control. 1.0 ± 0.1 ml of an appropriate dilution of a 24 hour culture of the test organism in nutrient broth was applied.

The recovery from the untreated control fabric swatches and treated test fabric swatches at 0 contact time (placed as soon as possible after inoculation) showing a count of appropriate number of organisms were recorded.

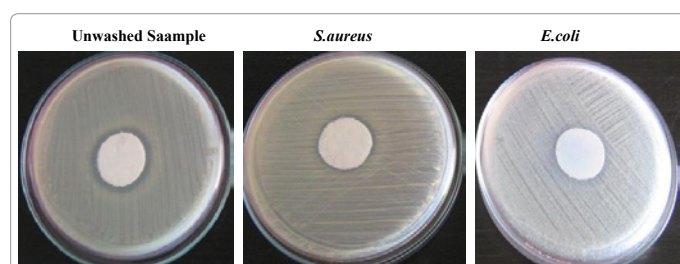


Figure 2: Antimicrobial activity of cotton fabric finished with encapsulated zinc oxide nano-particles (AATCC 147).

Test organism	Particles used	Zone of Clearance (unwashed sample) (mm)	Zone of Clearance after 5 washes (mm)	Zone of Clearance after 10 washes (mm)
<i>Staphylococcus aureus</i>	Micro-encapsulated zinc oxide nano-particles	2.8	2.3	1.9
<i>Escherichia coli</i>	Micro-encapsulated zinc oxide nano-particles	2.5	2.1	1.6

Table 2: Antimicrobial activity of cotton fabric finished with micro-encapsulated zinc oxide nano-particles (AATCC 147).

A 24 hour culture of the test organism was shaken and allowed to stand for 15-20 minutes before preparing the inoculums. The swatches were placed in a sterile Petri dish. Using a micropipette, inoculation was done making sure that there was even distribution of the inoculums. The swatches were then transferred aseptically to the jar. The jar tops were closed tightly to prevent evaporation. Immediately after inoculation about 100 ml of neutralizing solution (sterile distilled water) was added to each of the jars containing the inoculated untreated control swatches, the inoculated treated test swatches and the un-inoculated treated swatches.

The jars were shaken vigorously for one minute. The serial dilutions were made with water and plated (in duplicate) on nutrient agar. Additional jars containing inoculated untreated control swatches and jars containing inoculated treated test swatches were incubated at $37 \pm 2^\circ\text{C}$ for 18-24 hours. Similar jars were incubated for 1-6 hours to provide information about the bactericidal activity of the treatment over such periods. After incubation, about 100 ml of neutralizing solution was added to the jars containing untreated control swatches and jars containing treated test swatches. The jars were vigorously shaken for 1 minute. Serial dilutions were made and plated (in duplicate) on nutrient agar. All the plates were incubated at $37 \pm 2^\circ\text{C}$ for 48 hours.

Evaluation

The bacterial counts were reported as the number of bacteria per sample (swatches in jar) not as the number of bacteria per ml of neutralizing solution. '0' counts at 10° dilution was reported as "less than 100". The percentage reduction of bacteria by the treated specimens was calculated using the formula:

$$100 (B-A) / B = R, \text{ where}$$

R - % reduction

A - The number of bacteria recovered from the inoculated treated test specimen swatches in the jar incubated over the desired contact period.

B - The number of bacteria recovered from the inoculated treated test specimen swatches in the jar immediately after inoculation (at '0' contact time)

The bacteriostatic and bactericidal effects were calculated by using the following formula:

$$\text{Growth control} \quad F = M_b - M_a$$

$$\text{General activity (Bacteriostatic activity)} \quad L = M_a - M_c$$

$$\text{Specific activity (Bactericidal activity)} \quad S = M_b - M_c$$

where,

M_a - initial concentration of cells (both treated and untreated control)

M_b - final concentration of cells in control sample after 18 hours

M_c - final concentration of cells in test sample after 18 hours

The percentage reduction of bacteria by the treated specimen against each test organism was recorded.

The coated fabric was subjected to 5 and 10 washes respectively as was done for AATCC 147 and the washed samples were evaluated. The result of the bacterial reduction before and after washing is given in the Table 2.

Test organism	% of Reduction (unwashed sample)	% of Reduction after 5 washes	% of Reduction after 10 washes
<i>Staphylococcus aureus</i>	99.99	96.52	92
<i>Escherichia coli</i>	92.71	89.12	86

Table 3: Antimicrobial activity of cotton fabric containing zinc oxide nano-particles against micro-organisms (AATCC 100).

Charaterisation of zinc oxide nano-particle finished fabric

The surface topography observation of encapsulated zinc oxide nano-particles finished fabric was carried out with a scanning electron microscope (SEM in Figure 1). The physical properties of the finished fabric was evaluated and compared with the unfinished fabric which served as the control fabric (Table 3).

Results and Discussion

Zinc oxide nano-materials have some excellent properties like exceptional thermal, electrical, antibacterial and germicidal properties. Zinc oxide is an essential nutrient for humans as well as bacteria, but in high doses, zinc ions can cause a series of negative events in bacterial cells.

The mechanism of destruction is very different from cations, which break the cell wall and membrane so that the walls fall apart, the cell collapses and its contents leak out, and the cell is no longer viable. Antibacterial finishes prevent bacteria-causing odors in textiles and/or reduction of the changes of bacterial infections resulting from contact with contaminated textiles.

The antimicrobial activities of cotton fabrics containing microencapsulated zinc oxide nano-particles against microorganisms by AATCC 147 test method are shown in Table 1. The treated fabric showed very high antimicrobial efficiency against *Staphylococcus aureus* and *Escherichia coli* with zones of clearance of 2.8 mm and 2.5 mm respectively before washing. The zone of clearance reduces only by few millimetres after ten washes. In the AATCC 100 test method, the fabric showed a very high percentage of reduction in bacteria at 99.99% and 92.71% respectively for the two test organisms used

(Table 2). The rate of antimicrobial activity showed a marginal fall of 3.47% and 7.99% after five washes and ten washes respectively against *Staphylococcus aureus* and 3.59% and 6.71% after five washes and ten washes respectively against *Escherichia coli*.

The Zinc oxide nano-particles used in this work have been found to be about 50 µm in size and cotton fibres are known to be around 5 µm in width. It is likely that the nano-particles reduce the free movement of the cotton fibres in the fabric during the mechanical action resulting from the tensile or tearing tests. This would cause a reduction in the load-bearing elements at any given time during the tests. The overall effect is to reduce mechanical performance.

Considering that the microencapsulated Zinc oxide nano-particle coated fabric is intended for use mainly for its functional property of anti-microbial resistance, its slightly decreased resistance to mechanical forces may not really be a disadvantage. Hospital-wear for example, for which this coated fabric would be ideally suited, will normally be passed through a much smaller life-cycle compared with that for a regular garment meant for everyday use. The big advantage of the coated fabric is the fact that it displays excellent resistance to bacteria, a factor of prime importance for hospital-wear.

Conclusion

In conclusion, it may be stated that the application of microencapsulated zinc oxide nano-particles to cotton fabric imparts to it the functional property of excellent antibacterial resistance.

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