PREPARATION AND CHARACTERIZATION OF NANO-CHEMICAL IN MEDICAL TEXTILES

A PROJECT REPORT

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BONAFIDE CERTIFICATE

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ABSTRACT

The cotton fabrics with Nano particles are sterile and can be useful to prevent or to minimize infection with pathogenic bacteria's such as Staphylococcus Aureus and Escherichia coli.

The major area of the work was focused on synthesis, characterization and application of ZnO2 nanoparticles on to samples of the textile substrates followed by the functional testing of the treated fabrics samples for functional properties antimicrobial activity. The synthesized nanoparticles were then characterized using typical methods of powder X-ray Diffractometry (XRD) and Scanning Electron Microscopy (SEM). These nanoparticles were then applied on to the sample fabrics using Pad-Dry-Cure method.

The treated fabric samples were also characterized using SEM and found to have nanoparticles on to the fabric surfaces. Then the samples were tested for the functions of Anti-microbial activity as per AATCC's standardized tests. The results of these tests showed that the treated fabric samples were added with desired functions to considerable extent when compared with untreated samples.

It has been found that the nanoparticles is most important factor determining the efficacy of the finish. The results of these studies and tests have been analyzed, discussed and reported with conclusions.

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LIST OF SYMBOLS AND ABBREVATIONS

Symbols

D - Crystalline diameter

N_e - English count

▲W - Full width of the X-ray pattern line at half peak

® - Registered trade name

X - Variable

λ - Wave length

Abbreviations

AATCC - American Association of Textile Chemists and Colourist

ASTM - American Standards of Testing Materials

Cm - Centimeter

D - Crystalline diameter

kPa - Kilo Pascal

nm - Nanometre

SA - Staphylococcus Aures

SEM - Scanning Electron Microscopy

XRD - X-ray Diffractory

ZnO - Zinc oxide

CHAPTER 1

INTRODUCTION

Wound healing is a complex process and has been the subject of intense research for a long time.

A survey of the literature showed the scarcity of published research work on the use and study of metal oxide nanoarticles treatments for the wound healing purpose. The process of textile finishing is always done with an aim to make the textile materials more suitable for their end uses. Finishing is the final step in the fabric manufacturing process and therefore, is the last chance to provide the properties that customers will value. Chemical finishing has always been important component of the textile processing, but in recent years the trend to 'high tech' products has increased the interest and use of chemical finishes.

The main purposes of this finishing process are:

- Develop the "product finishing" in all its fundamental elements.
- Give the finished fabric some properties that grant optimum behaviors through its makeup.

The work described herein, is the detailed investigation of the Anti-microbial activity of woven fabrics made of 100% cotton incorporated with ZnO nanoparticles. The fabric samples were bleached under the standard conditions.

Stable ZnO nano particles were prepared by soft chemistry. The characterization results of the nano ZnO particles were studied by the typical methods of X-ray Diffractometry and Scanning Electron Microscope. The nanoparticles were spherical in shape and mono disperse. These nanoparticles were applied on the fabric samples using Pad-Dry-Cure method. Then the characterization of the finished fabric is done and the Anti-microbial activity is tested in standard conditions.

CHAPTER 2

LITERATURE REVIEW

2.1 NANO PARTICLES

"A particle having one or more dimensions of the order of 100nm or less".

There is a note associated with this definition: "Novel properties that differentiate nanoparticles from the bulk material typically develop at a critical length scale of under 100nm".

The "novel properties" mentioned are entirely dependent on the fact that at the nano-scale, the physics of nanoparticles mean that their properties are different from the properties of the bulk material.

In nanotechnology, a particle is defined as a small object that behaves as a whole unit in terms of its transport and properties. Particles are further classified according to size: in terms of diameter, fine particles cover a range between 100 and 2500 nanometers. On the other hand, ultrafine particles are sized between 1 and 100 nanometers. Similar to ultrafine particles, nanoparticles are sized between 1 and 100 nanometers. Nanoparticles may or may not exhibit size-related properties that differ significantly from those observed in fine particles or bulk materials.[1][2] Although the size of most molecules would fit into the above outline, individual molecules are usually not referred to as nanoparticles.

Nanoclusters have at least one dimension between 1 and 10 nanometers and a narrow size distribution. Nanopowders are agglomerates of ultrafine particles, nanoparticles, or nanoclusters. Nanometer-sized single crystals, or single-domain ultrafine particles, are often referred to as nanocrystals.

Nanoparticle research is currently an area of intense scientific interest due to a wide variety of potential applications in biomedical, optical and electronic fields.

There is no strict dividing line between nanoparticles and non-nanoparticles. The size at which materials display different properties to the bulk material is material dependant and can certainly be claimed for many materials much larger in size than 100nm.

The sort of nanomaterial has led to the extension of the idea of nanomaterials being considered as such if any one of their structural features are on a scale of less than 100nm, that cause their properties to be different from that of the bulk material.

TABLE 1 NANO PARTICLES CATEGORIES AND APPLICATON

NANO STRUCTURE`	APPLICATION
Nano tubes	Carbon (fullerenes)
Nano wires	Semi conductors, metals. Oxides
Nano crystals	Insulators, magnetic materials
Other nano particles	Ceramic oxides

2.2 NANO PARTICLE IN MEDICAL TEXTILES AND WOUND DRESSING

Nanomaterials are at the leading edge of the rapidly developing field of nanotechnology. Their unique size-dependent properties make these materials superior and indispensable in many areas of human activity. This brief review tries to summarise the most recent developments in the field of applied nanomaterials, in particular their application in biology and medicine, and discusses their commercialisation prospects.

Nanotechnology is enabling technology that deals with nano-meter sized objects. It is expected that nanotechnology will be developed at several levels: materials, devices and systems. The nanomaterials level is the most advanced at present, both in scientific knowledge and in commercial applications. A decade ago, nanoparticles were studied because of their size-dependent physical and chemical properties . Now they have entered a commercial exploration period .

Living organisms are built of cells that are typically 10 μm across. However, the cell parts are much smaller and are in the sub-micron size domain. Even smaller are the proteins with a typical size of just 5 nm, which is comparable with the dimensions of smallest manmade nanoparticles. This simple size comparison gives an idea of using nanoparticles as very small probes that would allow us to spy at the cellular machinery without introducing too much interference.

nderstanding of biological processes on the nanoscale level is a strong driving force behind evelopment of nanotechnology .

acterial infection from medical devices is a major problem and accounts for an increasing umber of deaths as well as high medical costs. Many different strategies have been developed to ecrease the incidence of medical device related infection. One way to prevent infection is by nodifying the surface of the devices in such a way that no bacterial adhesion can occur. This equires modification of the complete surface with, mostly, hydrophilic polymeric surface oatings. These materials are designed to be non-fouling, meaning that protein adsorption and ubsequent microbial adhesion are minimized. Incorporation of antimicrobial agents in the bulk naterial or as a surface coating has been considered a viable alternative for systemic application of antibiotics. However, the manifestation of more and more multi-drug resistant bacterial strains estrains the use of antibiotics in a preventive strategy. The application of silver nanoparticles on he surface of medical devices has been used to prevent bacterial adhesion and subsequent biofilm formation. The nanoparticles are either deposited directly on the device surface, or applied in a polymeric surface coating. The silver is slowly released from the surface, thereby killing the bacteria present near the surface. In the last decade there has been a surplus of studies applying the concept of silver nanoparticles as an antimicrobial agent on a range of different medical devices. The main problem however is that the exact antimicrobial mechanism of silver remains unclear. Additionally, the antimicrobial efficacy of silver on Polymers 2011, 3 341 medical devices varies to a great extent. We will review existing antimicrobial coating strategies and discuss the use of silver or silver nanoparticles on surfaces that are designed to prevent medical device related infections.

The aim of this review is firstly to give reader a historic prospective of nanomaterial application to biology and medicine, secondly to try to overview the most recent developments in this field, and finally to discuss the hard road to commercialisation. Hybrid bio nanomaterials can also be applied to build novel electronic, optoelectronics and memory devices.

Zinc oxide as a mixture with about 0.5% iron(III) oxide (Fe_2O_3) is called calamine and is used in calamine lotion. There are also two minerals, zincite and hemimorphite, which have been historically called calamine. When mixed with eugenol, a chelate, zinc oxide eugenol is formed which has restorative and prosthodontic applications in dentistry.

Reflecting the basic properties of ZnO, fine particles of the oxide have deodorizing and antibacterial action and for that reason are added into various materials including cotton fabric, rubber, food packaging, etc. Enhanced antibacterial action of fine particles compared to bulk material is not intrinsic to ZnO and is observed for other materials, such as silver.

Zinc oxide is widely used to treat a variety of other skin conditions, in products such as baby powder and barrier creams to treat diaper rashes, calamine cream, anti-dandruff shampoos, and antiseptic ointments.It is also a component in tape (called "zinc oxide tape") used by athletes as a bandage to prevent soft tissue damage during workouts.

When used as an ingredient in sunscreen, zinc oxide sits on the skin's surface i.e. is not absorbed into the skin, and blocks both UVA (320-400 nm) and UVB (280-320 nm) rays of ultraviolet light. Because zinc oxide (and the other most common physical sunscreen, titanium dioxide) are not absorbed into the skin, they are nonirritating and nonallergenic.

However, many sunscreens use nano zinc oxide (along with nano titanium dioxide) which does get absorbed into the skin. This could cause as of yet unknown health problems and requires further study.

Zinc oxide can be used in ointments, creams, and lotions to protect against sunburn and other damage to the skin caused by ultraviolet light (see sunscreen). It is the broadest spectrum UVA and UVB reflector that is approved for use as a sunscreen by the FDA, and is completely photostable.

The significance of the nanoparticles can be summed up by their unique effects at quantum and surface levels. The quantum effects of the nanoparticles are due to the restriction of the space available to the electrons at the lower end of nanoscale. Thus, the properties of the nanoparticles are different from the bulk properties especially in electronic, optical and magnetic behavior. The surface effects of the nanoparticles are due to the higher ratio of surface area to volume.

Thus nanotechnology provides the ability to work on a nanoscale to create suitable molecular structures that have fundamentally different performances enhancing characteristics. Therefore, working on a nanoscale can allow us to build molecular architectures that can be specifically designed to create the desirable attributes in textiles (Kwon et al 2002).

2.2.1 Bioactive electrospun silver nanoparticles-containing polyurethane nanofibers as wound dressings:

Nanofibrous membrane (NFM) intended as wound dressing was prepared by electrospinning polyurethane (PU) solution containing silver ion, followed by reduction of silver ion to silver nanoparticles. The electrospun PU membrane has high surface area-to-volume ratio, controlled evaporative water transmission rate, good fluid drainage ability, and excellent antimicrobial activity. With an aim to promote wound healing, collagen was grafted to fiber surface by low temperature oxygen plasma treatment, which could improve surface hydrophilicity and facilitate covalent binding of collagen molecules to the plasma-treated PU surface. A NFM with no bead formation was obtained with fiber diameters around 159 nm. The presence of embedded silver nanoparticles and surface-grafted collagen was confirmed qualitatively and quantitatively. After modification, the NFM's antimicrobial activity improved to approximately 100% inhibition of bacterial growth with concomitant increase of membrane water absorption ability, which facilitates its use as a functional wound dressing. From animal studies, the NFM was better than gauze and commercial collagen sponge wound dressing in wound healing rate.

2.3 Escherichia coli

FIGURE 1 ESCHERICHIA COLI GENERE

Escherichia coli

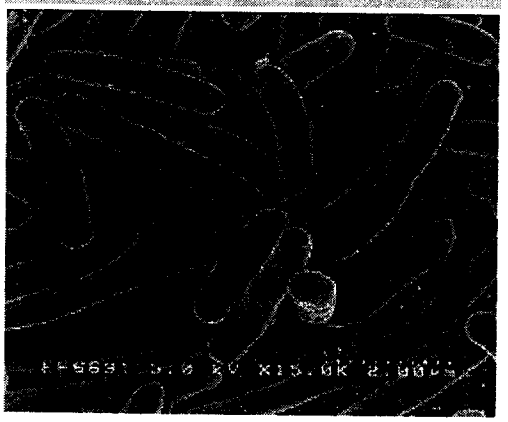


TABLE 2 ESCHERICHIA COLI

Scientific classification	
Domain:	Bacteria
Phylum:	Proteobacteria
Class:	Gammaproteobacteria
Order:	Enterobacteriales
Family:	Enterobacteriaceae
Genus:	*Escherichia*
Species:	E. coli

Escherichia coli (commonly abbreviated **E. coli**; named after Theodor Escherich) is a Gram negativerod-shapedbacterium that is commonly found in the lower intestine of warm-blooded organisms (endotherms). Most E. coli strains are harmless, but some, such as serotypeO157:H7, can cause serious food poisoning in humans, and are occasionally responsible for product recalls. The harmless strains are part of the normal flora of the gut, and can benefit their hosts by producing vitamin K_2, and by preventing the establishment of pathogenic bacteria within the intestine.

E. coli are not always confined to the intestine, and their ability to survive for brief periods outside the body makes them an ideal indicator organism to test environmental samples for fecal contamination. The bacteria can also be grown easily and its genetics are comparatively simple and easily manipulated or duplicated through a process of metagenics, making it one of the best-studied prokaryotic model organisms, and an important species in biotechnology and microbiology.

E. coli was discovered by German paediatrician and bacteriologist Theodor Escherich in 1885 and is now classified as part of the Enterobacteriaceae family of gamma-proteobacteria.

Role as normal microbiota

E. coli normally colonizes an infant's gastrointestinal tract within 40 hours of birth, arriving with food or water or with the individuals handling the child. In the bowel, it adheres to the mucus of the large intestine. It is the primary facultative anaerobe of the human gastrointestinal tract. (Facultative anaerobes are organisms that can grow in either the presence or absence of oxygen.) As long as these bacteria do not acquire genetic elements encoding for virulence factors, they remain benign commensals.

2.4 Staphylococcus Aureus

FIGURE 2 STAPHYLOCOCCUS AURES GENERE

Scanning electron micrograph of S. aureus, 20,000x, false color added.

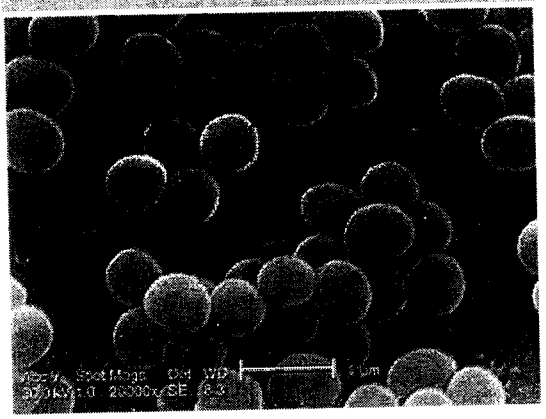


TABLE 3 Staphylococcus Aures

Scientific classification	
Domain:	Bacteria
Kingdom:	Eubacteria
Phylum:	Firmicutes
Class:	Bacilli
Order:	Bacillales
Family:	Staphylococcaceae
Genus:	Staphylococcus
Species:	**S. aureus**

Yellow colonies of S. aureus on a blood agar plate. Note regions of clearing around colonies, caused by lysis of red cells in the agar (beta hemolysis)

Staphylococcus aureus, literally the "golden cluster seed" or "the seed gold" and also known as golden staph and Oro staphira) is a facultatively anaerobic, Gram-positivecoccus and is the most common cause of staph infections. It is frequently part of the skin flora found in the nose and on skin. About 20% of the human population are long-term carriers of S. aureus.[The carotenoidpigmentstaphyloxanthin is responsible for S. aureus' characteristic golden colour, which may be seen in colonies of the organism. This pigment acts as a virulence factor with an antioxidant action that helps the microbe evade death by reactive oxygen species used by the host immune system. Staph organisms which lack the pigment are more easily killed by host defenses.

S. aureus can cause a range of illnesses from minor skin infections, such as pimples, impetigo, boils (furuncles), cellulitis folliculitis, carbuncles, scalded skin syndrome, and abscesses, to life-threatening diseases such as pneumonia, meningitis, osteomyelitis, endocarditis, toxic shock syndrome (TSS), chest pain, bacteremia, and sepsis. Its incidence is from skin, soft tissue, respiratory, bone, joint, endovascular to wound infections. It is still one of the five most common causes of nosocomial infections, often causing postsurgical wound infections. Abbreviated to S. aureus or Staph aureus in medical literature, S. aureus should not be confused with the similarly named and similarly dangerous (and also medically relevant) species of the genusStreptococcus.

S. aureus was discovered in Aberdeen, Scotland in 1880 by the surgeon Sir Alexander Ogston in pus from surgical abscesses. Each year, some 500,000 patients in American hospitals contract a staphylococcal infection.

Microbiology

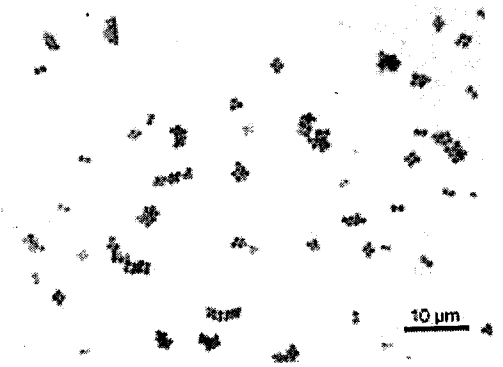


FIGURE 3 MICROBIOLOGY OF Staphylococcus Aures

Gram stain of S. aureus. Staphlococcus aureus typically occur more in clusters than chains, and the cells take up Gram stain well

S. aureus is a facultatively anaerobic, Gram-positivecoccus, which appears as grape-like clusters when viewed through a microscope and has large, round, golden-yellow colonies, often with hemolysis, when grown on blood agar plates. The golden appearance is the etymological root of the bacteria's name; aureus means "golden" in Latin.

S. aureus is catalase-positive (meaning that it can produce the enzyme "catalase") and able to convert hydrogen peroxide (H_2O_2) to water and oxygen, which makes the catalase test useful to distinguish staphylococci from enterococci and streptococci. A small percentage of S. aureus can be differentiated from most other staphylococci by the coagulase test: S. aureus is primarily coagulase-positive (meaning that it can produce the enzyme "coagulase") that causes clot formation, whereas most other Staphylococcus species are coagulase-negative.However, while the majority of S. aureus are coagulase-positive, some may be atypical in that they do not produce coagulase (the most common organism in patients with nosocomial bacteria is coagulase-negative staphylococcus). Incorrect identification of an isolate can impact implementation of effective treatment and/or control measures.

Mechanisms of antibiotic resistance:

Staphylococcal resistance to penicillin is mediated by penicillinase (a form of β-lactamase) production: an enzyme that cleaves the β-lactam ring of the penicillin molecule, rendering the antibiotic ineffective. Penicillinase-resistant β-lactam antibiotics such as methicillin, nafcillin, oxacillin, cloxacillin, dicloxacillin, and flucloxacillin are able to resist degradation by staphylococcal penicillinase.

Resistance to methicillin is mediated via the mecoperon, part of the staphylococcal cassette chromosome mec (SCCmec). Resistance is conferred by the mecA gene, which codes for an altered penicillin-binding protein (PBP2a or PBP2') that has a lower affinity for binding β-lactams (penicillins, cephalosporins, and carbapenems). This allows for resistance to all β-lactam antibiotics and obviates their clinical use during MRSA infections. As such, the glycopeptidevancomycin is often deployed against MRSA.

Aminoglycoside antibiotics such as kanamycin, gentamicin, streptomycin, etc. were once effective against Staphylococcal infections until strains evolved mechanisms to inhibit the aminoglycosides action, which occurs via protonated amine and/or hydroxyl interactions with the ribosomal RNA of the bacterial 30S ribosomal subunit There are three main mechanisms of aminoglycoside resistance mechanisms which are currently and widely accepted: Aminoglycoside modifying enzymes, Ribosomal mutations, and active efflux of the drug out of the bacteria.

Aminoglycoside-modifying enzymes inactivate the aminoglycoside by covalently attaching either a phosphate, nucleotide, or acetyl moiety to either the amine and/or the alcohol key functional group of the antibiotic. This changes the charge or sterically hinders the antibiotic, decreasing its ribosomal binding affinity. In S. aureus, the best-characterized aminoglycoside modifying enzyme is ANT(4')IA Aminoglycoside adenylyltransferase 4' IA. This enzyme has been solved by x-ray crystallography.[26] The enzyme is able to attach an adenyl moiety to the 4' hydroxyl group of many aminoglycosides including kamamycin and gentamicin.

Glycopeptide resistance is mediated by acquisition of the vanA gene. The vanA gene originates from the enterococci and codes for an enzyme that produces an alternative peptidoglycan to which vancomycin will not bind.

Today, S. aureus has become resistant to many commonly used antibiotics. In the UK, only 2% of all S. aureus isolates are sensitive to penicillin with a similar picture in the rest of the world. The β-lactamase-resistant penicillins (methicillin, oxacillin, cloxacillin, and flucloxacillin) were developed to treat penicillin-resistant S. aureus and are still used as first-line treatment. Methicillin was the first antibiotic in this class to be used (it was introduced in 1959), but, only two years later, the first case of methicillin-resistant S. aureus (MRSA) was reported in England.

Despite this, MRSA generally remained an uncommon finding even in hospital settings until the 1990s when there was an explosion in MRSA prevalence in hospitals where it is now endemic.

MRSA infections in both the hospital and community setting are commonly treated with non-β-lactam antibiotics such as clindamycin (a lincosamine) and co-trimoxazole (also commonly known as trimethoprim/sulfamethoxazole). Resistance to these antibiotics has also led to the use of new, broad-spectrum anti-Gram-positive antibiotics such as linezolid because of its availability as an oral drug. First-line treatment for serious invasive infections due to MRSA is currently glycopeptide antibiotics (vancomycin and teicoplanin). There are number of problems with these antibiotics, such as the need for intravenous administration (there is no oral preparation available), toxicity, and the need to monitor drug levels regularly by blood tests. There are also concerns that glycopeptide antibiotics do not penetrate very well into infected tissues (this is a particular concern with infections of the brain and meninges and in endocarditis). Glycopeptides must not be used to treat methicillin-sensitive S. aureus (MSSA) as outcomes are inferior.

Because of the high level of resistance to penicillins and because of the potential for MRSA to develop resistance to vancomycin, the Centers for Disease Control and Prevention have published guidelines for the appropriate use of vancomycin. In situations where the incidence of MRSA infections is known to be high, the attending physician may choose to use a glycopeptideantibiotic until the identity of the infecting organism is known. After the infection is

confirmed to be due to a methicillin-susceptible strain of S. aureus, treatment can be changed to flucloxacillin or even penicillin as appropriate.

Vancomycin-resistant S. aureus (VRSA) is a strain of S. aureus that has become resistant to the glycopeptides. The first case of vancomycin-intermediate S. aureus (VISA) was reported in Japan in 1996; but the first case of S. aureus truly resistant to glycopeptide antibiotics was only reported in 2002. Three cases of VRSA infection have been reported in the United States as of 2005.

2.5 DIFFERENT METHODS OF SYNTHESIS OF ZnO NANOPARTICLES

2.5.1 INTRODUCTION

In the past decade, a great deal of interest has been invested in synthesis and characteristics of nanoparticles or quantum dots. Nanoparticles or quantum dots are defined as small particles with1-100 nm in diameter. As particles diameter approaches to their Bohr diameter, the optical properties begin to change and quantum confinement effect begins to play a much more important role. It results in great differences in physical and electronic properties between the nanometer-scale particles and bulk materials. Thus, nano-scale particles possess different physical and chemical properties compared to bulk materials. Better sinterability, higher catalytic activity and other unusual properties may be expected because of their nano-sized crystallite, large surface area and different surface properties (such as surface defect) etc. Zinc oxide (ZnO) is an n-type , direct band gap. Semiconductor material.ZnO nanostructures are used in a wide range of applications including field emission displays. Nano-photonic devices, piezoelectric transducers, varistors, phosphors and transparent conducting films. This is possible as ZnO has three key advantages (Baglioni et al 2003).

First, it is semiconductor, with a direct wide gap of 3.37 eV and a large excitation binding energy (60 meV). It is an important functional oxide, exhibiting near-ultraviolet emission and transparent conductivity. Secondly, because of its noncentralsymmetry ,ZnO is piezoelectric, which is a key property in building electromechanical coupled sensors and transducers. Finally, ZnO is bio safe and biocompatible, and can be used for biomedical applications without coating.

With these three unique characteristics ZnO could be one of the most important nanomaterials in future research and applications (Ivanova et al 2007).

2.5.2 Levitational gas condensation

The levitational gas condensation (LGC) method is one of the physical approaches to fabricate nanoparticles reported by Uhm et al (2004). It is a simple one step process for synthesizing nanoparticles with uniform size distribution. Uhm et al also reported about the ZnO nanoparticles synthesized by the newly modified LGC ad phase evolution (Uhm 2007). High purity ZnO powders were synthesized by the LGC method shown in figure. The apparatus consist of high frequency induction generator of 2.5kW, levitation and evaporation chamber, and oxygen concentration control unit. The wire feeding velocity (VZn) and mixed Ar and O2 gas pressure in chamber was 50mm/min and 18kPa, respectively.

2.5.3 A Novel combustion synthesis method

Hwang et al (2004) reported the synthesis and characterization of nano crystalline ZnO powders by a novel combustion synthesis method. The basis of this method is described in detail here : the mixtures composed of metal nitrates added with a suitable fuel powders in an appropriate ratio, are to be ignited ad burnt to form ceramic oxides. In a sense, gunpowder can generate a lot of gases when burned. If such application is utilized in the synthesis of ceramic powders, a great deal of gases can be released via combusting reactants.

This study was done with glycine (NH_2CH_2COOH) as fuel since its price in inexpensive and its combustion heat (-3.4 kcal/g) is more negative when compared with urea(-2.98kcal/g). On the other hand zinc nitrate$Zn(NO_3)2.6H_2O$ is utilized in the present study because of its dual role of being the zinc source and the oxidant. The description of the experimental procedure is as given below. ZnO powder was synthesized by an amount of 25g per batch according to the flow chart shown in the figure. Analytic grade Zinc nitrate and glycine were directly mixed at a desired molar ratio without adding water. From our experiment, it was found that zinc nitrate possess hygroscopicity. The reactant mixture is easy to absorb moisture from the air and to become a transparent slurry matter.

Therefore, glycine and zinc nitrate can be mixed well by stirring, which makes them almost as homogeneous mixtures. This slurry mixtures was heated using a hot plate at 100 °C to dehydrate. The dried mixture possess the characteristic of combustion, which can be ignited to start combustion reaction by using mini gas burner. This is closely followed by the combustion of the precursor with the evolution of a large volume of gases, producing a loose product. When comparing with other methods, it is a simple, quick and inexpensive method involving a single step reaction.

2.5.4 Sol gel synthesis

The sol-gel process is a wet-chemical technique (also known as chemical solution deposition) widely used recently in the fields of materials science and ceramic engineering. Such methods are used primarily for the fabrication[disambiguation needed] of materials (typically a metal oxide) starting from a chemical solution (sol, short for solution) which acts as the precursor for an integrated network (or gel) of either discrete particles or network polymers. [23]

Typical precursors are metal alkoxides and metal chlorides, which undergo hydrolysis and polycondensation reactions to form either a network "elastic solid" or a colloidal suspension (or dispersion) – a system composed of discrete (often amorphous) submicrometer particles dispersed to various degrees in a host fluid. Formation of a metal oxide involves connecting the metal centers with oxo (M-O-M) or hydroxo (M-OH-M) bridges, therefore generating metal-oxo or metal-hydroxo polymers in solution. Thus, the sol evolves towards the formation of a gel-like diphasic system containing both a liquid phase and solid phase whose morphologies range from discrete particles to continuous polymer networks.[24]

In the case of the colloid, the volume fraction of particles (or particle density) may be so low that a significant amount of fluid may need to be removed initially for the gel-like properties to be recognized. This can be accomplished in any number of ways. The most simple method is to allow time for sedimentation to occur, and then pour off the remaining liquid. Centrifugation can also be used to accelerate the process of phase separation.

Removal of the remaining liquid (solvent) phase requires a drying process, which is typically accompanied by a significant amount of shrinkage and densification. The rate at which the

solvent can be removed is ultimately determined by the distribution of porosity in the gel. The ultimate microstructure of the final component will clearly be strongly influenced by changes implemented during this phase of processing. Afterwards, a thermal treatment, or firing process, is often necessary in order to favor further polycondensation and enhance mechanical properties and structural stability via final sintering, densification and grain growth. One of the distinct advantages of using this methodology as opposed to the more traditional processing techniques is that densification is often achieved at a much lower temperature.

The precursor sol can be either deposited on a substrate to form a film (e.g. by dip-coating or spin-coating), cast into a suitable container with the desired shape (e.g. to obtain a monolithic ceramics, glasses, fibers, membranes, aerogels), or used to synthesize powders (e.g. microspheres, nanospheres). The sol-gel approach is a cheap and low-temperature technique that allows for the fine control of the product's chemical composition. Even small quantities of dopants, such as organic dyes and rare earth metals, can be introduced in the sol and end up uniformly dispersed in the final product. It can be used in ceramics processing and manufacturing as an investment casting material, or as a means of producing very thin films of metal oxides for various purposes. Sol-gel derived materials have diverse applications in optics, electronics, energy, space, (bio)sensors, medicine (e.g. controlled drug release) and separation tehnology.

2.5.5 DC thermal plasma synthesis

Ko et al(2006) reported a rapid fabrication technique of ZnO nanopowders by DC thermal plasma synthesis with a high production rate. The growth rate and shapes of the nanopowders could be controlled by changing plasma gas combination and flow rate. In this ,ZnO nanopodwers were synthesized in a novel de plasma reactor operated at 70kW and atmospheric pressure as shown in the figure.

Commercial zinc powders containing impurities of Cr, Fe and Pb less than 50 ppm were used as raw materials. The Zn powders were fed into plasma flame through nitrogen carrier gas and subsequently underwent vaporization, oxidation and quench process. The ZnO nanopowders synthesis rate could be 1.2kg/h(Lin et al 2007).

Zinc oxide is a unique material that exhibits semi conducting, piezoelectric and pyroelectric multiple properties. The various methods developed and tried by researchers have been compared review for their merits and demerits. It can be concluded that the choice of method ad precursors will entirely depend on the end use requirements of the synthesized nanoparticles (Feiet all 2007).

2.6 CHARACTERIZATION OF NANO PARTICLES

The characterization of nanoparticles can be done by the following tests:

I. X-ray powder Diffraction Method (XRD)

II. Scanning Electron Microscopy(SEM)

2.6.1 X-ray Powder Diffraction method (XRD)

XRD is a very important experimental technique that has been used to address all issues related to the crystal structure of solids, including lattice constants and geometry , identification of unknown materials, orientation of single crystals, preferred orientation of polycrystals, defects, stresses etc. In XRD, a collimated beam of Xrays, with a wavelength typically ranging from 0.7 to 2 A, is incident on a specimen and is diffracted by the crystalline phases in the specimen according to Bragg's law:

$A=2d \sin\theta$

Where d is the spacing between atomic planes in the crystalline phase and A is the X-ray wave length. The intensity of the diffracted X-rays is measured as a function of the diffraction angle 2θ and the specimen's crystalline phases and to measure its structural properties. XRD is non destructive and does not require elaborate sample preparation, which partly explains the wide usage of XRD method in materials characterization.

Monochromatic X-rays are used to determine the interplanarspacings of the unknown materials. The X-ray spectra generated by this technique, thus, provide a structural fingerprint of the unknown. Mixture of crystalline materials can also be analyzed and relative peak heights of multiple materials may be used to obtain semi-quantitative estimates of abundances.

Evaluation of crystallite size by peak profile analysis is one of the important application of powder XRD method to study on nano particles. It means that crystallite size under 100 nm can quantitatively be evaluated with usual powder diffractometer. The crystallinity of the nano particles was determined by XRD using a Bruker D* Advance X rays Diffractometer equipped with a Cu K α (λ=1.54 A°) sources (applied voltage 40kV, current 40mA). About 0.5g 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 degrees with a scan rate of 1.5°/min. Scherrer's equation (Jenkins and Synder 1996):

$$D= 0.89*\lambda / \blacktriangle W* \cos \theta$$

Where λ is the wavelength of the incident X ray beam, θ is the Bragg's diffraction angle, ▲W is the full width of the X ray pattern line at half peak height in radians.

2.6.2 SCANNING ELECTRON MICROSCOPY

SEM is one of the most widely used techniques used in characterization of nanomaterials and nanostructures. SEM provides the image of the morphology and microstructures of bulk amd nano structured materials and devices. The resolution of the SEM approaches a few nanometers and the instrument can operate at magnifications that are easily adjusted from 10 to over 300000. Not only does the SEM produce topographical information as optical microscopes do, it can also provide the chemical composition information near the surface(Howard et al 1980). 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 Jeol model JSM-6360.

2.7 METHODS OF FABRIC TREATMENT WITH THE NANOPARTICLES

In order to impart the samples fabrics with the multifunctionality, the sample fabrics can be treated with the nanoparticles using anyone of the following methods of application of the nanoparticles tot the fabric substrate (Mahltig et al 2003).

I. **Simple dip and dry method**: In this method, the known quantity of nano particles to be applied on to the fabric is taken in a beaker where the nanoparticles are kept in colloidal state by means of a dispersing agent or peptizing agent. The fabric is simply dipped in this colloidal suspension for about 10 minutes by immersing it fully in the suspension. The treated fabric is then taken out and dried by any one of the three drying methods namely, sun drying or hot air drying or microwave oven drying till the fabric is fully dry.

II. **Pad-Dry-Cure method:** A padding mangle is used for the purpose of applying the nano particles from the suspension to the fabric surface. When the fabric is fed to the nip of the padding rollers, the pressure of the nip helps the nanoparticles to be pressed on to the fabric surface. The treated fabric is then dried and then cured at a high temperature to ensure the fixation of the nano particles on to the fabric surface. This method invariably uses binder chemical which is used for the binding of the nanoparticles on to the fabric. It is important to check the compatibility of the binder with that of the dispersing agent and the nanoparticles. This method also requires higher quantity of nano particles and liquor as it necessitates the padding mangle

2.8 CHARACTERIZATION OF FABRIC SAMPLES INCORPORATED WITH NANO ZnO PARTICLES WITH ANTIBACTERIAL ACTIVITY

Microbes- bacteria, virus, fungi and yeast are present almost everywhere. Whereas human beings have an immune system to protect against accumulation of micro organisms, materials such as textiles can easily be colonized by high number of microbes. Micro organisms can adhere to textile substrates. At the same time, functional properties like tensile strength and water permeability are lost due to the fact that the fibers are degraded. It's a well known fact that the growth of bacteria and microorganisms in food or water is prevented when stored in silver vessels due to its antibacterial properties. Silver ions have a broad spectrum of anti microbial activities.

Therefore, antibacterial disinfection and finishing techniques have been developed for many steps of textiles including treatment of textile fibers by padding colon with nano sized silver, titanium dioxide and zinc oxide colloidal solutions (25-50ppm). Metallic ions display a certain degree of sterilizing effect.

It was seen that part of oxygen in the air or water is turned into active oxygen by means of photo-catalysis with the metallic ion, thereby dissolving the organic substance to create a sterilizing effect. With the use of the nano-sized particles, the number of particles per unit area is increased and thus anti-bacterial effects can be maximized (Duran et al 2007).

With the general trend towards increasingly stringent hygiene standards, the consumers desire greater safety and better comfort by avoiding these inconvenient phenomena. These factors are the driving force behind the development of antibacterial materials.

CHAPTER 3

MATERIALS AND METHODS

3.1 SYNTHESIS OF ZnO NANO PARTICLES

- Method - Sol-gel method
- Material - Zinc nitrate Hexa hydrate
- Precursors- Sodium Hydroxide
- Stabilizing agent- Amylose starch
- Apparatus- High Speed stirrer(centrifuged- 12000*g)
- Drying- 110°C.

EXPERIMENTAL PROCEDURE:

1. Different concentrations of soluble starch (1%) were dissolved in 500ml of distilled water by using microwave oven.
2. Zinc nitrate hexahydrate, 14.874 g (0.1 mol) was added in the above starch solution.
3. Then the solution was kept under constant stirring using magnetic stirrer to completely dissolve the zinc nitrate.
4. After complete dissolution, 500 ml of NaOH 1N was added drop-wise under constant string, drop by drop touching the walls of the vessel.
5. The aqueous clear solution turned into a milky white colloid without any precipitation. The reaction was allowed to proceed for 2 hours after complete addition of NaOH.
6. After the complete of reaction, the solution was allowed to settle for overnight and the supernatant was discarded carefully.
7. The remaining solution was centrifuged at 12000 X g for 10 min and the supernatant solution was discarded.
8. The NANO-ZnO was washed thrice using distilled water to remove the byproducts and the excessive starch that were bound with nanoparticles.

9. After complete washing, the NANO-ZnO was dried at 110 °C for 3 complete conversion of $Zn(OH)_2$ to ZnO (solid) and then it is converted into powder form.

3.2 CHARACTERIZATION OF NANO-ZnO PARTICLES

- Scanning Electron Microscope (SEM)
- XRD

EXPERIMENTAL PROCEDURE

3.2.1 SCANNING ELECTRON MICROSCOPE (SEM):

The NANO-ZNO samples were mounted on specimen stubs with double-sided adhesive tape and coated with gold/palladium in a sputter coater and examined with Philips XL 30 scanning electron microscope(SEM) at 10-12KV with tilt angle of 45 degree.

3.2.2 X-ray Powder Diffraction method (XRD):

Crystallite size under 100 nm can quantitatively be evaluated with usual powder diffrectometer. The crystallinity of the nano particles was determined by XRD using a Bruker D* Advance X rays Diffractometer equipped with a Cu K α (λ=1.54 A°) sources (applied voltage 40kV, current 40mA). About 0.5g 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 degrees with a scan rate of 1.5°/min. Scherrer's equation (Jenkins and Synder 1996):

$$D= 0.89*\lambda / \blacktriangle W* \cos \theta$$

Where λ is the wavelength of the incident X ray beam, θ is the Bragg's diffraction angle, ▲W is the full width of the X ray pattern line at half peak height in radians. The XRD characterization tests of the nanoparticles synthesized were carried out in the Testing and Instrumentation centre (TIC) of Kaunya University, Coimbatore, Tamilnadu.

3.3 COATING OF FABRICS WITH NANO-ZnO

- Fine-Medium weight 60s count 100% cotton fabric.
- Application Technique – **'Pad-Dry-Cure'** method.

EXPERIMENTAL PROCEDURE

1. A fine weight 100% cotton woven fabric (Plain weave, GSM 82, ends 75/inch; picks 60/inch, warp and weft count 40^s N).
2. ZnO nanoparticles were applied on cotton using pad-dry-cure method.
3. The cotton fabric cut to the size of 90 x 30 cm was immersed in the solution containing Nano-ZnO (0.5% & 1.0%) and acrylic binder (1%) for 5min and then it was passed through a padding mangle, which was running at a speed of 15m/min with a pressure of 15 kgfcm $^{-2}$ to remove excess solution.
4. The Material to Liquor ratio was 1:20.
5. A 100% wet pick-up was maintained for all of the treatments.
6. After padding the fabric was air-dried and then cured for 3 min at 140 degree C to polymerize the acrylic binder.
7. The fabric was then immersed for 5 min in 2 gl^{-1} of sodium lauryl sulfate to remove unbound nanoparticles.
8. Then the fabric was rinsed at least 10 times to completely take out all the soap solution. The fabric thus washed was air-dried.

3.4 CHARACTERIZATION OF FABRIC SAMPLES TREATED WITH NANO PARTICLES

I. 3.4.1 SCANNING ELECTRON MICROSCOPY(SEM)

The nano ZnO samples were mounted on specimen stubs with double sided adhesive tape and coated with gold/ palladium in a sputter coater and examined with Philips® XL30 scanning electron microscope(SEM) at 10-12 kV tilt at an angle of 45°.

II. 3.4.2 EVALUATION OF ANTIMICROBIAL ACTIVITY

The evaluation of antibacterial activity was done by the Quantitative assessment method as per AATCC test method 100-2004. The test was carried out with Staphylococcus Aureus American type culture collection No.6538 (for Gram Positive bacteria) and E-coli , American Type culture collection No.4352 for Gram egative Bacteria. The percentage reduction of bacteria by the 100% 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).

III. 3.4.3 PHYSICAL TESTING OF FABRICS

The physical properties of both the woven and knitted fabrics such as Air permeability (ASTM D737) of both treated and untreated samples were tested using the standard testing procedures and equipment's after conditioning the specimens at 65% RH and 21°C for 24 hrs y bringing them to approximate moisture equilibrium in the standard atmosphere for preconditioning textiles as directed in practice D 1776 in an environmental chamber(ASTM 2008).

CHAPTER 4

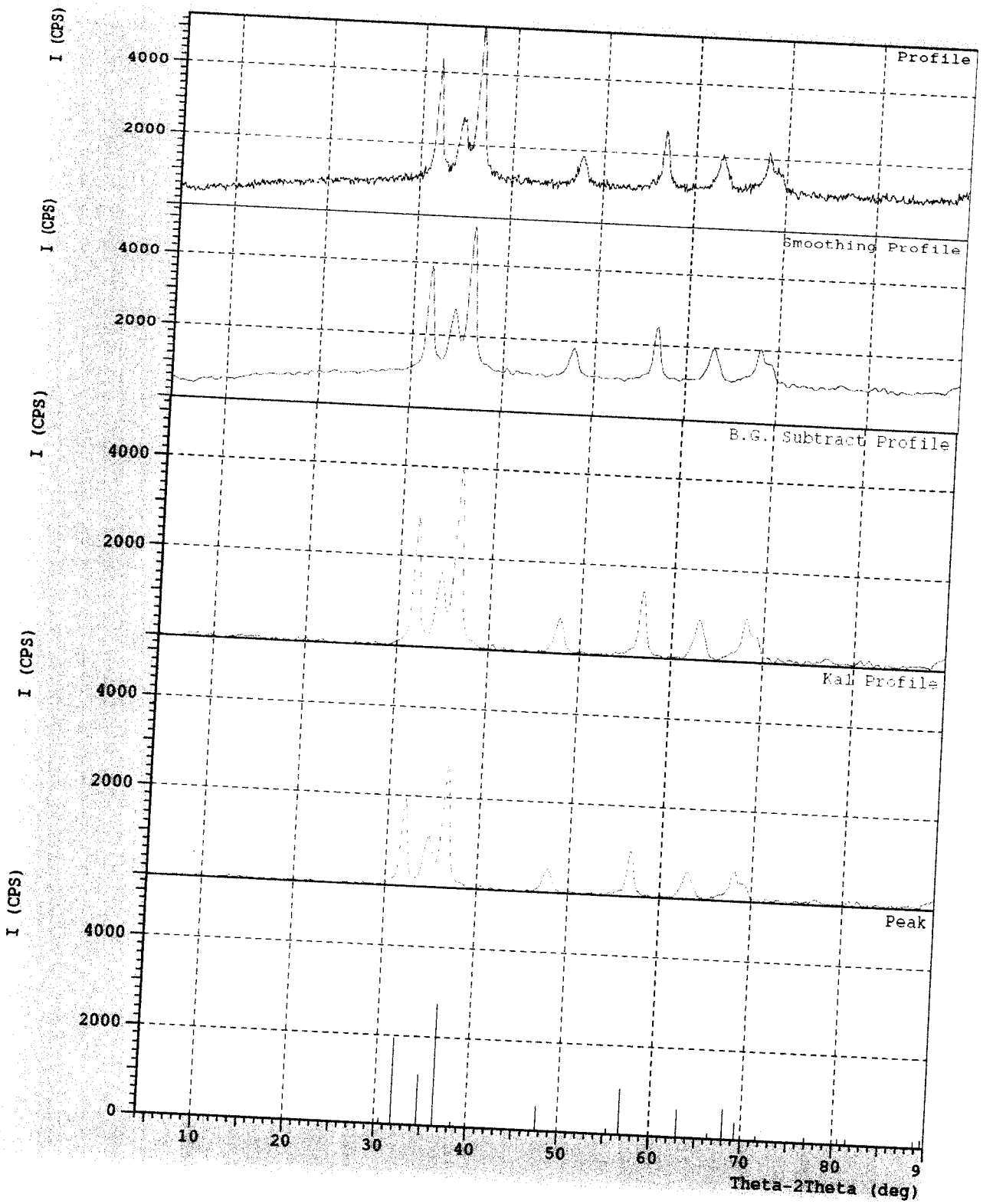
RESULTS AND DISCUSSIONS

4.1 CHARACTERIZATION OF NANO ZnO PARTICLES

4.1.1 X-RAYDIFFRACTION (XRD)

Figures show the XRD spectra of the two sets of ZnO nanoparticles Z1 and Z2 synthesized using the two different procedures III and IV. The spectra show well-defined package peaks typical of ZnO in the crystal structure of zincite, according to the joint committee on powder diffraction standard (JCPDS) card number 36-1451.This presence of well defined peaks indicates crystallinity of the synthesized solids. Traditionally, the broadening of the peaks in the XRD patterns of solids is attributed to particle size effects.(Jenkins and Snyder 1996).The mean crystallite size of a powder sample was estimated from the full width at half maximum(FWHM) of the diffraction peak according to the Scherrer's equation. The important aspect to be noted is that the peaks from synthesizes procedure IV(Figure) are broader than the peaks from synthesis procedure III(Figure).This inference suggests that the particles obtained from synthesis procedure IV are smaller than the particles obtained from synthesis procedure III. This has been confirmed by the results of TEM(Figures).

FIGURE 4 SHOWS THE XRD TEST RESULTS OF ZnO PARTICLES



4.1.2 SCANNING ELECTRON MICROSCOPY (SEM)

The surfaces of the treated fabrics were characterized by SEM. The SEM micrograph of Figure 4.2 shows the nanosized Zno particles on cotton sample(before washing).The nanoparticles are evenly spread on the fiber surface, although some aggregated nanoparticles are still visible. The particle's size plays a primary role in determining its adhesion to the fibers and thus the fabric. It is reasonable to expect that the larger particle agglomerates will be easily removed from the fiber surface, while the smaller particles will penetrate deeper and adhere strongly into the fabric matrix. Figure confirms that the large agglomerates are removed from the textile surface after Washing.

FIGURE 5 SIZE OF NANO ZnO THROUGH SEM

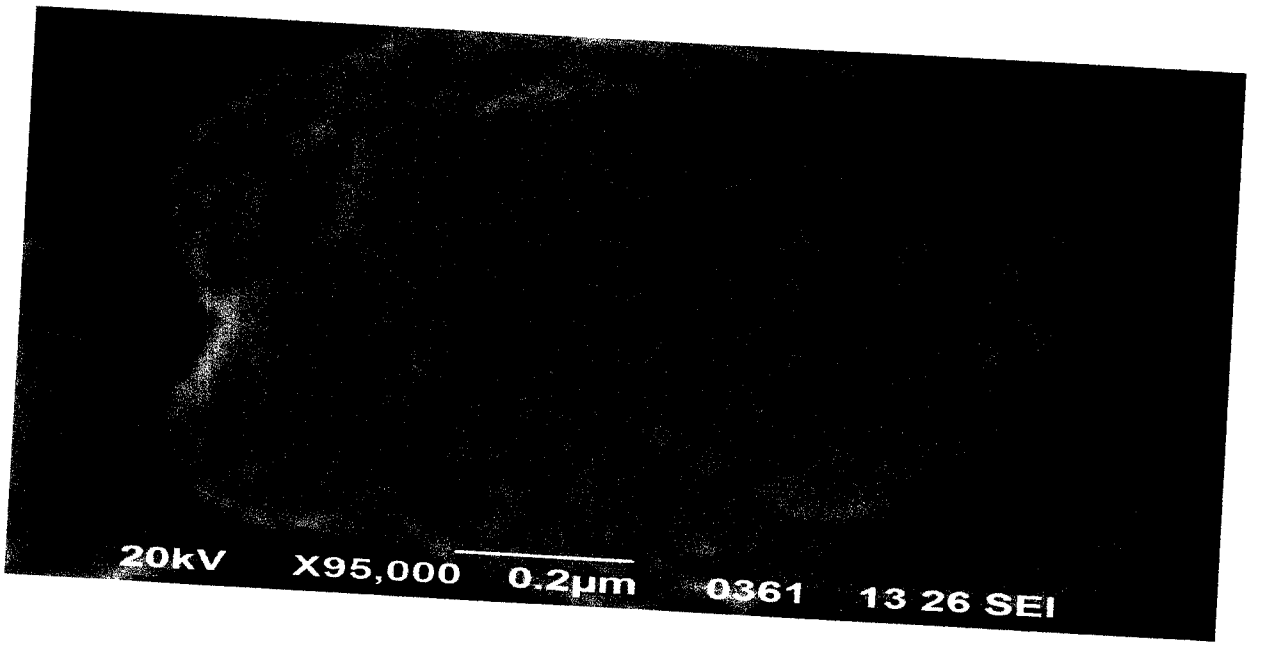
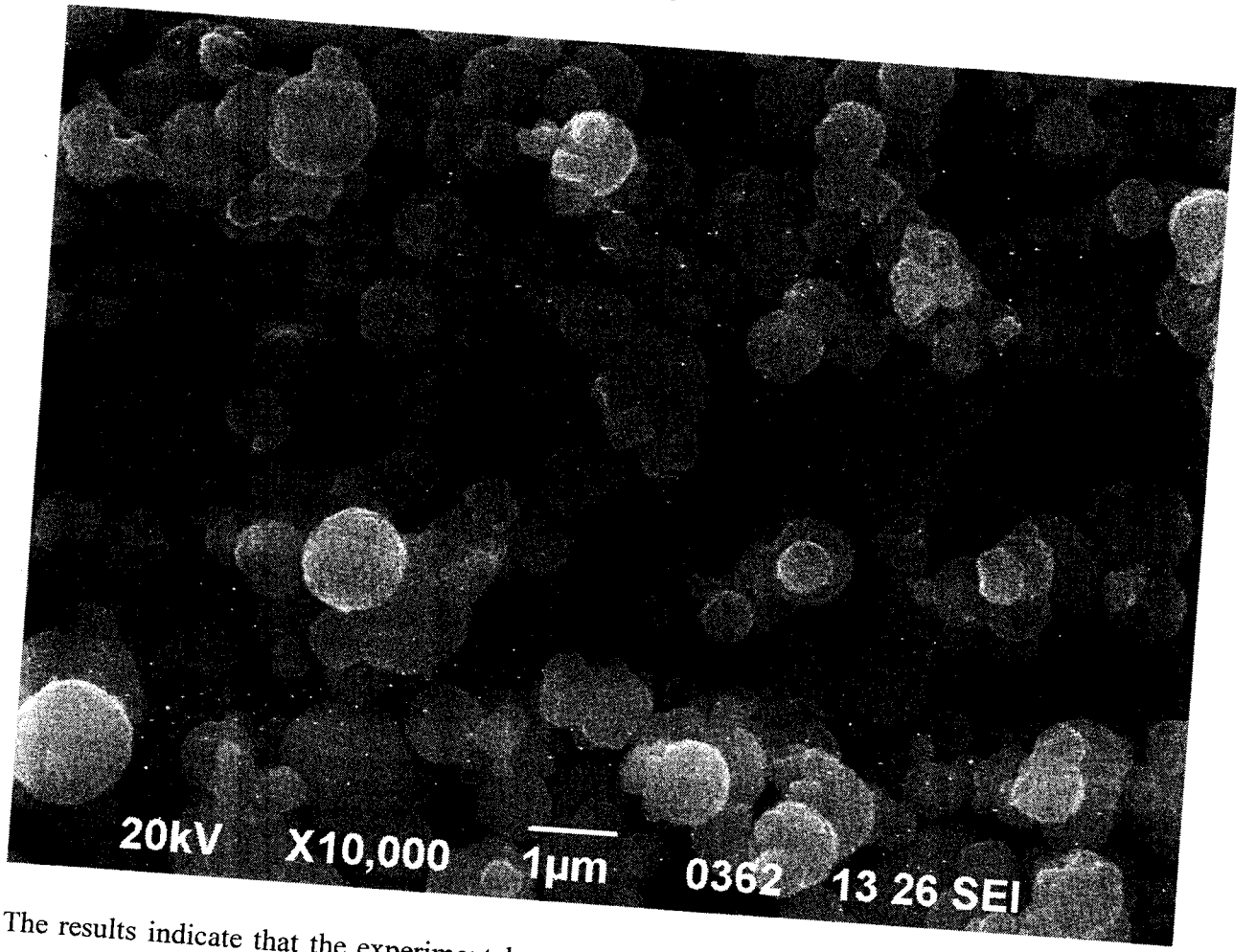


FIGURE 6 SEM PHOTOGRAPH OF NANO ZnO



The results indicate that the experimental conditions greatly affect the morphology and size of the particles, prepared with the different procedures(Table).In fact, on increasing the reaction temperature from 90^0 in water up to 150^0 in 1, 2-ethanedoil results in a significant lowering of the nanoparticles size and of their agglomeration number Uncalculated as(Shvalagin 2007):

$$N=4/3*R^3*N_A*\rho/M$$

Where N_A,R, ,and M are the Avogadro number, the radius of the nanoparticles, the density 5.47106 g/m^3) and the molecular weight (86.709 g mol^{-1}) of zinc oxide, respectively.

4.2 PROPERTIES OF FABRIC TREATED WITH ZnO NANOPARTICLES

4.2.1Functional testing of antimicrobial activity

To investigate the antibacterial activity of the treated woven fabrics, impregnation of the fabrics was done with ZnO nanoparticles using Pad-Dry-Cure method. Antibacterial test wss carried out with Staphylococcus Aureus, American type culture collection No.6538(Gram positive organism) and Escherichia coli, American type culture collection No.4352 (Gram negative organism). The quantitative assessment was done by AATCC test method 100-2004(2006). The fabric samples were tested for antibacterial activity and the results of the same are given below in the Table.

TABLE 4 ANTIMICROBIAL – ZONE OF INHIBITION

S.No	BACTERIA	Zone of Inhibition
1	Staphylococcus Aures	24 mm
2	Escherichia coli	13 mm

FIGURE 7 PETRI PLATE WITH STAPHYLOCOCCUS AURES SUB CULTURE

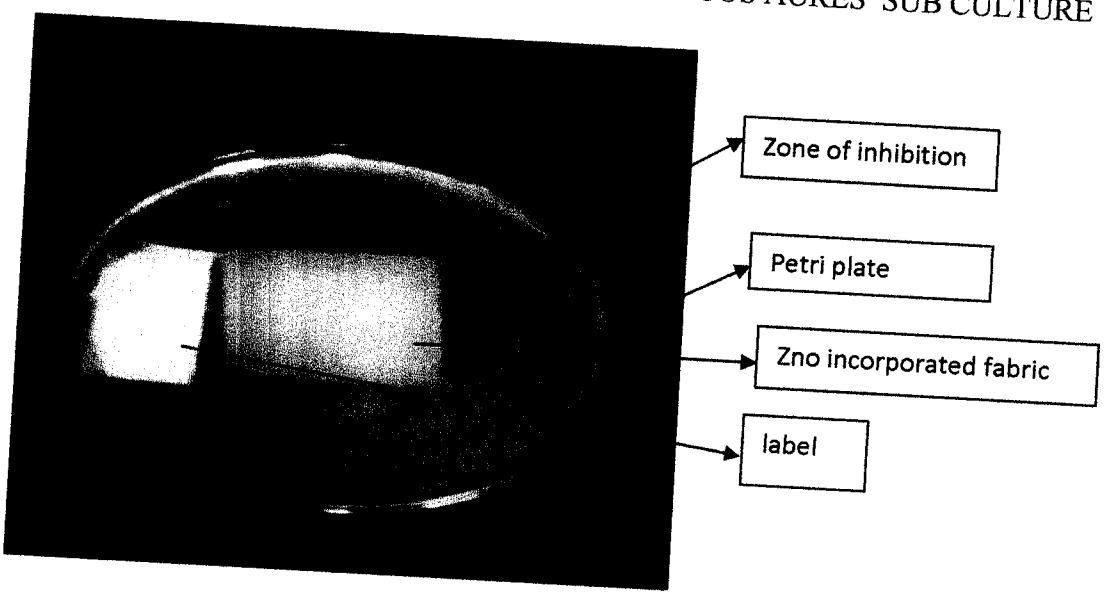


FIGURE 8 PETRI PLATE WITH ESCHERICHIA COLI SUB CULTURE

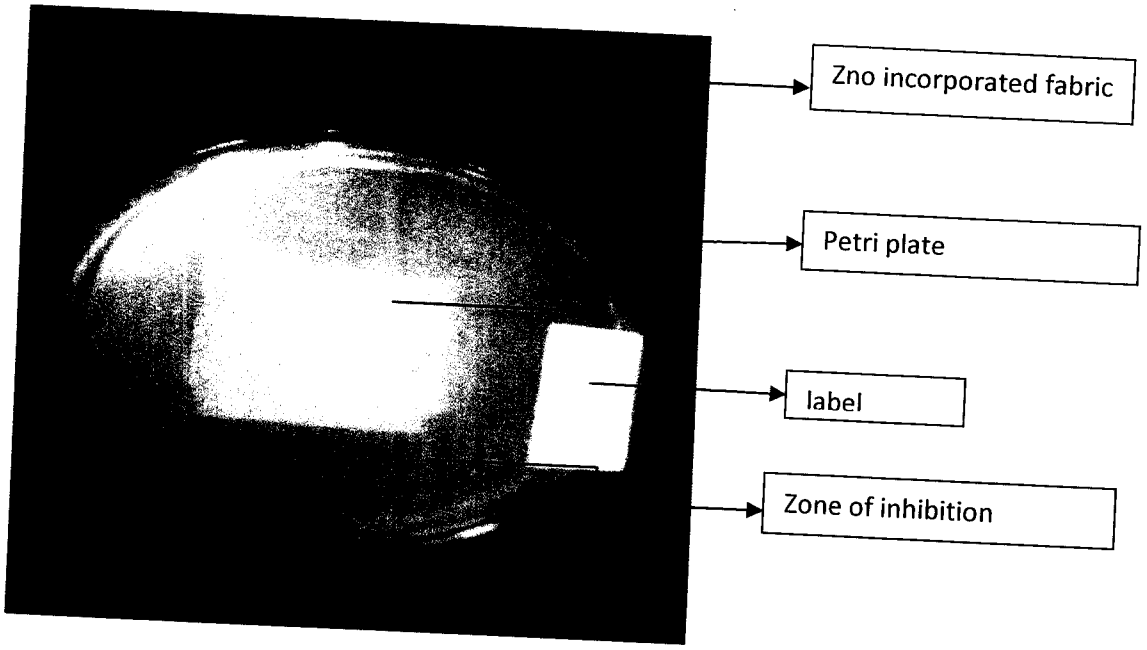
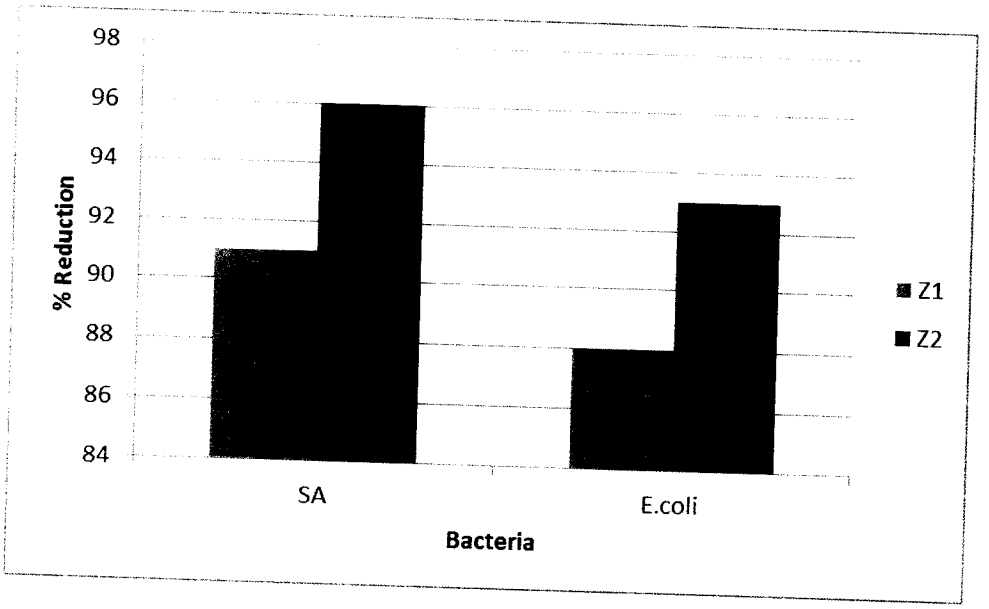


TABLE 5 SHOWS ANTIMICROBIAL TEST RESULT OF NANO ZnO

Details of Nano ZnO Particles	Fabric Particulars	Percentage reduction of Staphylococcus Aures	Percentage reduction of Escherichia Coli
Z_1 24 nm	Untreated	NIL	NIL
Z_1 24 nm	Woven 100% cotton	91	88
Z_2 18 nm	Untreated	NIL	NIL
Z_2 18 nm	Woven 100% cotton	96	93

The Table 4.2 shows the percentage reduction in bacteria for two representative types for the four types of fabric samples treated with two sets of ZnO nanoparticles viz. Z_1,Z_2. The sizes (in nm), conditions of synthesis, and codes(Z_1,Z_2) of the thus synthesized nanoparticles have also been indicated in this table for ready reference along with the sample fabric particulars. From these test results we can see that treated fabric samples shows excellent antimicrobial activity.

FIGURE 9 SHOWS THE BAR CHART OF ANTIMICROBIALITY TEST REULTS



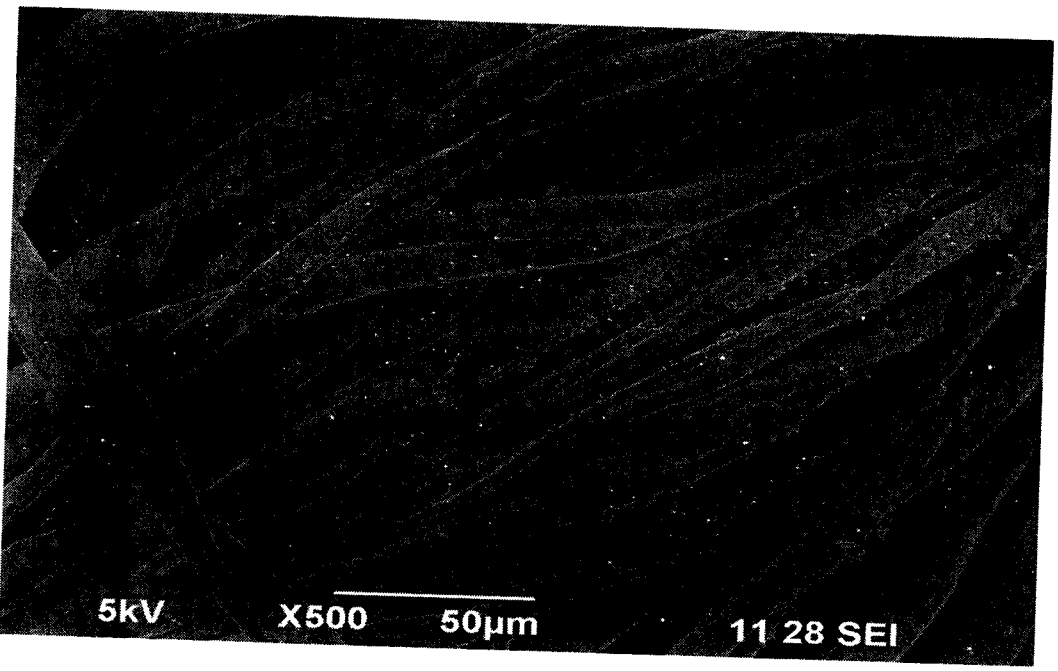
From the values of the test results of antimicrobial activity, we can see the fabrics treated with two sizes of the nano ZnO particles have shown excellent antimicrobial activity. It is very clear that the antimicrobial activity of the treated fabrics is due to the treatment of ZnO nanoparticles.

The 100% cotton woven fabrics treated with Z_2ZnO nano particles show better antibacterial property when compared to the 100% cotton woven fabrics with Z_1ZnO nano particles for both types of bacteria.

4.2.2 SCANNING ELECTRON MICROSCOPE:

The nano ZnOtreated fabric samples were punched in small dia of 1mm mounted on specimen stubs with double sided adhesive tape and coated with gold/ palladium in a sputter coater and examined with Philips® XL30 scanning electron microscope(SEM) at 10-12 kV tilt at an angle Of 45°.

FIGURE 10 SHOWS FABRIC INCORPORATED WITH ZnO NANO PARTICLES



The figure 4.8 shows the nano ZnO particles applied on the fabric. And the particle size achieved in applying to the fabric is VARYING FROM 172 nm TO 350 nm.

FIGURE 11 SHOWS SIZE OF NANO ZnO IN THE FABRIC

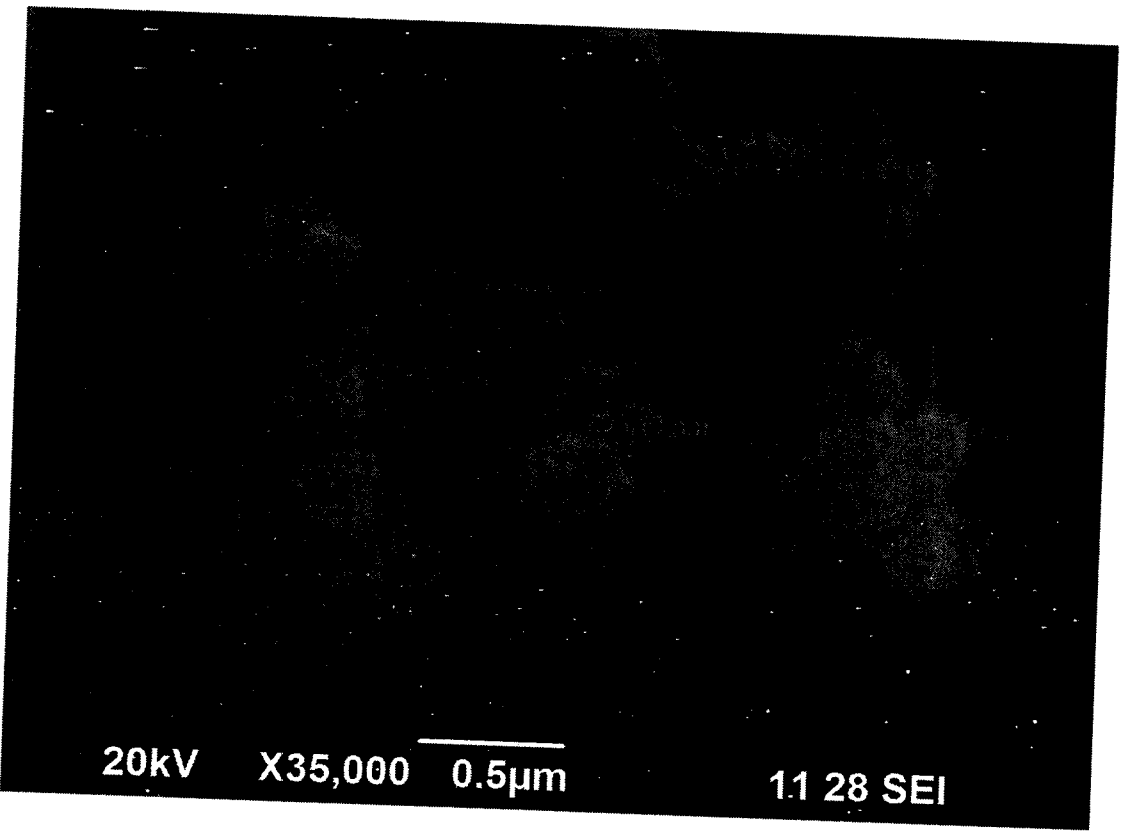
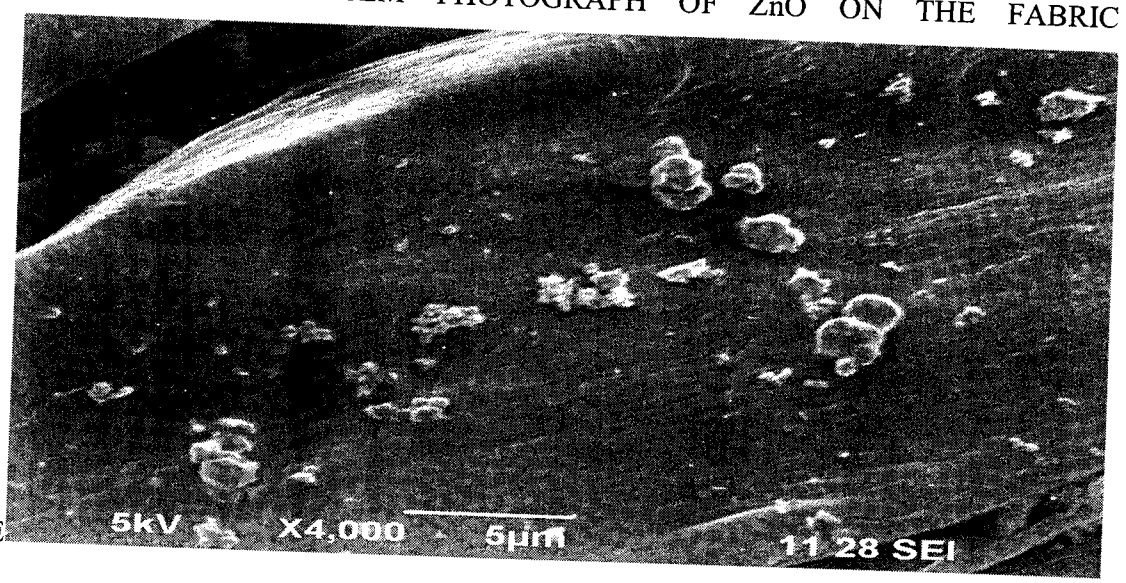


FIGURE 12 SHOWS THE SEM PHOTOGRAPH OF ZnO ON THE FABRIC SAMPLE



4.2.3 AIR PERMEABLITY TESTS:

The physical properties of both the woven and knitted fabrics such as Air permeability (ASTM D737) of both treated and untreated samples were tested using the standard testing procedures and equipment's after conditioning the specimens at 65% RH and 21°C for 24 hrs y bringing them to approximate moisture equilibrium in the standard atmosphere for preconditioning textiles as directed in practice D 1776 in an environmental chamber(ASTM 2008).

FIGURE 6 AIR PERMEABLITY TEST TABULATION

SERIAL NO.	AREA OF FABRIC TESTED	RESULT in litres/minute
1	$50cm^2$	78
2	$50\ cm^2$	73
3	$50\ cm^2$	76
4	$50\ cm^2$	79
	AVG	**76**

CHAPTER 5

CONCLUSION

- Synthesis of nanoparticles of ZnO in the laboratory using soft chemistry are feasible
- The nanoparticles thus synthesized have been found to be spherical, mono disperse, narrow size range. The synthesized nanoparticles have been successfully stabilized by using peptisizing agents.
- The experimental conditions greatly affect themorphology and size of the particles, prepared by the different procedures in the synthesis of ZnO.
- The analytical test methods such as XRD and SEM confirm the synthesis of nanoparticles.
- Woven fabrics can be imparted with the desired functions of antimicrobial by treating these fabrics with the nanoparticles of ZnO.
- In case of antimicrobial activity function, it has been seen that the fabrics that are treated with smaller sized ZnO nanoparticles show better results than the fabrics treated with slightly large sized ZnO particles. This is in full confirmation with the established fact that the smaller ZnO particles have a better anti microbial activity.
- ZnO nanoparticles show comparable performances of the three desired functions as their band-gap energy difference is very low. ZnO is preferable due to its lower cost .

CHAPTER 6

SCOPE OF FUTURE WORK

- The study on the functional finishes on the fabric coated with nano-particles can be done.
- Toxicity of the applied nano particle on the textile substrate can be studied.
- Influence of the activity of the nano particles prepared using different methods can be studied.
- Influence of different synthesis methods on the particle distribution and the intensity of the anti-microbial properties can be studied.

CHAPTER 7

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