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Research Article

SYNTHESIS AND CHARACTERIZATION OF ZINC OXIDE NANOPARTILCE AND ITS APPLICATION ON FABRICS FOR MICROBE RESISTANT DEFENCE CLOTHING

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ABSTRACT

Health and hygiene are the primary requirements for human beings to live comfortably and work with maximum efficiency. To protect the mankind from pathogens and to avoid cross infection, a special finish like antimicrobial finish has become necessary particularly in defence, where the group of people live together. The defence clothing system has very high potential for cross infections because they are living in groups in various camps and in extreme climates. Bed linen, socks, woolen blankets and clothing used by them are the carriers of microbes and hence cross infections are possible. Nanotechnology is considered as the fourth industrial revolution through the long history of human civilization. This research work attempted the synthesis and characterization of Zinc oxide nano particles and their application on Woven and Knitted Defence clothing (100% cotton & 45/55% polyester/cotton) for antibacterial activity. The Zinc oxide (ZnO) nano particles were prepared with two reacting media namely water and 1, 2 Ethanediol (Ethylene glycol). These nanoparticles have an average size 21 and 10 nm respectively, which was confirmed by Transmission Electron Microscope (TEM). 1% of the nano ZnO were applied on both woven and knitted fabrics. The presence of nano particles on the fabric surface was confirmed by Scanning Electron Microscope (SEM). Untreated fabric showed no Bacterial reduction, Zinc oxide nano particles (synthesized with water medium) treated 100% cotton woven fabric shows 93% and 94% reduction, 45/55% polyester/cotton woven fabric shows 93% and 93% reduction, 100% cotton knitted fabric shows 90% and 91% and 45/55% polyester/cotton knitted 89% and 91 % against Staphylococcus aureus and Klebsiella Pneumoniae bacteria respectively. Zinc oxide nano particles (synthesized with 1,2 Ethanediol medium) treated 100% cotton woven fabric shows 97% and 98% reduction, 45/55% polyester/cotton woven fabric shows 98% and 99% reduction, 100% cotton knitted fabric shows 95% and 94% and 45/55% polyester/cotton knitted 97% and 94 % against Staphylococcus aureus and Klebsiella Pneumoniae bacteria respectively. Basic mechanical properties such as tensile strength, elongation, crease recovery and air permeability of nano coated fabrics did not change considerably than the untreated fabrics.

Keywords: Zinc oxide nano particle, Antibacterial activity, Zinc oxide synthesis, Nanotextiles

INTRODUCTION

All the leading textile industries are focusing on value added applications such as microbe resistance, electro magnetic protection and thermoregulatory fabrics. Materials and system for human protection will enhance the performance and well being of the human society. To protect defence personnel from microbe and cross infection, a special finish like antimicrobial finish has become a necessity on clothing, socks, blankets and bed linens used by them. Nanotechnology is an umbrella term covering a wide range of technologies concerned with structures and processes on the nanometer scale. Because of its potential to change fundamentally whole fields of technology, nanotechnology is regarded as a key technology which will not only influence technological development in the near future, but will also have economic, social and ecological implications.

Fanzhang Employed settlement experiment to estimate the dispersion of nano ZnO in aqueous solution, which was surface modified with different surfactants, through the deposition time. Suitable surfactant was selected to improve the dispersion effect. Antistatic finishing agent, which was compounded with nano ZnO, was applied to polyester fabric by pad-dry-cure process and the optimal processing conditions were obtained by orthogonal test. By the static test, the results showed that the antistatic property of the treated fabric was better[1].

Nano Tio_2 particles were prepared by sol-gel method using tetra butyl titanate as precursor and ethanol as solvent reported by Haixia Li et al[2]. When distributed evenly on polyester fabric with fine dispersity and stability, the finished fabrics demonstrated exceptional anti-ultraviolet performance with a phenomenal UPF ascendance reaching up to 50+ without influencing the breaking strength [2]. Becheri coated cotton and wool fabric using zinc oxide nano particles to impart UV blocking property and tested the coated sample for UPF[3].

Lu sun et al work described the effect of the fading on dyed polyester fabrics in artificial sunlight, when the Ultra Violet (UV) component of the radiation was blocked by coating the fabric with zinc oxide nano particles, dispersed in an acrylic polymer. Zinc oxide is photoactive and generates superoxide and hydroxyl radicals (Reactive Oxygen Species; ROS) when irradiated with UV in the presence of oxygen and water[4]. The results showed that different dye chromophores interact differently with ROS. Selection of dyes with anti oxidant properties or addition of other anti oxidants may reduce the adverse effects of ROS. UV blocking by preparing Zinc oxide nano particles using zinc nitrate and applied it on bleached cotton fabric study reported by Yadav et al[5].

Zinc oxide–soluble starch nano composites were synthesized using water as a solvent and soluble starch as a stabilizer and impregnated onto cotton fabrics to impart antibacterial and UV-protection functions[6]. Wong reviewed the use of nano technology in the textile industry which has increased rapidly due to its unique and valuable properties. The present status of nanotechnology use in textiles was reviewed, with an emphasis on improving various properties of textiles[7]. Polypropylene/silver nano composite fibres for permanent antibacterial activity prepared by Xin *et al*[8].

The observation of unidirectional plasmon propagation in metallic nanowires over distances >10 µm. Work of Dickson et al[9]. Through control of the incident excitation wavelength and rod composition, he demonstrated the selective coupling of photons into the plasmon mode of a 20 nm diameter nanowire. As expected from previous studies of plasmon excitation in nanoparticles and thin films, he observed a strong wavelength and material dependence of this phenomenon. This metal-dependent plasmon propagation was exploited to produce a wire through which plasmons propagate unidirectionally. Synthesized ZnO nano particles by addition of LiOH to an ethanolic zinc acetate solution. It was found that aging of particles was governed by temperature, the water content, and the presence of reaction products. Water and acetate induced considerably accelerated particle growth[10,11]. This work attempted the synthesis and characterization of Zinc oxide nano particles and application on Woven and Knitted fabric for application as microbe resistance.

MATERIALS AND METHODS

Two kinds of woven and Knitted fabrics were made and the technical specifications of the two fabrics are shown in Table 1.

Table 1: Specifications woven and knitted fabrics

	Woven		Knitted	
Specification	100% cotton	45/55% polyester/cotton	100%cotton	45/55% polyester/cotton
Structure	Plain weave	Plain weave	Pique	Pique
Width	49”	48”	-	-
GSM	130	130	130	130
Ends/inch	98	92	-	-
Picks/inch	72	78	-	-
Warp count	$1/40^s$	$1/40^s$	34^s	34^s
Weft count	$1/40^s$	$1/40^s$	34^s	34^s

Method

Zinc oxide nano particle synthesis was done in two different media as follows:

- Zinc oxide nano particle synthesis in Water medium
- Zinc oxide nano particle synthesis in 1,2 Ethanediol (Ethylene glycol) medium

Zinc oxide nano particle synthesis in water medium

5.5gms of the zinc chloride was dissolved in 100ml of distilled water in a beaker. This solution was kept under constant magnetic stirring till zinc chloride totally dissolved in the distilled water. The temperature of the beaker was raised to 90°C by electric hot plate heating. Meanwhile 20gms of sodium hydroxide was dissolved in 100ml of distilled water in a separate vessel. From the prepared sodium hydroxide solution, 16 ml of sodium hydroxide is added to the beaker with constant stirring, drop by drop touching the walls of the beaker. The aqueous solution turned into a milky white colloid without any precipitation. The reaction was allowed to proceed for 2 hrs after complete addition of sodium hydroxide. After the complete reaction, the solution was allowed to settle and the supernatant solution was removed by washing with distilled water for 5 times. After complete washing, the zinc nano was dried at 100°C for 30 min and then it changed into powder form.

Zinc oxide nano particle synthesis in 1, 2 Ethanediol medium

5.5gms of the zinc chloride was dissolved in 200ml of 1, 2 Ethanediol in a beaker. This solution was kept under constant magnetic stirring till zinc chloride totally dissolved in 1, 2 Ethanediol. The temperature of the beaker was raised to 150°C by electric hot plate heating. Meanwhile 20gms of sodium hydroxide was dissolved in 100ml of distilled water in a separate vessel. From the prepared sodium hydroxide solution, 16 ml of sodium hydroxide is added to the beaker under constant stirring, drop by drop touching the walls of the beaker. The aqueous solution turned into a white colloid without any precipitation. The reaction was allowed to proceed for 30 minutes after complete addition of sodium hydroxide. After the complete reaction, the solution was allowed to settle and the supernatant solution was removed by washing with distilled water for 5 times. After complete washing, the zinc oxide nano particles were dried at 160˚C for 20 minutes and then it changed into powder form.

Characterization of Nano particles

Characterization of the nano particles was done by three tests such as X-ray Diffraction Method (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) and transmission electron microscope. The crystallinity was determined by XRD using a Bruker D8 Advance X rays Diffractometer equipped with a Cu Kα (λ = 1.54 A °) source (applied voltage 40 kV, current 40 mA). About 0.5 g of the dried particles were deposited as a randomly oriented powder onto a plexiglass sample container, and the XRD patterns were recorded at angles between 20° and 80°, with a scan rate of 1.5˚/min.

The crystallite domain diameters (D) were obtained from XRD peaks according to the Scherrer’s equation:

$$D = \frac{0.89\,\lambda}{\Delta W \cos\theta}$$

Where λ is the wavelength of the incident X-ray beam (1.54 Å for the Cu Kα), Ө is the Bragg’s diffraction angle, ΔW the width of the X-ray pattern line at half peak-height in radians. The chemical composition of the synthesized materials was checked by FTIR spectroscopy with a Biorad FTS-40 spectrometer.

The shape and size of the nano particles were obtained through TEM, using a Philips EM201C apparatus operating at 80kv. The samples for TEM measurements were placed on carbon-coated copper grids. The samples for TEM measurements were prepared from much diluted dispersions of the particles in 2-propanol. Surface area measurements were determined from BET on a Coulter SA 3100 surface area analyzer, under N2 flow.

Finishing Process

The woven and knitted fabric of 100% cotton and 45/55% polyester/cotton were applied with zinc oxide nano particles by Spraying using spray gun and also Pad-Dry-Cure method.

Procedure

Nano particle were applied on the face side of the fabric with concentration 1%, Material to liquor ratio1:20, Acrylic binder 1%. The 100% Cotton and 45/55% polyester/ cotton woven and knitted fabric were cut to the size of 30 x 30 cm. These fabrics were coated with zinc oxide nano particles by using a spray gun. A dispersion of nano particle was filled in the hand spray gun. The fabric substrate was fixed on a vertical board. The nano particle solution was evenly sprayed over the fabric by maintaining a constant distance between the fabric and spray gun nozzle. The excess solution was squeezed using a padding mangle which was running at a speed of 15 m/min with a pressure of 15 kg/cm^2 after padding the fabric was dried naturally and then cured for 3 minutes at 150˚C.

Characterization of Nano Finished Fabric

- Scanning Electron Microscope

The nano finished samples were mounted on a specimen stub with double-sided adhesive tape and coated with gold in a sputter coater and examined with a Scanning Electron Microscope (SEM) Jeol Model JSM-6360.

Functional Testing Of Finished Fabric Samples

To investigate the antibacterial activity of woven and knitted fabrics impregnation was done with zinc oxide nano particles separately. Antibacterial test AATCC 100-2004 was carried out against Staphylococcus aureus (Gram positive organism) and Klebsiella Pneumoniae (Gram negative organism).

The percentage reduction of bacteria by the 100%cotton and 45/55% polyester/cotton fabrics is reported as R,

$$R = 100(B - A)/B.$$

Where R = % reduction

A = the number of bacteria recovered from the inoculated treated test specimen swatches in the jar incubated over 24 hours

B = the number of bacteria recovered from the inoculated treated test specimen swatches in the jar immediately after inoculation (at ‘0’ contact time)

RESULTS AND DISCUSSIONS

Characterization of Nano Particles Using XRD, FTIR And TEM

The results shown in figure 1, 2, 3, 4, 5 & 6 indicate that the experimental conditions greatly affect the morphology and size of the particles, prepared with the different condition. In fact, increasing the reaction temperature, results in a significant lowering of the nano particles size and their agglomeration number D, calculated as

$$D = \frac{0.89\ \lambda}{\Delta W \cos \theta}$$

The FTIR spectrum of the material obtained from synthesis via water medium shows the Zn –O absorption band at 444.95 cm^{-1}. The peaks at 1637.50 cm^{-1} and 3438.20 cm^{-1} indicate the presence of –OH and C=O residues, probably due to atmospheric moisture and Co_2 respectively. The same spectrum was obtained via synthesis in 1, 2 Ethanediol.TEM size distributions were obtained for nano particles, as shown in Figure 5 & 6. The mean crystallite size is presented in Table 2.

Analysis of Zinc oxide finished fabrics using Scanning Electron Microscope (SEM)

Figure 7 & 8 shows the SEM Micrographs of untreated 45/55% polyester/cotton and treated fabric respectively. The nano particles were well dispersed on the fibre surface in both cases, although some aggregated nano particles are still visible. The particle size plays a primary role in determining their adhesion to the fibres. It is reasonable to expect that the largest particle agglomerates will be easily removed from the fiber surface, while the smaller particles will penetrate deeper and adhere strongly into the fabric matrix.

Table 2: Diameter of nano particle

Metal oxide Nano particle	Media	Temperature °C	Average Diameter (nm)
Zinc oxide	Water	90	21
Zinc Oxide	1,2 Ethanediol	150	10

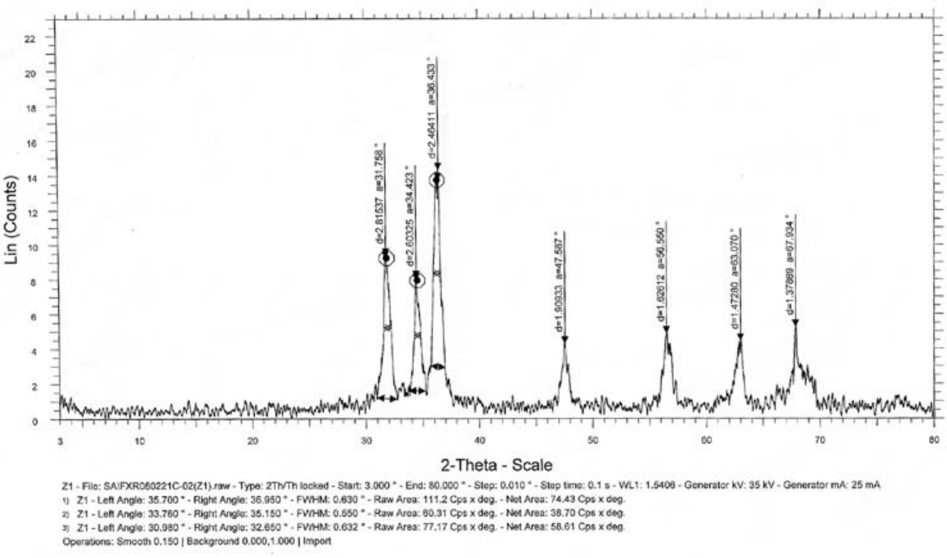

Fig. 1: XRD of Zinc oxide nano particle (Water medium)

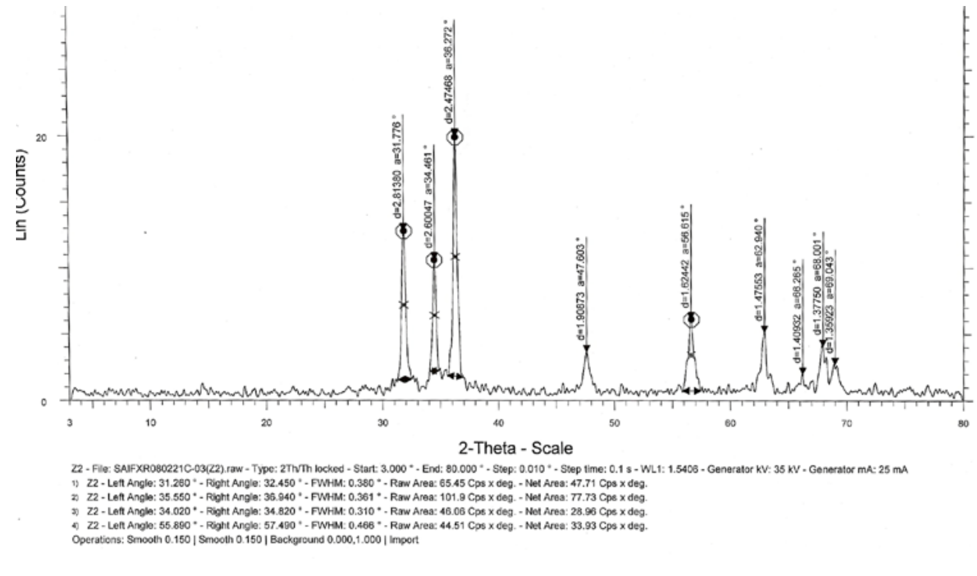

Fig. 2: XRD of Zinc oxide nanoparticle (1, 2 Ethanediol medium)

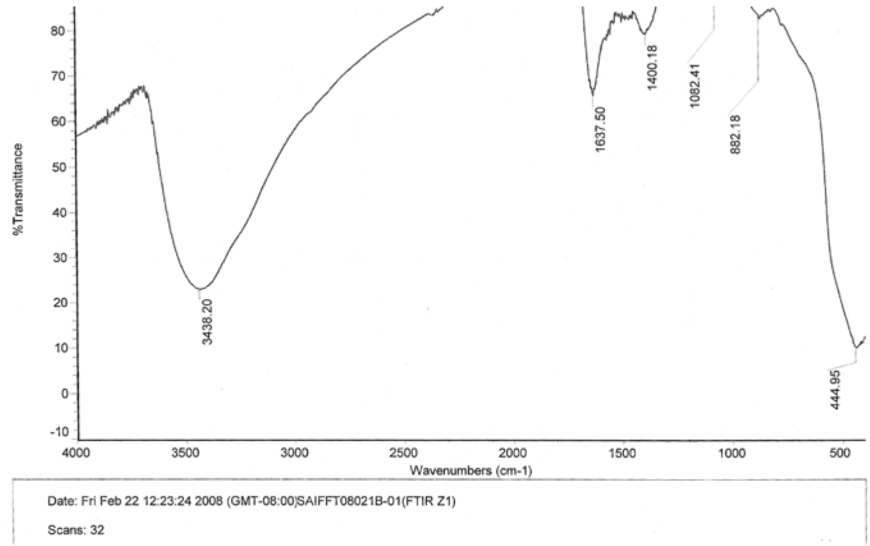

Fig. 3: FTIR of Zinc oxide nano particle (Water medium)

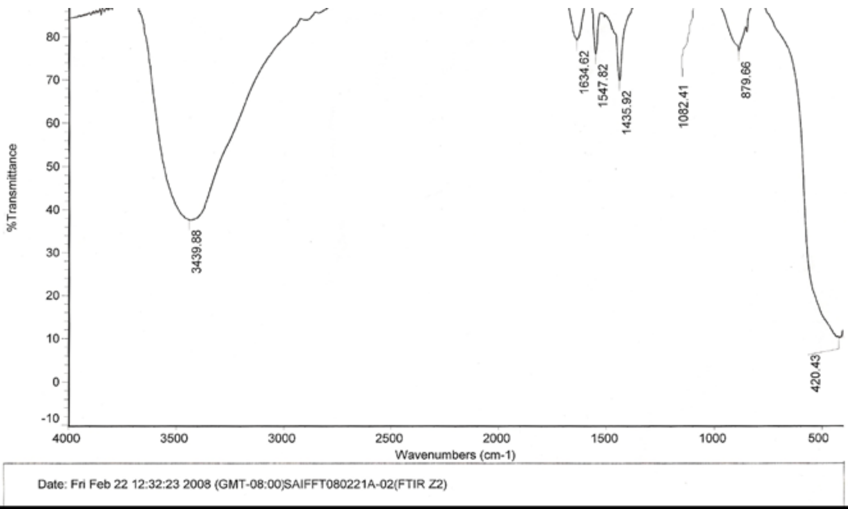

Fig. 4: FTIR of Zinc oxide nano particle (1, 2 Ethanediol medium)

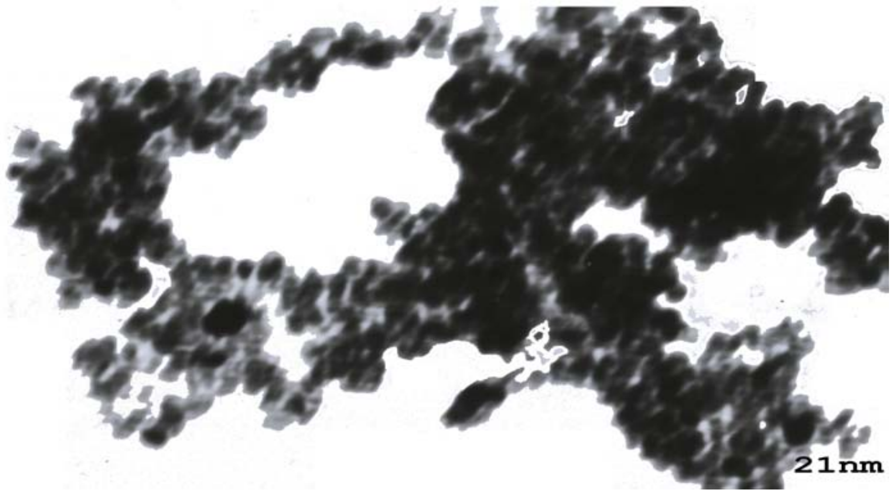

Fig. 5: TEM of Zinc oxide nanoparticle (water Medium)

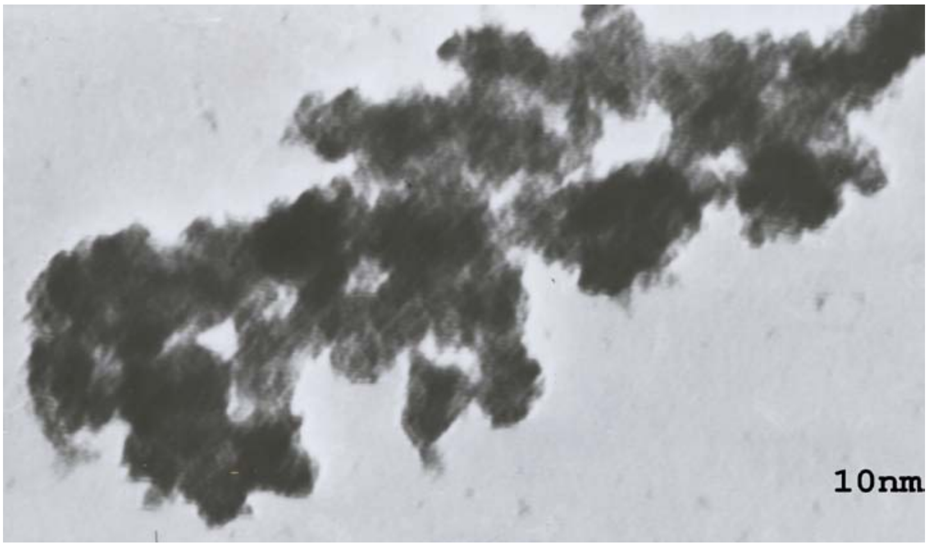

Fig. 6: TEM of Zinc oxide nanoparticle (1, 2 Ethanediol Medium)

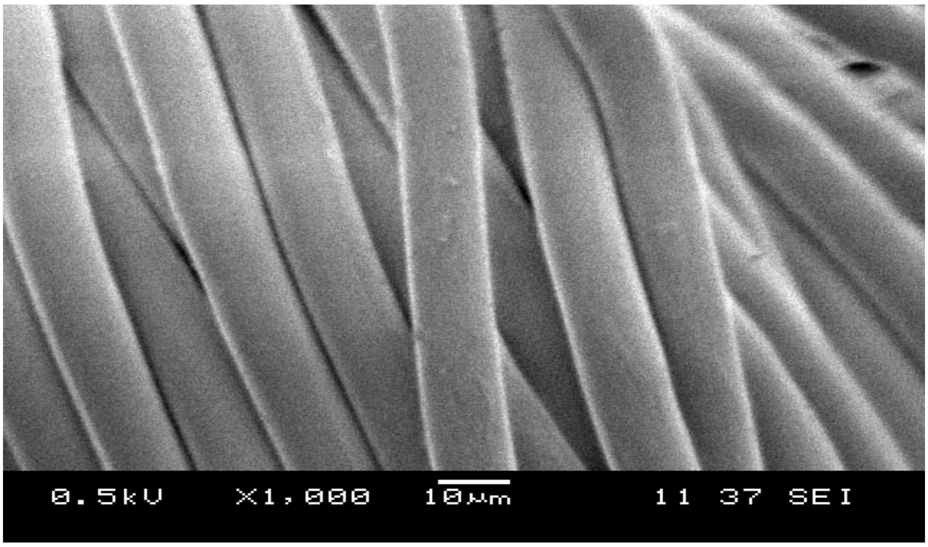

Fig. 7: SEM image of the Untreated 45/55% Polyester/Cotton woven

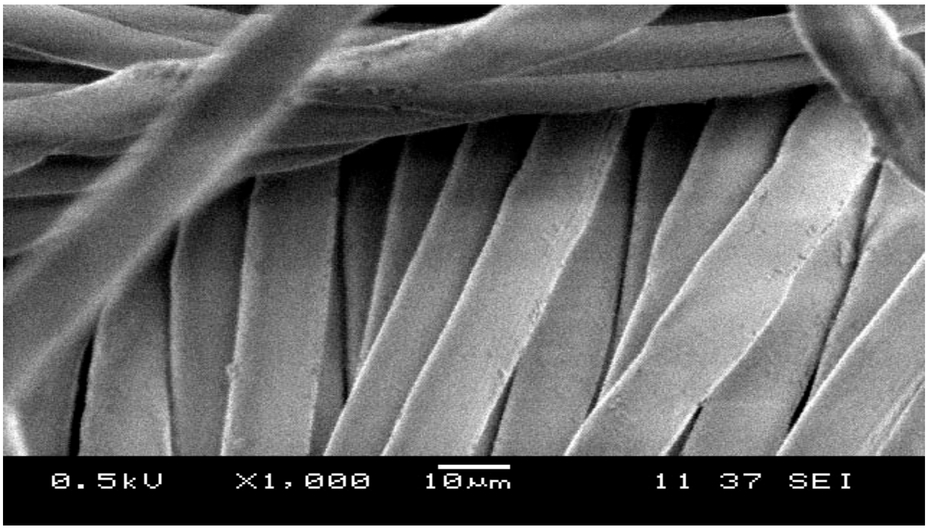

Fig. 8: SEM image of the treated 45/55% Polyester/Cotton woven

Evaluation of Zinc oxide nano particle coated fabrics for Antibacterial activity

Fabrics are an excellent medium for the growth of micro organisms when the basic requirements such as nutrients, moisture, oxygen and appropriate temperature are present. Natural fibers like cotton are more susceptible to microbial attack than synthetic fibers. Hence, cotton and polyester/cotton coated with nano zinc oxide were evaluated for antibacterial activity.

Table 3: Antibacterial activity of zinc oxide nano particle coated fabrics

Metal oxide Nano particles	Fabric samples	Staphylococcus aureus	Klebsiella Pneumoniae
Zinc oxide (Water medium)	Untreated	No reduction	No reduction
	Woven 100% cotton	93%	94%
	Woven 45/55%Polyester/Cotton	93%	93%
	Knitted 100% Cotton	90%	91%
	Knitted 45/55%Polyester/Cotton	89%	91%
Zinc oxide (1,2 Ethanediol medium)	Untreated	No reduction	No reduction
	Woven 100% cotton	97%	98%
	Woven 45/55%Polyester/Cotton	98%	99%
	Knitted 100% Cotton	95%	94%
	Knitted 45/55%Polyester/Cotton	97%	94%

Fabrics treated with Zinc oxide (1, 2 Ethanediol medium) shows antibacterial property to a much higher extent than water medium. The woven fabrics show much higher resistance when compared to the knitted fabrics treated with 1, 2 Ethanediol. Among these two media Fabrics treated with Zinc oxide (1, 2 Ethanediol medium) show better microbe resistance than 1.0 % Zinc oxide (water medium).

Woven fabrics treated with zinc oxide exhibit better antibacterial resistance than knitted fabrics. Among the composition 45/55% polyester/cotton blend shows better reduction than the 100% cotton against the microbes of Staphytococcus aureus and Klebsiella Pneumoniae.

Evaluation of Zinc oxide coated fabric – basic mechanical properties

The effect of coating on fabric, tensile strength, elongation, Crease recovery angle and air permeability were analyzed and is shown in Table 4.

Mechanical Properties of Untreated and Nano Treated Fabrics

Table 4: Mechanical Properties of Untreated and Nano Treated Fabrics

Samples		Fabric Tensile strength (Kg)			Elongation %		Crease recovery angle		Air permeability $Cm^3/Cm^2/Sec$	
		Untreated	Treated		Untreated	Treated	Untreated	Treated	Untreated	Treated
Woven 100% Cotton	Warp	54	53.5 42.2	42	9	8.7	129	129	36.29	37.41
	Weft				16.4	17	130	130		
Woven 45/55% Polyester/Cotton	Warp	33.2	33		8.6	8.6	122	122	36.21	36.68
	Weft	30.2	31		27.4	27.5	120	120		
Knitted 100% Cotton	Lengthwise	16.7	16.5		28.1	27.9	-	-	65	64.9
	Widthwise	15.2	15.3		45.1	44.6	-	-		
Knitted 45/55% Polyester/Cotton	Lengthwise	14.5	14.7		20.4	20.8	-	-	142.2	143
	Widthwise	11.7	12		27.5	26.9	-	-		

CONCLUSION

ZnO treated woven and knitted fabric showed excellent antibacterial activity against two representative bacteria, Staphylococcus aureus and Klebsiella pneumoniae. Hence it is most suitable for defence clothing like bed linens, gloves and T-shirts to avoid cross infections. This work provides a simple method for aqueous preparation of ZnO nano composites and their application onto 100% cotton & 45/55% polyester/cotton fabrics to impart antibacterial property and the reduction % is around 97% against Staphylococcus aureus and 98% for Klebsiella pneumonia to control cross infections for defence personnel in extreme climates.

REFERENCES

1. Fan Zhang, Junling Yang: Preparation of nano-ZnO and its application to the Textile on antistatic finishing, International Journal of Chemistry 1: (2009) 18-22.
2. Haixia Li, Hua Deng, Jing Zhao: Polyester Fabric Treated by nano titanium dioxide Anti-Ultraviolet finishing, International Journal of Chemistry 1: (2009) 57-60.
3. Becheri, A, Durr, M, Lonostro P, Bagloini P J: Synthesis and characterization of zinc oxide nanoparticles: application to textiles as UV-absorbers, J.Nanoparticle Research 2(10): (2008) 679-689.
4. Lu Sun, John A, Peter G, Xungai W, Ken King: Nano zinc oxide for UV protection of Textiles, International Journal of Technology 7: *(*2008) 224-235.
5. Yadav A, Virendra Prasad, Kathe AA: Functional Finishing of cotton fabrics using Zinc oxide nanoparticles, Material Science 29: (2006) 641-645.
6. Kumar S, Kathe AA., Varadarajan PV, Prasad: Functional Finishing of Cotton Fabrics using Zinc Oxidesoluble Starch Nanocomposites, Nanotechnology (2006) 5087-5094.
7. Wong YWH, Yuen CWM, Leung MYS, Ku SKA, Lam HLI: Selected Applications of nanotechnology, Autex Research Journal 6: (2006) 1-10.
8. Xin JH, Yeo SY, Lee HJ, Jeong SH: Preparation of nano compsite fibres for permanent antibacterial effect, Journal of Material. Science 38: (2003) 2143-2147.

9. Dickson M.R., Lyon AL: Unidirectional Plasmon Propagation in Metallic Nanowires, Journal of Physical Chemistry 104: (2000) 6095-6098.
10. Eric, A: Synthesis and Growth of ZnO Nanoparticles, Journal of Chemistry (1998)
11. 5566-5572.
12. Sindhuramachandran CV, Sreekumar PK: Synthesis, characterization and antibacterial evaluation of 2(5h)-furan one derivatives from highly functionalized mucobromic acid,
13. International Journal of Pharmacy and Pharmaceutical Sciences 3(1): (2011) 225-228.