

Synthesis of zinc oxide nanoparticles and its applications in the surface modification of textile materials

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The application of nanotechnology in the textile finishing is increasingly being explored due to its unique and valuable characteristics. This has brought up many innovative finishes as well as new application techniques. The nano-finished textile materials are found to have better physical properties than the conventionally finished textiles, in areas such as anti-microbial properties, UV blocking, soil-resistance, etc. In the present work, zinc oxide nano-particles were prepared by wet chemical method using zinc nitrate and sodium hydroxide as precursors and solubilized starch as stabilizing agent. These nano-particles were impregnated onto cotton fabrics by pad-dry-cure method using acrylic binder. A fine medium weight cotton fabric samples were used for this. The aims are to impart anti-microbial functions to the textile substrate and the functional properties of coated fabrics. The nano-ZnO impregnated cotton fabrics showed excellent antimicrobial activity against two types of representative bacteria viz. gram-positive organism (*S. aureus*) and gram-negative organism (*E. coli*).

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Keywords: Zinc oxide nano-particles, wet chemical method, zinc nitrate, sodium hydroxide, pad-dry-cure method.

1. Introduction

Over recent years one can observe increasing interest in the antibacterial finishing of textiles, many textiles currently used in hospitals and hotels constitute a potential source of infection and disease caused by micro-organisms as a result of secondary infection. Medical and hygienic applications of textiles have become important areas in the textile industry. Antimicrobial properties can be imparted to textiles by physical or chemical treatment with appropriate agents. Textile materials with such properties are grouped into two categories, i.e. fabrics with unstable properties and those durably functionalized. Unstable bio active properties can be easily obtained by finishing processes, but they are quickly lost during laundering. In this case antibacterial agents are incorporated into textile in the process of wet finishing. The antibacterial properties of textiles decay if the fabrics are impregnated with antibacterial agents only, with no covalent bonds between each other. zinc oxide antiparticles which have an average size of 40 nm, were coated on the bleached cotton fabrics (plain weave, 40 s count) using acrylic binder and functional properties

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of coated fabrics were studied. On an average of 75%, UV blocking was recorded for the cotton fabrics treated with 2% ZnO nanoparticles. Air permeability of the nano-ZnO coated fabrics was significantly higher than the control, hence the increased breathability. In case of nano-ZnO coated fabric, due to its nano-size and uniform distribution, friction was significantly lower than the bulk-ZnO coated fabric as studied by Instron® Automated Materials Testing System. Nano-ZnO provides effective photo catalytic properties once it is illuminated by light, and so it is employed to impart anti-bacterial properties to textiles. Hence the present study was planned with the main objective of using zinc oxide nano particles to impart antibacterial and antifungal properties in 100% cotton fabrics and also to standardize the preparation of the zinc oxide nanoparticles under laboratory conditions.

2. Experimental methods

The methods and testing process carried out are described here below. All the chemicals used are of analytical grade without further purification. Different concentrations of soluble starch (0.5 % & 1 %) were dissolved in 500 cc of distilled water by using microwave oven. Zinc nitrate hexahydrate, 14.874 g (0.1 mol) was added in the above starch solution. Then, the solution was kept under constant stirring using magnetic stirrer to completely dissolve the zinc nitrate. After complete dissolution, 500 cc of NaOH was added drop-wise under constant stirring, drop by drop touching the walls of the vessel. The clear aqueous solution gets turned into a milky white colloid without any precipitation. The reaction was allowed to proceed for 2 hours after complete addition of NaOH.

After the completion of the reaction, the solution was allowed to settle for overnight and the supernatant solution was discarded carefully. The remaining solution was centrifuged at 3000 rpm for 10 min and the supernatant solution was discarded. The Nano-ZnO that is thus obtained was washed thrice using distilled water to remove the byproducts and the excessive starch that were bound with Nano-particles. After complete washing, the Nano-ZnO was dried at 80 °C for 3 hours for complete conversion of Zn (OH)₂ to ZnO (solid) and then it is converted in to powder form. 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 X 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, which was running at a speed of 15 m/min with a pressure of 15 kgf/cm² to remove excess solution. 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.

3. Results and discussion

ANTIBACTERIAL TESTING

To test the antibacterial activity of fabrics, there are many standard methods, like AATCC 147, AATCC 100 and AATCC 30, etc.

Basically two test organisms are used

Escherichia coli – Gram negative organism

Staphylococcus aureus – Gram positive organism

AATCC 147 (QUALITATIVE TEST)

Sterile AATCC bacteriostasis agar medium was dispensed in to the sterile petridishes.

Overnight culture were used as a inoculum

By using sterile swab the test organism were inoculated over the surface of the agar plate.

The test specimens were gently pressed in the center of the Mat culture.

The plates were incubated at 37°C for 18-24 hrs.

AATCC 100 (QUANTITATIVE TEST)

About 5.0 cm diameter of the treated fabric was taken and it was immersed in sterile AATCC broth with 0.1 ml inoculum of each culture (*Staphylococcus aureus* and *Escherichia coli*) and left overnight at 37° C in shaker. Control was also maintained with untreated fabric.

AATCC broth with the fabric was taken for appropriate dilutions.(101-107)

Prepare AATCC agar medium allow it to solidify.

3.1. Antibacterial Qualitative Assessment of the nano-ZnO impregnated cotton fabrics

(Method: AATCC 147)

Test Organism Used: *Escherichia coli* ATCC 11229 & *Staphylococcus aureus* ATCC 6538 Initial inoculum: *E. coli* – 2.7×10^9 cfu/ml *S. aureus* – 2.4×10^9 cfu/ml

Samples	Antibacterial activity (Zone of Inhibition in mm)	
	<i>Escherichia coli</i>	<i>Staphylococcus aureus</i>
Untreated	0	0
Treated	24	27

3.2. Antibacterial Qualitative Assessment of the nano-ZnO impregnated cotton fabrics

(Method: AATCC 100)

Test Organism Used: *Escherichia coli* ATCC 11229 & *Staphylococcus aureus* ATCC 6538 Initial inoculum: *E. coli* – 2.7×10^9 cfu/ml
S. aureus – 2.4×10^9 cfu/ml

Samples	Bacterial reduction (in %)	
	<i>Escherichia coli</i>	<i>Staphylococcus aureus</i>
Untreated	0	0
Treated	80	100

3.3. Analysis of fabric tear strength between untreated & treated fabric

S. No	Type of test performed	Test Method	Result
1	Fabric – Tear strength (Untreated Fabric)	ASTMD-2261-96	Warp. gf 563.2
			Weft gf. 534.4
2	Fabric – Tear strength (Treated fabric)	ASTMD-2261-96	Warp. gf 556.8
			Weft gf. 556.8

3.4. Analysis of fabric stiffness between untreated & treated fabric

S. No	Type of test performed	Test Method	Result
1	Fabric – Stiffness (Bending length in cm) (Untreated Fabric)	BS:3356-61	Warp. 1.98
			Weft 1.65
2	Fabric – Stiffness (Bending length in cm) (Treated Fabric)	BS:3356-61	Warp. 1.73
			Weft 1.63

3.5. Analysis of fabric tensile strength between untreated & treated fabric (strength)

S. No	Type of test performed	Test Method	Result
1	Fabric – Tensile Strength (Untreated Fabric)	ASTMD-5034-95	Warp. Strength Kg 26.40
			Weft Strength Kg 24.98
2	Fabric – Tensile Strength (Treated Fabric)	ASTMD-5034-95	Warp. Strength Kg 25.49
			Weft Strength Kg 20.67

3.6. Analysis of fabric tensile strength between untreated & treated fabric (elongation%)

S. No	Type of test performed	Test Method	Result
1	Fabric – Tensile Strength (Untreated Fabric)	ASTMD-5034-95	Warp. Elongation % 6.9
			Weft Elongation % 17.35
2	Fabric – Tensile Strength (Treated Fabric)	ASTMD-5034-95	Warp. Elongation % 12.05
			Weft Elongation % 16.15

3.7. Air permeability

S. No	Type of test performed	Test Method	Result
1	Air permeability ($\text{cm}^3 / \text{cm}^2/\text{sec}$) Untreated fabric and Treated fabric	BS : 5636-90	24.9 & 16.4

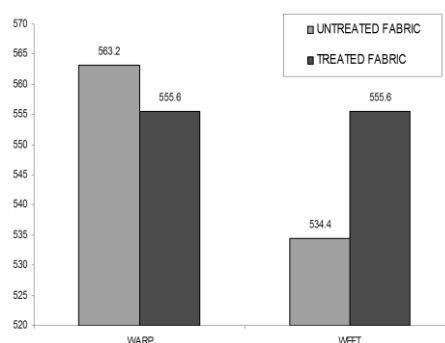


Fig. 1. Analysis of fabric tear strength between untreated & treated fabric.

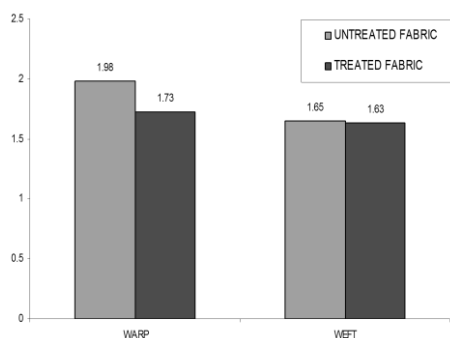


Fig. 2. Analysis of fabric stiffness between untreated & treated fabric.

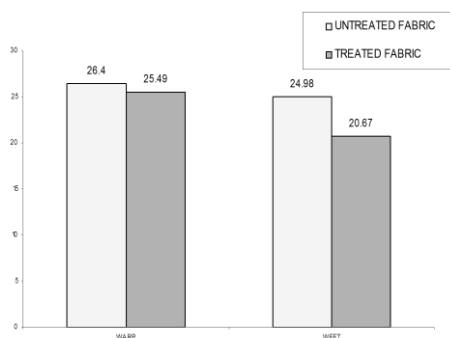


Fig. 3. Analysis of fabric tensile strength between untreated & treated fabric (strength).

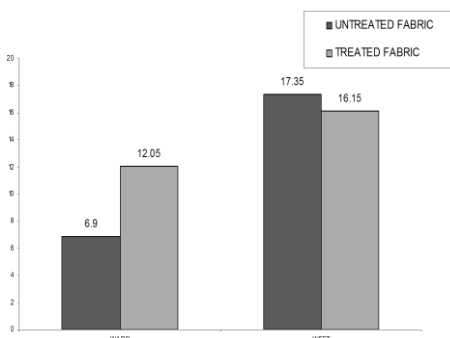


Fig. 4. Analysis of fabric tensile strength between untreated & treated fabric (elongation%).

4. Conclusions

Based on the analysis of the results for the experimental study the following conclusions can be arrived: This work provides a novel and simple method for aqueous preparation of ZnO and their application on to cotton fabrics to impart antibacterial functions. The ZnO nano-particles were finished inside the cotton with a pad dry cure technique which successfully dispersed. The nano-particles were analyzed through electron microscopy,

It is also shown that the nano-ZnO impregnated onto cotton textiles showed excellent antibacterial activity against two representative bacteria, *S. aureus* and *E. coli*.

The synthesis of the ZnO nanoparticles have carried out successfully under laboratory condition. The formation of nano particles were standardised under optimal conditions.

The characterization of the produced ZnO nanoparticles were done and the size and other characters were confirmed. The fabric were selected and finished with ZnO nanoparticles. Pad dry cure method was found to be efficient for finishing the antimicrobial efficiency of the finished fabrics using standard AATCC methods were validated and found to be efficient.

The physio chemical properties of the finished fabrics under standard conditions were measured and found to be under the permissible limits. Undoubtedly, nanotechnology holds an

enormously promising future for textiles. Such result can be exploited for the commercialization in the case of consumer textiles.

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