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# FUNCTIONALISATION OF POLYCOTTON FABRIC WITH HYDROTHERMALLY SYNTHESIZED ZnO NANOPARTICLES

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## ABSTRACT
Functional property of the inorganic materials are drawing increasing attention in the textile application. Zinc oxide nanoparticles were prepared by hydrothermal method using zinc nitrate as precursor and Cetyl Trimethyl bromide as surfactant. The nanoparticles were characterized by Dynamic light scattering, Scanning electron microscope, X-ray diffraction and Diffused reflectance spectroscopy. Then the ZnO nanoparticles were treated on polycotton fabric by pad dry cure method. The treated fabrics were assessed for microbial resistance against bacteria (*E.coli, S.aureus*) and fungi (*A.niger, C.albicans*) by qualitative method. The wash durability of the treated samples were tested using AATCC100 method and the fabric showed antibacterial activity even after 30 washes.

**KEYWORDS**: Antimicrobial textiles, metal oxide nanoparticles, Pad-dry cure method

## INTRODUCTION
Textiles have always played a central role in the evolution of human culture by being at the forefront of both technological and artistic development. Textiles can be exposed to microbes (bacteria, fungi, algae) during production and usage. Microbes are normally found on human skin, nasal cavities and other areas [1]. The inherent properties of the textile fibers provide room for the growth of micro-organisms and it can survive for prolonged periods [2] [3]. Microbial shedding from our body contributes to microorganism spreading into a textile material, either directly in clothes or on surrounding textile materials. Recent studies strongly support that contamination of textiles in clinical settings may contribute to the dispersal of pathogens to the air which then settle down and infect the immediate and non-immediate environment. It is one of the most probable causes of hospital infections [4]. Consumers demand for hygienic clothing had created a substantial market for antimicrobial textile products [5].There are many antimicrobial agents used in textile applications such as aromatic halogen compounds, benzoic esters, organometallics, metal salts, quaternary ammonium salts and chitosan [6]. Inorganic materials such as metals and metal oxides have attracted lots of attention over the past decade due to their ability to withstand harsh process conditions [7] [8]. Zinc oxide has a direct band gap of (3.37 eV) and is widely used in different areas because of its unique photo-catalytic, electrical, electronic, optical, dermatological and antibacterial properties. Mostly ZnO nanoparticles are synthesized by traditional high temperature solid state method which is energy consuming and difficult to control the particle properties [9]. Hydrothermal technique is a promising alternative synthetic method because of the low process temperature, control on particle size, catalyst-free growth, low cost, homogeneity, environmental friendliness and less hazardous [10].

## MATERIALS AND METHODS

**Synthesis of zinc oxide nanoparticles:**

Zinc nitrate (0.1M) solution was prepared and cetyl trimethyl ammonium bromide (1%) was added to the solution and stirred. Then equimolar concentration of sodium hydroxide was added dropwise and stirred. The contents were then transferred to a teflon lined autoclave and heated for 12 hours at four different temperatures 140 ºC, 160 ºC, 180 ºC and 200 ºC.

**Characterization of nanoparticles:**

Zinc oxide nanoparticles were analyzed using various techniques. Horiba SZ100 Particle Size Analyzer was used to analyze the average particle size, Panalytical Xpert Pro X-Ray Diffractometer with Cu Kα radiation of wavelength 1.54 Å was used to confirm the crystallinity, Analytik Jena Specord 210 Plus UV-Visible Spectrometer was used to analyze the absorption spectrum and Carl Zeiss Sigma Scanning Electron Microscope was used to study the morphology of the prepared zinc oxide nanoparticles.

**Finishing and characterization of fabric with nanoparticles:**

The fabric was treated with zinc oxide nanoparticles by Pad-dry cure method. The polycotton fabric was dried at 100 °C for 4 mins to remove the moisture content. The dried fabric cut into the size of 30 ×30 cm was immersed in the solution containing zinc oxide nanoparticles (2%) and acrylic binder (1%) for 10 min and then it was passed through a padding mangle running at the speed of 20 rpm and with pressure of 1.0 $kg/cm^{-2}$. A 100% wet pick-up was maintained for the treatment. 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 in distilled water to completely remove the soap solution [11]. Bruker Alpha T Fourier Transform Infra-Red Spectrometer was used to confirm the presence of zinc oxide nanoparticles in treated poly cotton fabric.

**Assessment of Antimicrobial activity:**

Antimicrobial activity was evaluated by qualitative test method and the wash durability of the finished fabric was assessed by quantitative test method. The following are the descriptions of test methods employed for this study.

*Parallel streak method (AATCC 147):*

The test organism (*Staphylococcus aureus* and *Escherichia coli*) was inoculated on the surface of the agar plate by making five parallel inoculums streaks without refilling the loop. The treated and untreated samples cut in to pieces of 25 mm x 50 mm size finished with the zinc oxide nanoparticles was gently pressed transversely, across the five inoculums of streaks to ensure intimate contact with agar surface. The plates were incubated at 37 ºC for 18-24 hours. After incubation, a streak of interrupted growth underneath and along the side of the test material indicates antibacterial effectiveness of the fabric. The inoculated plates were examined for the interruption of growth along the streaks of inoculum beneath the fabric and for a clear zone of inhibition beyond the fabric edge. The average width of the zone of inhibition around the test specimen was calculated in mm using the formula,

$$\text{Zone of inhibition (mm)} = (T - I) / 2 \qquad (1)$$

where, T referred to width of zone of inhibition and I referred to width of specimen [12].

*Assessment of antifungal activity - Agar diffusion method (AATCC 30):*

Potato dextrose agar medium was prepared and dispensed in petridish and the uniform spore suspension of *Aspergillus niger* and *Candida albicans* were inoculated into 50±2 ml of sterile distilled water containing few glass beads and shaken vigorously to bring the spores into suspension. About 1.0±0.1 ml of inoculum was distributed evenly over the surface of the agar. The test specimens were cut into 3.8±0.8 cm and placed in contact with hardened agar medium over which 0.2±0.001 ml of the inoculums was evenly distributed by means of a sterile pipette. The plates were incubated at 27 ºC for 5 days. At the end of the incubation period the antifungal activity was reported by measuring the zone of mycostasis underneath and alongside of the fabric [13].

*Wash Durability Test:*

The treated fabrics were tested for their wash fastness using wash durability testing. Washing was carried out by using a neutral soap (5gpl) at 40±2 ºC for 30 minutes, keeping the material:liquor ratio at 1:50, followed by rinsing, washing and drying in an industrial laundrometer [14]. After drying, the test samples were assessed for antimicrobial activity using AATCC 100 procedure. Washing was carried up to 30 laundering cycles and results were evaluated after counting the bacterial colonies upon incubation and calculated for the bacterial reduction using the formula as in quantitative bacterial reduction test (AATCC 100).

*Quantitative Bacterial Reduction Test (AATCC 100):*
About 1 ml of the test inocula (*Staphylococcus aureus* and *Escherichia coli)* were loaded on the swatches (treated and untreated) of 4.8±0.1cm diameter. They were then transferred to the respectively labeled sterile AATCC Bacteriostasis broth. After an incubation of 24 hours, serial dilutions were made up to $10^{-7}$ for all the samples [15]. About 0.1 ml sample from each dilution were spread plated on to the sterile AATCC Bacteriostasis agar plates and incubated at 37 ºC for 24 hours. The inoculated plates were examined for the presence of bacterial colonies. The percentage reduction of bacteria by the treatment can be calculated by the formula (2)

$$100 (B - A) / B = R \qquad (2)$$

where R = % reduction of A and B are the number of bacteria recovered from the inoculated treated and untreated swatches respectively.

## RESULTS AND DISCUSSION

**Material Characterization:**

X-Ray diffraction pattern, particle size analysis, scanning electron microscope image and diffused reflectance of spectrum the synthesized zinc oxide nanoparticles were given in the figure 1. XRD pattern of the zinc oxide nanoparticles matches with the zinc oxide pattern in the JCPDS (PDF-792205) and has a hexagonal crystal structure. By comparing the diffraction pattern of nanoparticles prepared at 140 ºC, 160 ºC, 180 ºC and 200 ºC, crystallinity improved with the increase in temperature, however nanoparticles prepared at 180 ºC and 200 ºC showed no significant difference. The average particle size of nanoparticles prepared at 140 ºC, 160 ºC, 180 ºC and 200 ºC were found to be 71 nm, 80 nm, 89 nm, 114 nm respectively, showing increase in particle size with increase in temperature. Eventhough the zinc oxide nanoparticles prepared at 140 ºC and 160 ºC were lesser in size they were not crystalline at the same time the nanoparticles prepared at 200 ºC was crystalline but higher in size. Zinc oxide nanoparticles prepared at 180 ºC was found to be crystalline and had comparably better particle size and it was taken for further studies. The size of the zinc oxide nanoparticles was confirmed to be in the range of 50 nm using scanning electron microscope. The band gap of the nanoparticles was calculated to be 3.36 eV from the reflectance spectrum using the equation E = hν and the bandgap matches with that of the zinc oxide.

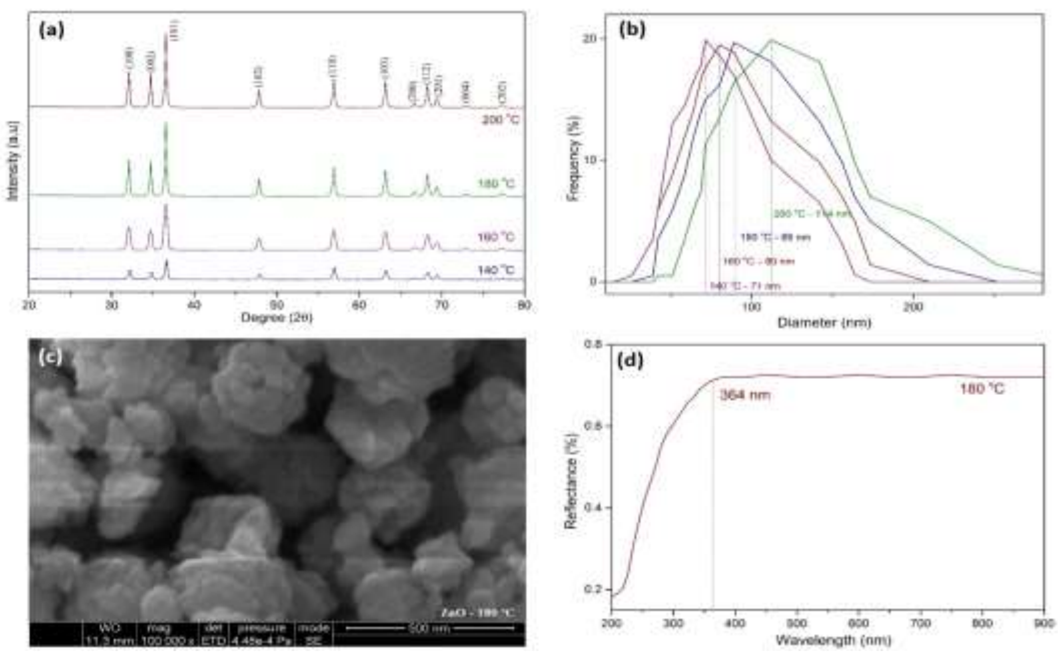


***Figure 1. Material Characterization of ZnO Nanoparticles a)X-Ray Diffraction Pattern, b)Particle Size Analysis, c)Scanning Electron Microscope Image(180, d) Diffused Reflectance Spectrum***

**Characterization of Finished Fabric**

The polycotton fabric was finished with zinc oxide nanoparticles prepared at 180 ºC. FTIR spectrum of the zinc oxide, polycotton and zinc oxide nanoparticles treated polycotton are given in the figure 2. Peak at 430 $cm^{-1}$ corresponding to the metal oxide (Zn-O) bonding was found in of both zinc oxide and zinc oxide treated polycotton spectrum, thus confirming the presence of nanoparticles in treated fabric. Remaining peaks at 712 $cm^{-1}$, 1015 $cm^{-1}$, 1236 $cm^{-1}$, 1711 $cm^{-1}$, 2870 $cm^{-1}$, 3278 $cm^{-1}$ of the untreated and treated fabric were attributed to C-H, C-O, C-N, C=O, C-H and adsorbed hydroxyl groups respectively [16]. Peaks at 1110 $cm^{-1}$, 1400 $cm^{-1}$ and 1625 $cm^{-1}$ of the zinc oxide spectrum were due the C-N, C-C and N-H groups respectively. These peaks were due to the residual surfactant from the nanomaterial synthesis process.

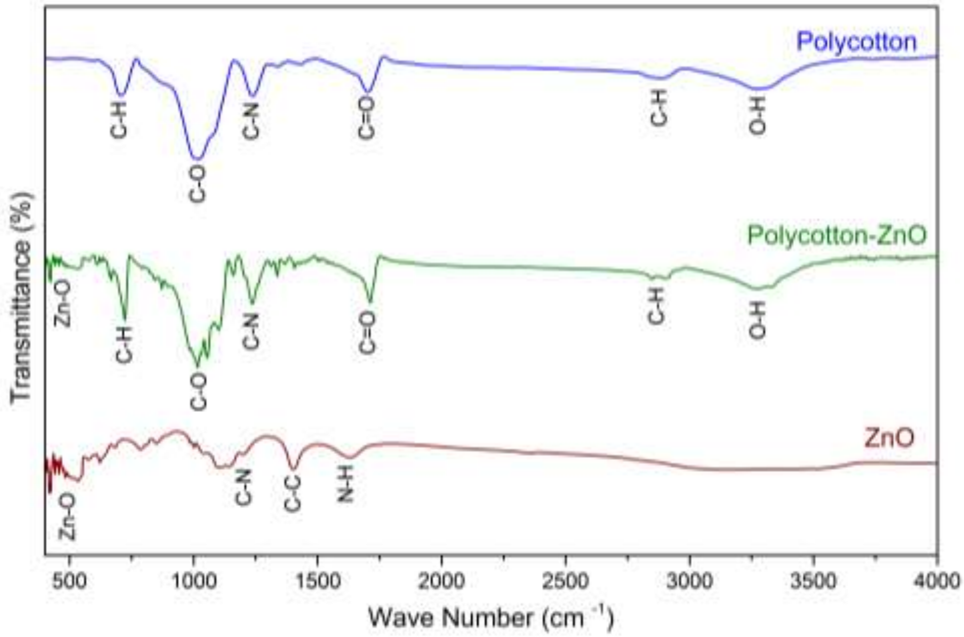


*Figure 2. FTIR spectrum of polycotton, ZnO nanoparticle treated polycotton and ZnO nanoparticle*

**Antimicrobial Efficiency of Finished Fabric**

The results of antibacterial activity of ZnO nanoparticles treated polycotton fabric was assessed qualitatively by parallel streak method. Figure 3 shows a clear zone of inhibition around ZnO treated fabric whereas the control fabric shows no sign of inhibition. The highest inhibitory effect was observed against *Staphylococcus aureus* about 19.8 mm zone of inhibition followed by *Escherichia coli* with 17.2 mm zone of inhibition. *Singh et al*., [17] have reported that the ZnO treated fabric disc exhibited pronounced activity of 18.5 mm against *S. aureus* followed by *E. coli* with a zone of inhibition of 13.5 mm diameter. 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.*, [18].

When assessed for antifungal activity by AATCC 30 test method the ZnO nanoparticles treated fabric sample showed a maximum inhibitory effect against *A.niger* with a zone of inhibition of 9.3 mm followed by *C.albicans* with a zone of inhibition of 5.2 cm and is shown in the table 1. The wash durability of the finished samples were determined by performing wash durability test. The percentage reduction of antibacterial activity for 30 laundering cycles was determined using AATCC100 the results of which is presented in the table 2. From the table the treated fabric showed a very high percentage of reduction in bacteria at 93% and 89% respectively for the two test organisms used after 5 washes. It is evident that, the ZnO nanoparticles treated sample has showed the wash durability of 20% in *S. aureus* after 30 washes were as 19% in *E. coli*.

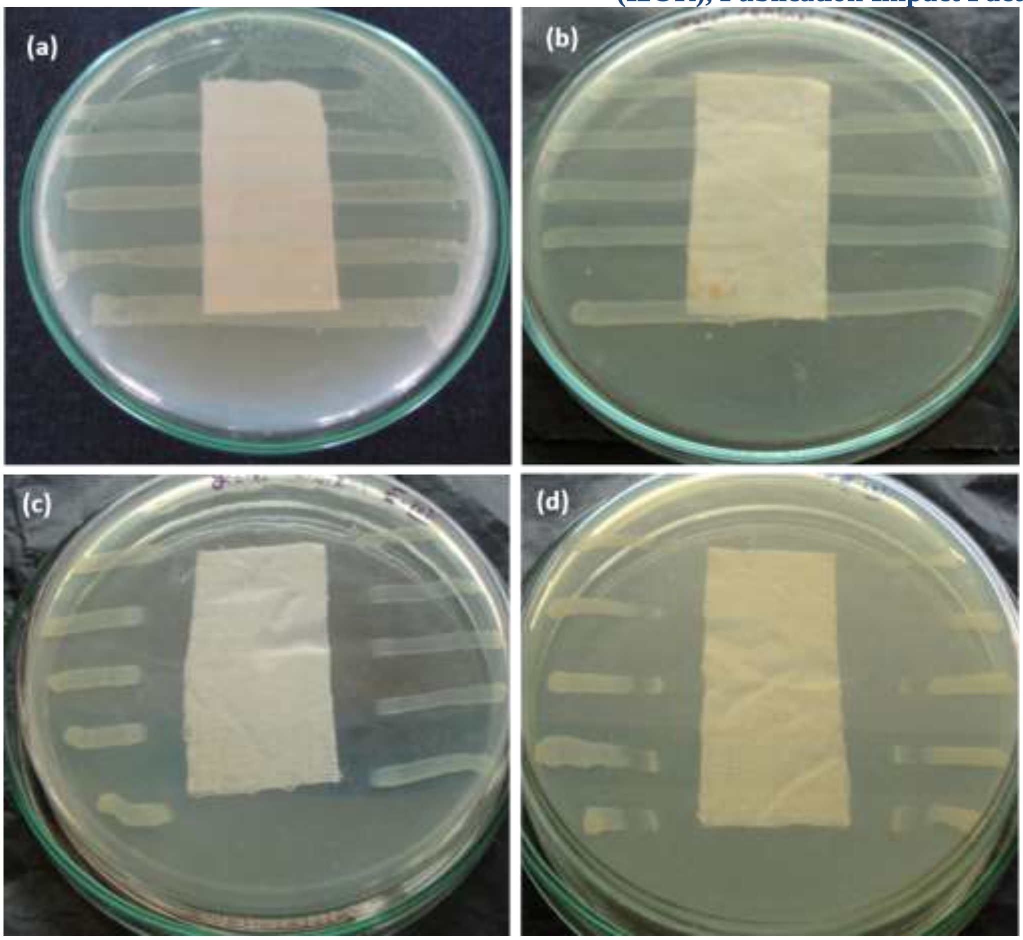


*Figure 3. Control Fabric: a) E.coli, b) S.aureus and ZnO Treated fabric: c)E.coli), d) S.aureus*

*Table 1. Assessment of antifungal activity - Agar diffusion method (AATCC 30)*

| S.No | Fabric Treatment | Antifungal Activity | |
|---|---|---|---|
| | | ***A. niger*** | ***C. albicans*** |
| 1 | Control Fabric | 0 | 0 |
| 2 | ZnO Treated Fabric | 9.3 | 5.2 |

*Table 2. Wash Durability of the Treated Fabrics by AATCC 100*

| **S.No** | **No. of laundering cycles** | **Treated samples** | |
|---|---|---|---|
| | | **Antibacterial Activity (Bacterial Reduction %)** | |
| | | ***S.aureus*** | ***E.coli*** |
| 1 | 5 | 93 | 89 |
| 2 | 10 | 85 | 77 |
| 3 | 15 | 79 | 68 |
| 4 | 20 | 67 | 43 |
| 5 | 25 | 40 | 34 |
| 6 | 30 | 27 | 19 |

## CONCLUSION

Zinc oxide nanoparticles were synthesized by hydrothermal method and characterized using various techniques. The nanoparticles prepared at 180 ºC was found be crystalline as well as lesser in particle size and was used to treat the polycotton fabric. The zinc oxide nanoparticle treated fabric showed a significant against bacterial and antifungal cultures. Also they showed a good wash durability even after 30 washes.

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