International Journal of Chemical, Materials and Biomolecular Sciences
ISSN: 2415-6620
Vol:14, No:11, 2020

Synthesis, Characterization and Coating of the Zinc Oxide Nanoparticles on Cotton Fabric by Mechanical Thermo-Fixation Techniques to Impart Antimicrobial Activity

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Abstract**—**The present study reports the synthesis, characterization and application of nano-sized zinc-oxide (ZnO) particles on a cotton fabric surface. The aim of the investigations is to impart the antimicrobial activity on textile cloth. Nanoparticle is synthesized by wet chemical method from zinc sulphate and sodium hydroxide. SEM (scanning electron micrograph) images are taken to demonstrate the surface morphology of nanoparticles. XRD analysis is done to determine the crystal size of the nanoparticle. With the conformation of nanoformation, the cotton woven fabric is treated with ZnO nanoparticle by mechanical thermo-fixation (pad-dry-cure) technique. To increase the wash durability of nano treated fabric, an acrylic binder is used as a fixing agent. The treated fabric shows up to 90% bacterial reduction for *S. aureus* (*Staphylococcus aureus*) and 87% for *E. coli* (*Escherichia coli)* which is appreciable for bacteria protective clothing.

Keywords**—**Nanoparticle, zinc oxide, cotton fabric, antibacterial activity, binder.

I. Introduction

NANO-science and use of nanoparticles are considered to be the key technology for the recent time. It is a growing interdisciplinary technology and seen as a new industrial revolution. According to the National Nanotechnology Initiative (NNI), nanotechnology is defined as utilization of structure with at least one dimension of nanometer size for the construction of materials, devices or systems with novel or significantly improved properties due to their nano-size [1]. The chemical and physical properties of a material changes from the bulk to the nanometer scale. So, nanoparticles are more reactive because of the high ratio of the surface area to its volume. Use of nano technology is increasing day by day due to its huge economic potential. The textile sector is also advancing by taking the benefits of nanoparticles. Moreover, increasing customer demand for durable and functional apparel manufactured in a sustainable manner has created an opportunity for nanomaterials to be integrated into textile substrates [2].

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Various nanoparticles like silver, ZnO, TiO_2, SiO_2 are used by the researchers to impart functional property and to meet the demand of the modern market. Those nanoparticles are mainly used as coating on a fabric surface by gun spray, padding and in-situ. However, the durability of those nanoparticles is not satisfactory. Furthermore, ZnO is highly attractive material because of its unique properties, such as optical transparency, electric conductivity, piezoelectricity, and near-UV emission and antimicrobial activity [3]-[6].

In the current study, ZnO nanoparticle is applied on cotton by mechanical thermo fixation method along with an acrylic binder. This binder makes a cross linking reaction with the fiber and nanoparticles which increases the strength of bonding of nanoparticles. So, the antimicrobial activity and the durability of nanoparticles on nano treated fabric will improve.

II. Experimental Section

Materials

Fabric of 100% cotton containing plain weave is collected from local industry, named the SIM group (Vulta, Rupganj, Narayanganj, Bangladesh) with 80 ends per inch (EPI) and 75 picks per inch (PPI) while GSM is 151 (grams per square meter). The raw cotton fabric is then scoured and bleached [7] before nanocoating to impart absorbency and permanent whiteness. The synthesis chemical zinc sulphate heptahydrate ($ZnSO_4.7H_2O$, 99% purity) is purchased from Merck Life Science Private Ltd, Mumbai, India. Ethanol and sodium hydroxide are purchased from Sigma Aldrich, Germany. Acrylic binder, Pretreatment chemical: wetting agent, sequestering agent, detergent, caustic soda, and hydrogen peroxide are collected from Orient Chemical Industries Co., Ltd.

Synthesis of ZnO Nanoparticles

The experimental setup of ZnO nanoparticle synthesis is shown in Fig. 1. Wet chemical process is applied to synthesized ZnO nanoparticle by following the method used by [8] with some modification. An aqueous solution of 0.2 M zinc sulfate ($ZnSO_4$) is made from zinc sulfate heptahydrate ($ZnSO_4.7H_2O$) in de-ionized water. Then 25 ml of 0.2 M NaOH (pH = 13.8) solution is prepared separately with purified water. The reaction is performed by slowly dropping

of NaOH into $ZnSO_4$ solution. The bath is placed on a magnetic stirrer for 30 minutes and kept for 4 hours at 60 °C temperature. The nanoparticles are obtained by centrifuging and drying at 60 °C after washing.

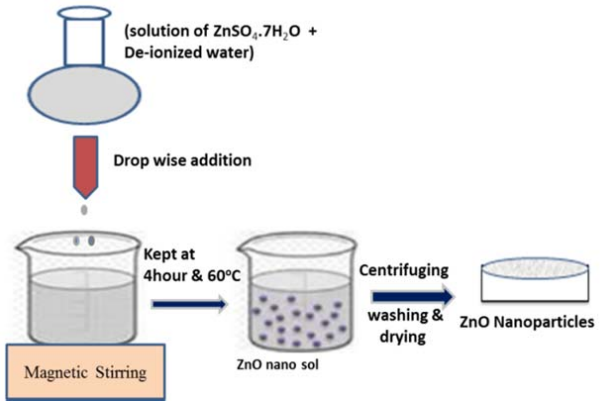

Fig. 1 Experimental set-up for the synthesis of ZnO nanoparticle

Application of Nanoparticle by Mechanical Thermo-Fixation

After synthesis, the ZnO nanoparticles are applied on cotton fabric by pad, dry and cure method. The experimental sequence is shown in Fig. 2. A solution of 2% (weight basis) of ZnO nanoparticle and 1% acrylic binder is dispersed in propanol. One piece of fabric is padded with the prescribed solution of nanoparticle at 73% pick up. The fabric is dried in a pre-heated oven at 90 °C for 10 minutes. Finally, it is cured at 150 °C for 5 minutes which makes thermal fixation of ZnO nanoparticles into the fabric.

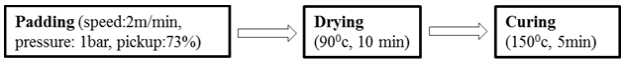

Fig. 2 Experimental sequence of thermo-fixation coating of nanoparticles on fabric surface

IV. Results and Discussion

Characterization of Nanoparticle

The synthesized nanoparticles are characterized by SEM and X-ray diffraction analysis. A field emission electron microscope: JSM-600, Tokyo, Japan, is used to determine the feature, surface characteristics and approximate size of the discrete particles. Fig. 3 shows the individual nano particle in various sizes and shapes. The SEM images show the particles with nearly spherical shape with various diameters. The approximate size is traced as 30 nm-100 nm from SEM. The crystalline shape and size is also obtained by X- Ray Diffractometer: Phillips, X'pert PRO, Holland. Fig. 4 shows the XRD pattern of ZnO nanoparticles. The measurement is carried out at scanning rate of 8°/min in 2θ range of 20°–80°, using Debye-Scherrer formula: Particle Size = (0.9 x λ)/(d cosθ), where λ = 1.54060 Å, 0.9 x λ = 1.38654, θ = 2θ/2, d = the full width at half maximum intensity of the peak which can be calculate using Origin pro software. From this equation, it is found that the calculated average size of ZnO nanoparticle is 25 nm. The peaks of XRD pattern are obtained at 2θ = 31.6^0, 34.3^0, 36.1^0, 47.43^0, 56.52^0, 62.77^0, 67.9^0, 72.1^0, and 76.98^0 which indicate the obvious formation of ZnO nanoparticle. Similar peaks of ZnO nanoparticles can also be found in the investigations of Sing et al. [9].

Surface Morphology of ZnO Nano Coated Fabric

To compare the treated fabric surface with untreated fabric, the morphological changes are observed by SEM. Remarkable change is visible on the surface morphology of treated and untreated fabrics. The obtained SEM images are shown in Figs. 5 (a) and (b). The untreated fabric image in Fig. 5 (a) represents the smooth fibrillar structure of cotton fabric at ×1000 magnification. On the other hand, Fig. 5 (b) shows the presence of rough agglomerated and various sizes of nanoparticles on the fabric surface. Higher magnification (×1000) is capable to trace the fabric interior and the grove present on cotton surface. The surface morphology changes significantly due to nano deposition on cotton which is an indication of the cross linking of nanoparticles with the fibril structure of the fabric.

Antimicrobial Activity and Wash Durability

The antimicrobial activity of treated and untreated fabric is quantitatively evaluated by AATCC test method 100 [10]. It is examined against *Staphylococcus aureus* (gram positive) and *Escherichia coli* (gram negative). Each culture is suspended in a small amount of nutrient broth, spread on the nutrient agar plate, and incubated at 37 ºC for 24 hours. Colonies are picked off with an inoculating loop from the agar plate, suspended in a 5 mL nutrient broth, and incubated for 18 hours at 100 rpm and 37 ºC. A final concentration of $1.5\text{-}3.0\times10^8$ colony forming units per milliliter (CFU/mL) is prepared by appropriately diluting each culture with a sterile buffer solution (0.3 mM phosphate buffer, p^H 7.2), which is used as a diluent in all experiments. The dilute culture solution is used for the antimicrobial test. The obtained results are presented in Table I. The untreated fabric demonstrates zero bacterial reduction after one-hour contact time against gram positive and gram negative bacteria whereas, the treated fabric shows significant reduction of bacterial (R %): 90% for *S. aureus* and 87% for *E. coli*. The antibacterial activity after washing is examined to note the durability of nanoparticles on the fabric surface. 5, 10 and 15 washing cycles are completed for nano ZnO coated fabric (1% acrylic binder) in a washing machine using the method: AATCC 61-2009. After every washing, the bacterial reduction is checked for treated fabric. The result indicates small amount of bacterial reduction (R %) decreases after washing. The increasing of washing cycle causes the decreasing of bacterial reduction. However, the rate of reduction is not too high for five and 10 washing cycle. Thus, the wash durability of nanoparticles is remarkable for ZnO nano coated fabric.

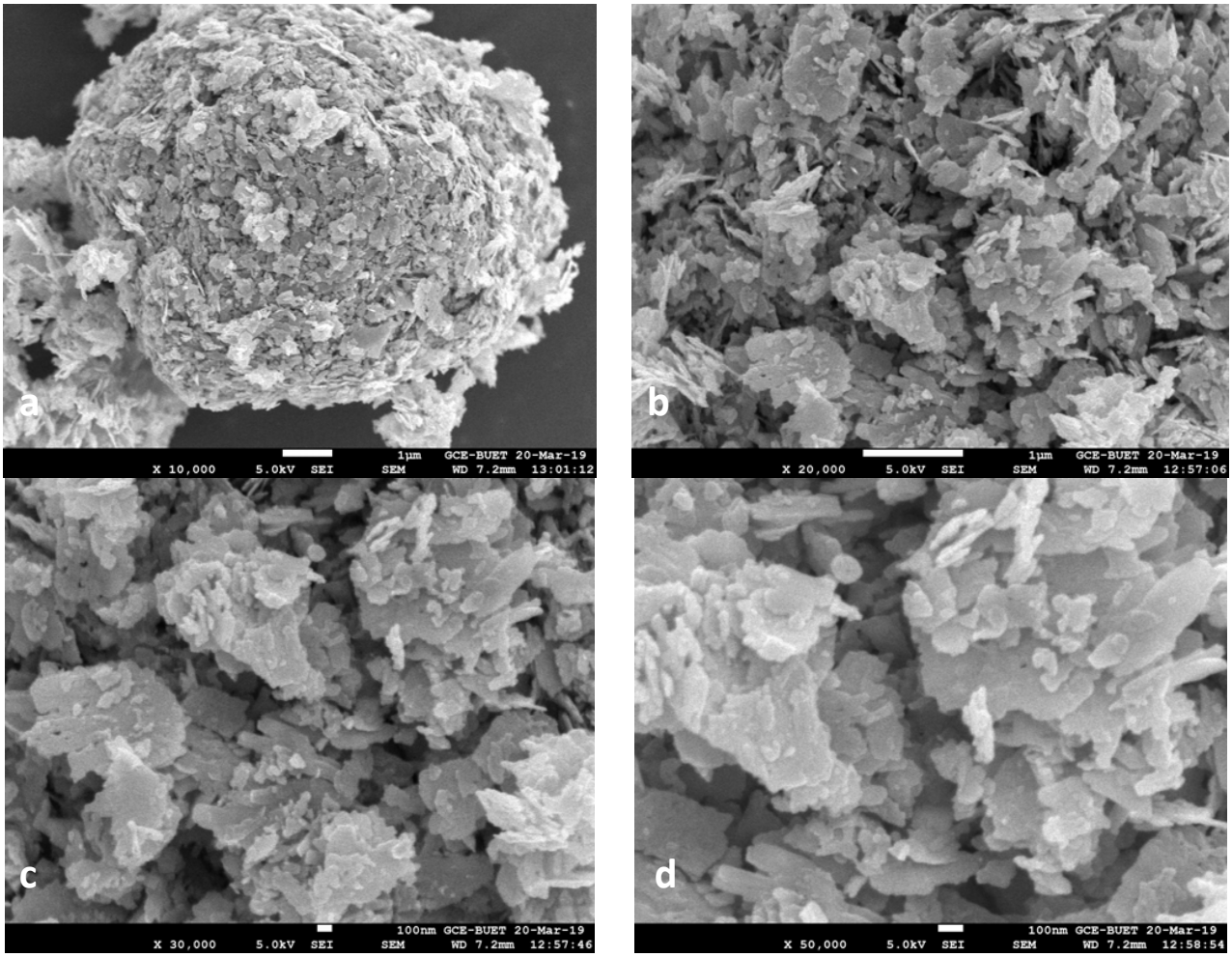

Fig. 3 SEM image of ZnO nanoparticle on different magnifications with (a) ×10000, (b) ×20000, (c) ×30000, (d) ×50000

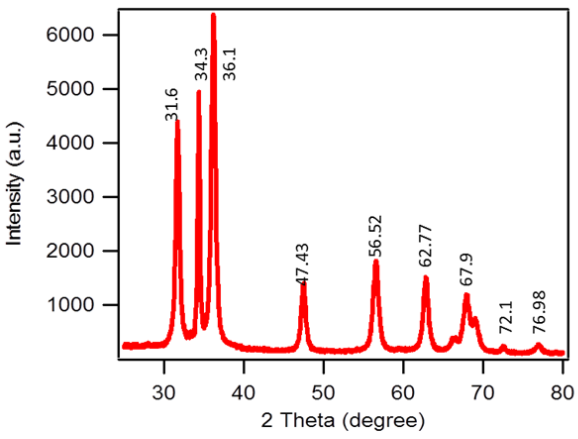

Fig. 4 XRD patterns of ZnO nanoparticles

V. Conclusions

ZnO nanoparticles are prepared and applied on cotton fabric by pad, dry and cure method. Nanoparticles in powder form and nano coated fabric are characterized by SEM. The approximate size range obtained from XRD pattern analysis is around 35 nm. Due to the nano ZnO coating, antimicrobial activity is imparted on cotton fabric which is an important biological property of cotton. So, the bacterial growth of cotton fiber decreases. Moreover, the treated fabric shows sufficient bacterial reduction after nanocoating. Treated fabric exhibits 90% bacterial reduction (R %) for *S. aureus* and 87% for *E. coli*. The wash durability of treated fabric is also satisfactory. The treated fabric retains remarkable bacterial reduction after 15 washes. Therefore, the ZnO nano coated fabric is useful as a medical textile for bacterial protective cloth.

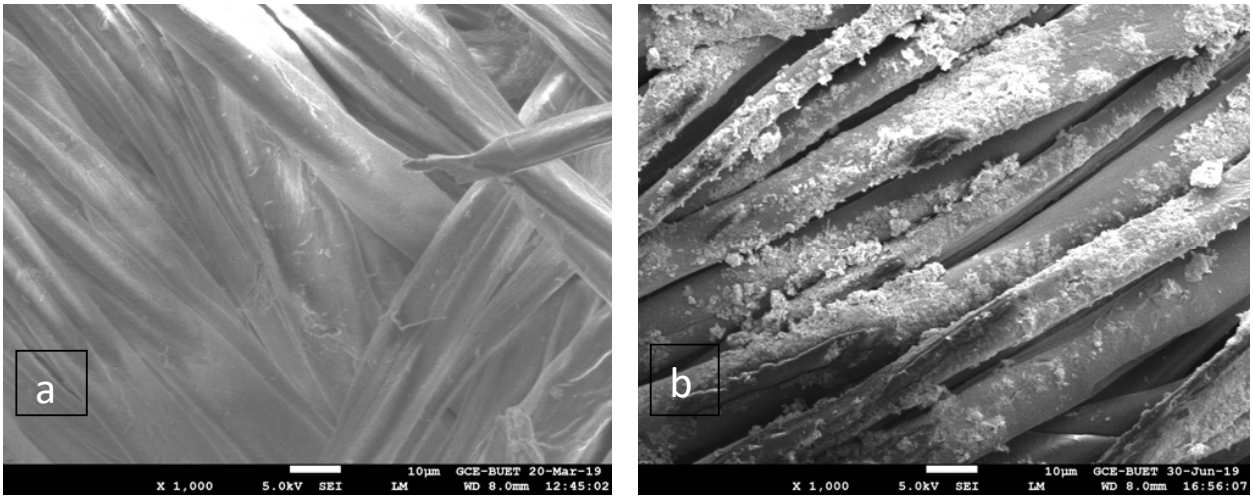

Fig. 5 Surface morphology of nano ZnO coated cotton fabric obtained by SEM in ×1000 magnification: (a) untreated and (b) treated fabric

TABLE I
BACTERIAL REDUCTION (R %) OF UNTREATED AND NANO ZNO COATED FABRIC

Sample	*Staphylococcus aureus* (Gram +Ve)			*Escherichia coli* (Gram –Ve)		
	Surviving Cells (CFU) after 0 Contact Time	Surviving Cells (CFU)after 1.0 hour Contact Time	Bacterial Reduction % (R%) after 1.0 hour	Surviving Cells (CFU)after 0 Contact Time	Surviving Cells (CFU)after 1.0 hour Contact Time	Bacterial Reduction % (R%) after 1.0 hour
Untreated	2.44×10^8	2.44×10^8	00	2.44×10^8	2.44×10^8	00
Nano coated						
Unwashed	2.44×10^8	22×10^6	90	2.44×10^8	35×10^6	85
5 wash	2.44×10^8	42×10^6	83	2.44×10^8	47×10^6	80
10 Wash	2.44×10^8	47×10^6	80	2.44×10^8	65×10^6	73
15 wash	2.44×10^8	71×10^6	70	2.44×10^8	90×10^6	63

ACKNOWLEDGMENTS

The authors are thankful to Mechanical Engineering as well as Glass and Ceramic department of BUET (Bangladesh University of Engineering and Technology) to provide the laboratory facilities to conduct the research. We would also like to acknowledge financial support under “NST fellowship” program of Ministry of Science and Technology, Bangladesh.

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