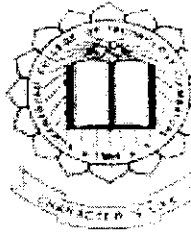


P-2599



**STUDY OF FUNCTIONAL FINISHING ON
SILK FABRIC USING NANO ZINC OXIDE**



A PROJECT REPORT

Submitted by

| | |
|---------------------|--------------------|
| PUSHPARAJ.D | 71205212027 |
| SIVAKUMAR.S | 71205212039 |
| SOWDESWARI.S | 71205212044 |
| SRINIVASAN.R | 71205212047 |



*In partial fulfilment for the award of the degree
of*

BACHELOR OF TECHNOLOGY
in

TEXTILE TECHNOLOGY

KUMARAGURU COLLEGE OF TECHNOLOGY, COIMBATORE

ANNA UNIVERSITY:: CHENNAI 600 025

APRIL 2009

ANNA UNIVERSITY: CHENNAI 600 025

BONAFIDE CERTIFICATE

Certified that this project report “**STUDY OF FUNCTIONAL FINISHING ON SILK FABRIC USING NANO ZINC OXIDE**” is the bonafide work of “**D.PUSHPARAJ, S.SIVAKUMAR, S.SOWDESWARI, R.SRINIVASAN**” who carried out the project work under my supervision.

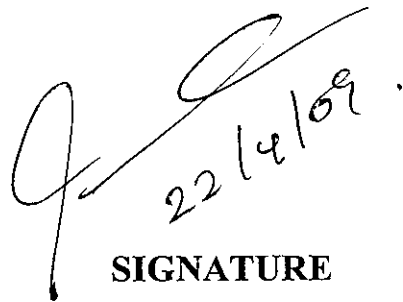


SIGNATURE

Dr. LOUIS D'SOUZA

HEAD OF THE DEPARTMENT

Department of Textile Technology,
Kumaraguru College of Technology,
Coimbatore-641006



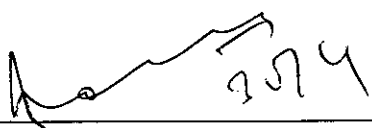
SIGNATURE

Prof. S.KATHIRVELU

SUPERVISOR

Assistant Professor
Department of Textile technology
Kumaraguru college of Technology
Coimbatore-641006

Viva- voce examination is conducted on ~~30-4-09~~



(INTERNAL EXAMINER)



(EXTERNAL EXAMINER)

ACKNOWLEDGEMENT

We convey our deepest sense of gratitude to our president **Prof.C.Ramasamy**, our principal **Dr.Joseph.V.Thanikal** and our Vice-Principal **Dr.M.Annamalai** under whose blessings this project has achieved its final shape.

Our wholehearted thanks to **Dr.Louis D'Souza**, Professor and Head, Department of Textile technology for his optimal encouragement and guidance.

We express our sincere gratitude to our guide **Prof.S.Kathirvelu**, Department of Textile Technology, who provided us a complete guidance in successful completion of this project.

We also express our sincere thanks to **Dr.R.Venckatesh**, Senior Lecturer , Department of science and Humanities, Kumaraguru College of Technology, for the care and support offered in doing this project.

We express our sincere thanks to South Indian Textile Research Association (**SITRA**), Department of Biotechnology, Kumaraguru College of Technology and Cochin University of science and Technology (**CUSAT**) for carrying out the testing work.

We heartly thank our Department Staffs and lab technicians for their support in completing our project.

We kindly thank our family and friends who lead our project to completion in a better way.

ABSTRACT

The project work was done in an attempt to study the functional finishing of silk fabric using nano zinc oxide . In the present work, nano zinc oxide were prepared by wet chemical method using zinc nitrate and sodium hydroxide as precursors and soluble starch as stabilizing agent. The synthesized nano particles were characterized using FTIR & XRD techniques. These nanoparticles, which have an average size of ~ 50 nm, were coated on the silk fabrics (plain woven and single jersey) using spraying technique. The finished fabric was done characterization test using SEM images. The finished fabrics were tested for their ultra-violet protection and antimicrobial characteristics using UV-Vis spectrophotometer and optical density method respectively. The various test results were recorded, studied, analysed and reported, the efficiency of the functional finishing was studied and reported. The results of the tests were analysed and discussed.

TABLE OF CONTENTS

| CHAPTER No. | TITLE | PAGE No. |
|----------------|---|----------|
| 1. | INTR ODUCTION | |
| 1.1 | GENERAL | 1 |
| 1.2 | MULTIFUNCTIONAL FINISHING | 1 |
| 1.3 | NANO FINISHING | 2 |
| 2. | REVIEW OF LITERATURE | |
| 2.1. | OVERVIEW OF FUNCTIONAL FINISHING OF TEXTILES | |
| 2.1.1. | Anti-bacterial finishing | 3 |
| 2.1.2. | UV protection finishing | 4 |
| 2.1.3. | Self cleaning | 6 |
| 2.1.4. | Comparison of traditional method & nano technology | 7 |
| 2.1.5. | Significance of nano particles | 8 |
| 2.2 | OVERVIEW OF SYNTHESIS OF ZINC OXIDE NANO PARTICLES | 9 |
| 2.3 | METHODS OF IMPARTING THE NANO PARTICLES OF METAL OXIDE ON TO TEXTILE SUBSTRATES | |
| 2.3.1 | Pad-dry-cure method | 28 |

| | | |
|-----------|---|-----------|
| 2.3.3 | Spraying | 29 |
| 3. | AIM & SCOPE | 30 |
| 4. | METHODOLOGY | |
| 4.1. | YARN PROCUREMENT | 31 |
| 4.2. | FABRIC MANUFACTURE | 31 |
| 4.3. | SYNTHESIS OF NANO ZINC OXIDE | 33 |
| 4.4. | APPLICATION OF NANO PARTICLES ONTO SILK FABRIC | 34 |
| 4.5. | TESTING | 34 |
| 5. | RESULTS AND DISCUSSION | |
| 5.1. | CHARACTERIZATION TESTS | |
| 5.1.1. | FTIR | 38 |
| 5.1.2. | XRD | 40 |
| 5.1.3. | SEM | 42 |
| 5.2. | FUNCTIONAL TESTING | 45 |
| 6. | CONCLUSIONS | 49 |
| 7. | REFERENCES | 51 |

LIST OF TABLES

| TABLE. NO | TITLE | PAGE NO |
|------------------|---|----------------|
| 1. | Solar protection factors (SPF) of undyed fabrics | 5 |
| 2. | Woven structure | 30 |
| 3. | Knitted structure | 31 |
| 4. | Test centers | 35 |
| 5. | Optical density value | 43 |

LIST OF FIGURES

| FIGURE .NO. | TITLE | PAGE NO. |
|-------------|---|----------|
| 1. | FTIR Graph | 38 |
| 2. | XRD Graph | 40 |
| 3. | SEM Untreated silk fabric 500x | 42 |
| 4. | SEM Untreated silk yarn 1000x | 42 |
| 5. | SEM Treated woven silk fabric 500x | 43 |
| 6. | SEM Treated woven silk fabric 1000x | 43 |
| 7. | SEM Treated knitted silk fabric 1000x | 44 |
| 8. | SEM Treated knitted silk fabric 500x | 44 |
| 9. | UV absorbance graph of nano zinc oxide | 46 |
| 10. | UV absorbance of treated woven fabric | 47 |
| 11. | UV absorbance of treated knitted fabric | 48 |

LIST OF SYMBOLS AND ABBREVIATIONS

| | |
|-----------|---------------------------------|
| C | - Celsius |
| FTIR | - Fourier transformed infra red |
| h | - Hour |
| Ppm | - Parts per million |
| SEM | - Scanning electron microscopy |
| SPF | - Solar protection factor |
| UV | - Ultra violet |
| XRD | - X-ray diffraction |
| μ | - Micron |
| λ | - Wavelength |
| θ | - Angle |
| Dia | - Diameter |

CHAPTER – 1

INTRODUCTION

1.1 GENERAL

The primary objective of modern textile manufacturers is to produce quality products at an economical price. Modern customers are interested in clothing that not only looks good, but also feels good. It has been identified, by both natural and synthetic garment manufacturers, that consumers increasingly give importance to comfort properties than to visual appearance. As a result comfort is being reinforced as a key parameter in clothing.

Comfort as a pleasant state of physiological, psychological and physical harmony between human being and the environment. Physiological comfort is related to the human body's ability to maintain life. Psychological comfort is the mind's ability to keep itself functioning satisfactorily with external help and physical comfort to the effect of the external environments of the body. Clothing plays an important role in providing comfort to its wearer, as it acts as a medium between humans and the environment. Hence, clothing with enhanced comfort properties is the need of the hour.

1.2 MULTI-FUNCTIONAL FINISHING

To achieve this, fabric should be given multiple finishing treatments. This involves increased usage of water and also it is not economical. Hence, a single process with multiple purposes has to be opted and **multi-functional finishing** is one such process where varied properties like Anti-Microbial, UV Protection,

Soil release, etc., can be achieved using a single finish (namely **Zinc oxide, titanium oxide**). This process involves less usage of water and cost of production is also less. Thereby, a product that is more economical and eco-friendly can be produced using this method.

1.3 NANO FINISHING

The finishing given to clothing should be durable, in order to make the finishes serve its purpose for a longer period of time. This level of durability for multiple properties can be obtained only using **nano finishing**. Nano finishing is a new era in textile finishing where particles of nano scaled size are used.

Nano particles can provide high durability for treated fabrics, with respect to conventional materials, because they possess large surface area and high surface energy that ensure better affinity for fabrics and lead to an increase in durability of the textile functions. Metal nano particles improve physical, chemical and biological properties of textile materials. Wash fastness is a particular requirement for textile and it is strongly correlated with the nano particles adhesion to the fibres. Wash fastness can be further improved with the formation of covalent bonding between nano particles and the fabrics surface.

Thus, the combination of multi-functional and nano finishing can produce **versatile** and **highly durable** material.

CHAPTER - 2

REVIEW OF LITERATURE

2.1 OVERVIEW OF FUNCTIONAL FINISHING OF TEXTILES

2.1.1. ANTI-BACTERIAL FINISHING

Anti-bacterial finishes can be divided into two types based on the mode of attack on microbes. One type consists of chemicals that can be considered to operate by a controlled-release mechanism. The antimicrobial is slowly released from a reservoir either on the fabric surface or in the interior of the fibre. This 'leaching' type of anti-microbial can be very effective against microbes on the fibre surface or in the surrounding environment. However, eventually the reservoir will be depleted and the finish will no longer be effective. In addition, the antimicrobial that is released to the environment may interface with other desirable microbes, such as those present in waste treatment facilities.

The second type of anti-microbial finish consists of molecules that are chemically bound to fibre surfaces. These products can control only those microbes that are present on the fibre surface, not in the surrounding environment. 'Bound' anti-microbials, because of their attachment to the fibre, can potentially be abraded away or become deactivated and lose long term durability.

Anti-microbial finishes that control the growth and spread of microbes are more properly called biostats, ie bacteriostats, fungistats. Products that actually kill microbes are biocides, ie bacteriocides, fungicides. This

distinction is important when dealing with governmental regulations, since biocides are strongly controlled. Textiles with biostatic properties, however, are subjected to fewer regulations.

The actual mechanisms by which antimicrobial finishes control microbial growth are extremely varied, ranging from preventing cell reproduction, blocking of enzymes, reaction with the cell membrane to the destruction of the cell walls and poisoning the cell from within.

2.1.2. ULTRAVIOLET PROTECTION FINISHING

When radiation strikes a fibre surface, it can be reflected, absorbed, transmitted through the fibre or pass between fibres. The relative amounts of radiation reflected, absorbed or transmitted depend on many factors, including the fibre type, the fibre surface smoothness, the fabric cover factor (the fraction of the surface area of the fabric covered by yarns) and the presence or absence of fibre delustrants, dyes and UV absorbers.

The effect of fibre type on the SPF of undyed fabrics of similar construction is demonstrated in table. Cotton and silk fibres offer little protection to UV radiation since the radiation can pass through without being markedly absorbed. Wool and polyester, on the other hand, have significant higher SPFs since these fibres will absorb UV radiation. Nylon falls in between these extremes. One factor influencing nylon and polyester absorbance is the presence of the delustrant TiO_2 , a material that strongly absorbs UV radiation.

Table : 1: Solar protection factors (SPF) of undyed fabrics

| Fabric description | Approximate SPF |
|------------------------------|-----------------|
| Cotton tricot | 4 |
| Wool tricot | 45 |
| Silk twill | 7 |
| Polyester tricot | 26 |
| Nylon/elastomer 80/20 tricot | 12 |

If the fibres absorb all of the incident radiation, then the only source of transmitted rays is from the spacing between the yarns. By definition, the theoretical maximum SPF is the reciprocal of 1 minus the cover factor.

$$SPF_{max} = \frac{1}{1 - cover\ factor}$$

Using a SPF value of 50 as the goal, a fabric with a cover factor of 0.98 and composed of fibres that absorb all of the non-reflected UV radiation. Of course, tight micro-fibre fabrics provide a better UV protection than fabrics made from normal sized fibres with the same specific weight and type of construction.

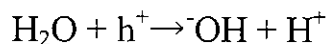
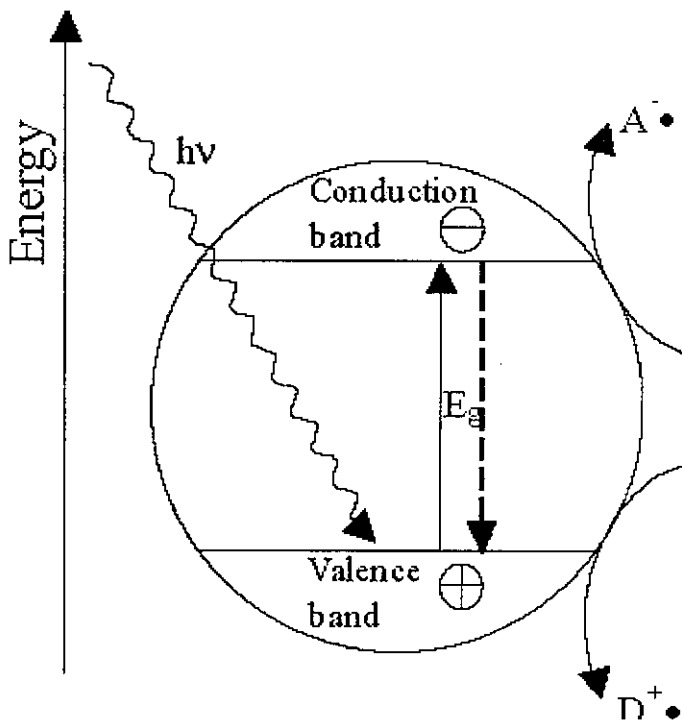
Many dyes absorb UV radiation as well as visible light. A cotton fabric dyed to a deep shade can achieve SPF values of 50 or higher just from the presence of the dye. Since fashion and comfort often dictate the use of

Dyestuff and auxiliary manufacturers have responded by developing a variety of materials suitable for use as UV protection finishes.

Rayleigh scattering theory states that “**scattering is inversely proportional to fourth power of wavelength**”. To scatter UV radiation between 200 and 400 nm, the optimum particle size should be between 20 and 40 nm. Hence, nano particles are more effective in scattering of UV rays than bulk particles, by which an enhanced UV protection can be provided.

2.1.3. SELF CLEANING

Nano-sized silver, Titanium dioxide and zinc oxide are used for imparting self-cleaning and anti-bacterial properties. Metallic ions and metallic compounds display a certain degree of sterilizing effect. It is considered that part of the oxygen in the air or water is turned into active oxygen by a catalyst containing the metallic ion, thereby destroying the organic substance to create a sterilizing effect. Nanoparticles possess enhanced catalytic activities due to their highly stressed surface atoms which are very reactive. With the use of nano-sized particles, the number of particles per unit area is enormously increased.



Operation of a photochemical excited ZnO particle

Zinc Oxide is a photocatalyst when it is illuminated by light of energy higher than its band gap, electrons in ZnO will jump from the valence band to the conduction band, and the electron (e-) and electric hole (h+) pairs will form on the surface of the photocatalyst. The negative electrons and oxygen will combine to form O_2^- radical ions, whereas the positive electric holes and water will generate hydroxyl radicals OH. Since both products are unstable chemical entities, when the organic compound falls on the surface of the photocatalyst it will combine with O_2^- and OH and turn into carbon dioxide and water. This cascade reaction belongs to the oxidation-reduction class.

During the reaction, photocatalyst is able to decompose common organic matter in the air, such as molecules causing odour, bacteria and viruses or organic stain and dirt.

Nano-ZnO provides effective photocatalytic properties once it is illuminated by light and so it is employed to impart anti-bacterial properties to textiles.

2.1.4.COMPARISON OF TRADITIONAL METHOD & NANO TECHNOLOGY

The traditional method of producing nano particles involves TOP DOWN approach. In this approach bigger particles are broken down into smaller particles. This method of nano particle synthesis has the following disadvantages:

- Less precise.
- Produces lots of waste and pollution.
- Consumes lots of energy.

On the other hand, nano technology involves **bottom up** approach. In this approach particles of sub-nano level are built into nano particles. It is a constructive process. Bulk particles can be built precisely in tiny building blocks. Hence,it possess the following advantages:

- Absolute Precision (down to one single atom).
- Complete control of process (no wastage).
- Less energy needed.
- More eco-friendly.

- Nanotechnology provides the ability to work on a nano or submicron scale to create intelligent structures that are stronger and have fundamentally different performance-enhancing molecular organizations.
- Working on a nano-scale allows the building of molecular architectures that can be specifically designed to create desirable attributes in *fabrics*.
- This type of enhancement is the most powerful method of uniting a performance benefit with your *marketable textiles*.

2.1.5. SIGNIFICANCE OF NANO PARTICLES

QUANTUM EFFECTS

- At the lower end of the nanoscale, space available to electrons is restricted.
- The properties of nano particles are different from bulk properties, especially electronic, optical and magnetic.

SURFACE EFFECTS

- Ratio of surface area to volume is relatively high for nano particles.
- When nano particles are used the surface properties are predominate and reactivity can be enhanced.
- Nano particles rendered are invisible and do cause any change in the visual appearance.

2.2. OVERVIEW OF SYNTHESIS OF METAL OXIDE NANO PARTICLES

2.2.1. SYNTHESIS OF ZnO NANO PARTICLES

Nanoparticles or quantum dots are defined as small particles with 1-100 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 sinter ability, 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. 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.

- It is semiconductor, with a direct wide band 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.
- ZnO is piezoelectric, because of its non central symmetry, which is a key property in building electromechanical coupled sensors and transducers.
- 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

techniques have been used to synthesize ZnO nano particles including chemical or physical methods.

PHYSICAL METHODS:

- a. Thermal hydrolysis technique
- b. Hydrothermal processing
- c. Sol-gel method.



CHEMICAL METHODS:

- a. Spray pyrolysis
- b. vapour condensation method
- c. Thermo chemical decomposition of metal organic precursors.

At present, the popular that are generally being used to obtain nano-sized ZnO powder are: vapor method and sol-gel method. For the vapor method, the resulting powders are agglomerates rather than separated powders because the reaction condition during the process is difficult to control. In addition, the method is time and energy-consuming. The sol-gel method produces uniform ZnO powders. However, strict control of the reaction condition is necessary because of its violent hydrolysis reaction in air during the synthesis. In addition, this method has high material costs, and so it is not commercialized but done only in small-scale laboratories. Therefore, the synthesis methods of nanocrystalline ZnO powders need to be improved. Nevertheless, the main problems of most methods have been the poor throughput efficiency and the difficulty in size control.

Diversified synthesis methods for ZnO nanoparticles were reported in recent years such as thermal decomposition, solgel techniques, supercritical precipitation and colloidal synthesis. Wang and Muhammed used a chemical precipitation method to synthesize ZnO nanoparticles with controlled morphology; however, the precursor powders must be heated in air in order to obtain ZnO. McBride et al. and Oliveira et al. had directly obtained microcrystalline ZnO by the reaction of zinc salts with sodium hydroxide. The sizes of the zinc oxide particles were within micron or sub-micron scales. Qian et al. reported a method for preparing nanometer-sized ZnO crystals via ultrasonic irradiation in absolute ethanol. Kim et al. reported a method for preparing mono dispersed ZnO nano particles by the thermolysis of Et ZnOiPr as a single molecular precursor and trioctylphosphine oxide, but high temperature(160°C) must be used to decompose the precursor.

However, most methods employed organic solvents or required rigorous reaction conditions and complicated procedures.

Therefore, it is important to develop a process in which particles having controlled characteristics including size, morphology, and composition can be produced. To be industrially relevant, the process needs to be low cost and involve both continuous operation and a high production rate. Functional materials with specific physical and chemical properties begin with molecular precursors that must be transformed into the product.

a. ZnO nano powders fabricated by DC thermal plasma synthesis:

Ko et al. reported a rapid fabrication technique of ZnO nano powders by dc thermal plasma synthesis with a high production rate. ZnO nano powders were successfully fabricated by Ko et al by a novel dc thermal plasma synthesis process with a high production rate. The growth rate and shapes of the ZnO nanopowders could be controlled by changing plasma gas combination and flow rate.

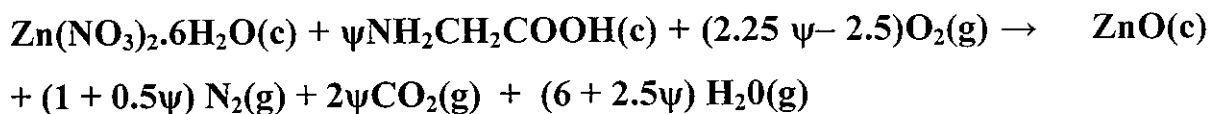
In this study, ZnO nano powders were synthesized in a novel dc plasma reactor operated at 70kW and atmospheric pressure. Commercial zinc powders containing impurities of Cr, Fe and Pb less than 50 ppm were used as the raw materials. The Zn powders were fed into plasma flame through nitrogen carrier gas and subsequently underwent vaporization, oxidation and quench processes. The ZnO nanopowders synthesis rate could be 1.2 kg/h.

b. Synthesis of ZnO powders by a novel combustion synthesis method:

Hwang et al. reported the synthesis and characterization of nano crystalline ZnO powders by a novel combustion synthesis method. The basis of this method is as follows: the mixtures composed of metal nitrates added with a suitable fuel powders in an appropriate ratio, are to be ignited and burnt to form ceramic oxides. In a sense, gun powder 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 the combusting reactants. They are conducive to heat loss through convection, resulting in rapid reduction of product temperature. On a practical level, it is suggested that fine powders could be produced due to the prevention from both the growth of grains and the agglomeration of particles.

This study was done with glycine ($\text{NH}_2\text{CH}_2\text{COOH}$) as fuels since its price is inexpensive and its combustion heat (-3.24 kcal/g) is more negative when compared with urea (-2.98 kcal/g) or citric acid (-2.76 kcal/g). On the other hand, zinc nitrate [$\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$] is utilized in the present study because of its dual role of being the zinc source and the oxidant.

The combustion reaction between zinc nitrate and glycine may be analogized and be expressed as follows: ($7.5 + 2.75$ moles gases produced)



The description of the experimental procedure is as given below. ZnO powder was synthesized by an amount of 25 g per batch. 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 possesses 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 mixture was heated using a hot-plate at 100°C to dehydrate. The dried mixture (hereafter termed as precursor) possesses the characteristic of combustion, which can be ignited to start combustion of the precursor with the evolution of a large volume of gases, producing a loose product. It was found that the nature of the combustion and the characteristics of as-synthesized product depend on the ψ value.

When comparing with other methods, it is a simple, quick, and inexpensive method involving a single-step reaction. It was interesting to note that the crystalline structure of as-synthesized powders becomes more defined as ψ increases.

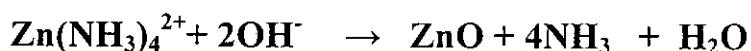
c. Low-temperature-synthesis of ZnO nano particles through ZnS

Lu et al. have successfully demonstrated the synthesis of ZnO nanoparticles through ZnS route. The detailed experimental process is given below. Zinc sulfide nano particles were synthesized by reaction of zinc acetate ($\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$) and thioacetamide (TAA). Zinc acetate was dried in oven at 100°C , firstly. Then, zinc acetate and thioacetamide were milled for 15 min separately. After milling, zinc acetate and thioacetamide were stoichiometrically mixed together and ball mixed for 30 min. Finally, the mixture reacted in the oven at 100°C , 150°C , 200°C and 300°C for 1 hr and zinc sulfide nano particles with different particle sizes were obtained. By annealing zinc sulphide nano particles obtained from the above method in air atmosphere at 600°C , the zinc sulphide nano particles were oxidized into zinc oxide nano particles.

d. Direct synthesis of monodispersed ZnO nano particles in an aqueous solution

Li et al. described a simple route to a direct precipitation of monodispersed ZnO nano particles with mesopores through the transformation of $\text{Zn}(\text{NH}_3)_4^{2+}$ precursor. $\text{Zn}(\text{NH}_3)_4^{2+}$ complexes were used as precursors; sodium oleate and hydrazine were added into the reaction solutions; the size of the sample was effectively controlled and monodispersed ZnO nanoparticles

ZnO nanoparticles was based on the transformation of $\text{Zn}(\text{NH}_3)_4^{2+}$ complexes in the presence of sodium oleate and hydrazine at 80°C . The pH value was maintained at 8.5 through the whole reaction process, which was adjusted by sodium hydroxide. 100ml of 0.38M ZnSO_4 and 0.71M ammonia were mixed and stirred for 10 min to form $\text{Zn}(\text{NH}_3)_4^{2+}$ complexes. Then 0.016 mol H_2NNH_2 and 0.08 g sodium oleate were added into the above solution. The obtained final solution was maintained at 80°C in water-bath to transform $\text{Zn}(\text{NH}_3)_4^{2+}$ complexes and pH value was controlled at 8.5 by 4M sodium hydroxide, along with the release of ammonia from the system. The reaction for the preparation of ZnO using the $\text{Zn}(\text{NH}_3)_4^{2+}$ as precursor according to the following reaction is as follows:



The reaction was carried out for 4 hr. The resulting white precipitates (ZnO) were percolated and washed with distilled water and absolute ethanol for several times and then dried in a vacuum at 60°C overnight.

e. ZnO nanoparticles obtained by mechano-chemical technique

Moballegh et al. reported synthesis of ZnO nanoparticles were synthesized through Mechano-chemical method by using ZnCl_2 , NaCl and Na_2CO_3 as raw materials. The experimental procedure of preparation of ZnO nano particles is given here below: The starting materials were anhydrous ZnCl_2 , anhydrous Na_2CO_3 and NaCl. The NaCl was used as a dilute additive to the starting powder. The stoichiometric mixture of the starting powders was milled, corresponding to the following reaction:



SEM

UNTREATED FABRIC SAMPLES

Figure:3: SEM Untreated silk fabric 500x

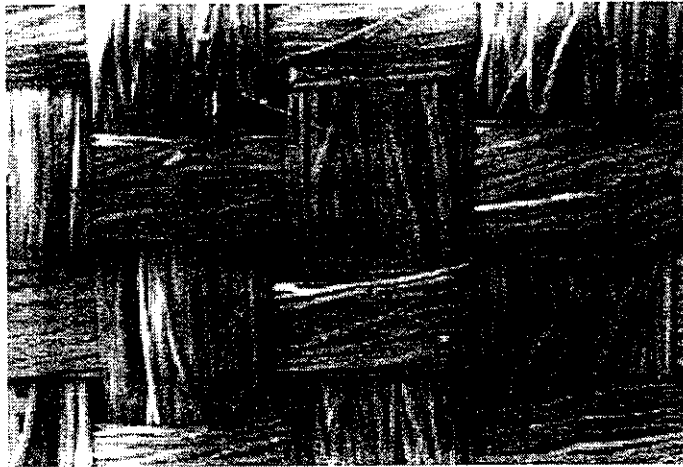


Figure:4: SEM Untreated silk yarn 1000x



Figure 4 shows the untreated single filaments of the silk yarn. The indicators shows the diameters of filaments.

The diameter of balls was 10 mm and the ratio of ball to powder mass was 10:1. Mechano-chemical milling was carried out with planetary mill for 9 h at 250 rpm. Powder was calcined in air at 300, 350, 400 and 450°C for 30 min, respectively.



The samples were washed with distilled water three times. Finally, ZnO nanoparticles were obtained from drying of washed powders.

f. Thermal decomposition of zinc alginate

Baskoutas et al. presented a novel method for the formation of zinc oxide nanocrystals which is based on the thermal decomposition of zinc alginate. Alginate is a biopolymer extracted from brown seaweed. It is a linear heteropolysaccharide composed of D-mannuronic acid and L-guluronic acid. Gelation of alginate is possible by interaction of carboxylate groups with divalent ions. Zinc alginate beads were prepared by dropping aqueous solution of sodium alginate into a zinc solution containing zinc nitrate or zinc acetate. Zinc solution was prepared either from zinc nitrate or from zinc acetate by dissolving the appropriate amounts of the respective salts in ultrapure water (conductivity – $0.1\mu\text{Scm}^{-1}$). Alginate solutions at a concentration of 1% w/w were prepared by dissolving the appropriate amounts of sodium alginate in ultrapure water under magnetic stirring. The Zinc alginate beads were produced by drop wise addition of 10 ml of alginate solution into 20 ml of zinc solution through a 0.49 mm inner diameter stainless steel needle. The formed gel beads were maintained in the gelling medium for 30 min under gentle stirring, and then separated from the solution through a stainless steel grid placed in a

porcelain crucible and heated at 450 or 800°C for 24 h with a heating rate of 10°C/min.

It has been shown that the thermal decomposition of zinc alginate beads resulted in the formation of ZnO nanocrystals. The size of ZnO nanocrystals thus formed is dependent on the burning temperature and the type of zinc cross linking agent. The ability of alginate to absorb metals such as Cu, Cd and Zn can enable us to consider this low cost biopolymer as a potential precursor for the preparation of other technologically important oxides.

g. Spray pyrolysis

A novel aerosol decomposition (spray pyrolysis) process for the continuous synthesis of nano particles as well as the electrospray pyrolysis was reviewed in depth and detail by Okuyama and Wuled Lenggoro. The production of spherical shaped porous silica particles with nano scale ordering porosity by means of a spray drying method using a colloidal mixture as the precursor and zinc oxide nano particles in silica matrix prepared by the combined sol-gel and spray drying method were reported.

h. Sol-gel Route

Though zinc oxide nanoparticles can be prepared by different methods, the sol-gel method is more popular because of its cheapness, reliability, repeatability, and simplicity. Vafae and Ghamsari reported about a novel Sol-Gel route where in triethanolamine (TEA) had been used as a surfactant for the first time. In the preparation of ZnO sols, the following procedure was carried out. First, the appropriate ratio of triethanolamine was added to ethanol. After complete mixing by a magnetic stirrer, zinc acetate

30 min by heating to 50-60°C. The amounts of ZnAc in sols were varied to 0.25, 0.5 and 0.75 M in order to assess the best ratio, and also the molar ratio of TEA to ZnAc was adjusted to 3:5, 6:5 and 9:5 with the same aim.

i. Wet chemical method using ZnCl₂

Alessio Becheri et al reported the successful synthesis of Zinc oxide nanoparticles that were synthesized following a procedure reported elsewhere by (Moroni et al. 2005). The synthesis was carried out a high degree of super saturation, in order to achieve a nucleation rate much greater than the growth rate. ZnCl₂ (5.5 g) was dissolved in 200 mL of water at 90°C in an oil bath. 16 ml of 5M NaOH aqueous solution were added dropwise to the zinc chloride solution with a gentle stirring over a period of 10 min at 90°C. The particles were separated from the supernatant dispersion washed five times with distilled water to lower the concentration of NaCl below 10⁻⁶ M. Each time, the dilution ratio between the concentrated suspension and washing solution was about 1:10. The complete removal of NaCl from the suspension was checked with a solution of AgNO₃. The purified particles were then peptized with 2-propanol in an ultrasonic bath for 10 min at room temperature. The peptization process is necessary to disrupt the micro agglomerates and release the nano units of zinc oxide. The particles were then collected by centrifugation at 6,000 rpm for 15 min. The washing procedure was repeated three times. Thermal treatment of the particles at 250°C for 5h lead to the formation of ZnO. The synthesis in 1,2 –ethanediol (ED) was carried out in the same way, but at 150°C. The ZnO nano particles thus synthesized were used as UV –absorbers, on efficient transfer to fabric materials through the application of ZnO nanoparticles on the surface of cotton and wool fabrics.

j. Wet chemical method using Zinc nitrate

Yadav et al reported the successful synthesis of the zinc oxide nanoparticles which were prepared by wet chemical method using zinc nitrate and sodium hydroxide as precursors and soluble starch as stabilizing agent. Different concentrations of soluble starch (0.1%, 0.5% and 1%) were dissolved in 500 ml of distilled water by using microwave oven. Zinc nitrate, 14.874g (0.1 mol), was added in the solution. Then the solution was kept under constant stirring using magnetic stirrer to completely dissolve the zinc nitrate. After complete dissolution of zinc nitrate, 0.2 mol of sodium hydroxide solution was added under constant stirring, drop by drop touching the walls of the vessel. The reaction was allowed to proceed for 2 h after complete addition of sodium hydroxide. After the completion of reaction, the solution was allowed to settle for overnight and the supernatant solution was then discarded carefully. The remaining solution was centrifuged at 10,000 rpm for 10 min and the supernatant was discarded. Thus obtained nano particles were washed three times using distilled water. Washing was carried out to remove the byproducts and the excessive starch that were bound with the nano particles. After washing, the nano particles were dried at 80°C for the overnight. During drying, complete conversion of $Zn(OH)_2$ into ZnO takes place.

k. Thermal decomposition of a Green single-source inorganic precursor in air

Nowadays, there is obviously an increased emphasis on the topic of green chemistry and chemical processes, which aim at the total elimination or at least the minimization of generated waste and the implementation of

sustainable processes through the adoption of 12 fundamental principles. Any attempt at meeting these goals must comprehensively address these principles in the design of a synthetic route, chemical analysis, or chemical process. Utilization of non-toxic and benign precursor and lowering the reaction temperature (less thermal consumption and less release/radiation) are the key issues that merit important consideration in a “green” solid-state thermolysis synthetic strategy.

Zhang et al. presents simple, cheap, no pollution, low-temperature (180°C)

Thermal decomposition route for the large-scale production of pure ZnO nanocrystallites with the sizes in the range of several nanometers from a “green” single-source inorganic precursor, ZnO₂ nanocrystallites, which were firstly synthesized through the hydrothermal reaction of Zn(Ac)₂ and H₂O₂ in an alkaline aqueous solution (pH=10) at 100°C for 12 h under sealed environment. Obviously, the choice of ZnO₂ nanocrystallites as the special precursor to ZnO. This work has some appealing features: easy preparation, low decomposition temperature, no unwanted by-products and free of pollution to both the target products and the environment upon thermal decomposition ($\text{ZnO}_2 = \text{ZnO} + 1/2\text{O}_2$) in air. All the chemical reagents used in this experiment are of analytical grade. In a typical synthesis, 1.0 g of Zn(Ac)₂·2H₂O powder was dissolved into 40ml of H₂O₂ (3%, v/v) aqueous solution, whose pH value was adjusted to 10.0 with dilute ammonia. The resultant starting solution was transferred into a Teflon-lined stainless steel autoclave of 50ml capacity, sealed and maintained at 100°C for 12 h, then air cooled to room temperature. The as-formed ZnO₂ precursor was filtered, washed with water, and dried in air at 80°C. Finally, the ZnO product could be produced by subjecting the as-made

l. Thermal decomposition of β -Cyclodextrin coated zinc acetate

Yang et al, reported a novel and facile approach to fabricate ZnO nano particles under the relatively simple and mild conditions, in which zinc acetate is coated by β -cyclodextrin (β -CD). Thermal decomposition of zinc salt is one of the versatile ways to obtain ZnO nano materials, in which zinc acetate ($\text{ZnO} \cdot \text{CH}_3\text{COO}$)₂ is often chosen as the precursor for its high solubility and low decomposition temperature. A total of 0.22g (1mmol) ($\text{ZnO} \cdot \text{CH}_3\text{COO}$)₂·H₂O and 2.28g (2mmol) β -CD were stirred for 120 min at room temperature (25°C and then evaporated by decompress at 40°C to remove the water. The resulting solid products were dried in vacuum and ground into powders before use. Then the samples was heat-treated in muffle at 500°C for 1 h in the air and white-coloured ZnO products were obtained.

m. Pulsed laser ablation in aqueous media

Singh and Gopal have reported synthesis of zinc oxide nanoparticles by pulsed laser ablation of zinc target in aqueous media with simultaneous flow of pure oxygen gas. It was reported that the nanoparticles produced by this method are smaller in size with narrower size distribution as compared to that synthesized by other conventional approaches.

Zinc Oxide nanoparticles in solution were synthesized by combination of pulsed laser ablation of a piece of zinc metal in an aqueous media of anionic Sodium dodecyl sulphate, C₁₂H₂₅SO₄Na (Glaxo Smith Kline; denoted as SDS Hereafter with simultaneous flow of pure oxygen. Oxygen gas was flowed

reaches at the center of laser ablated plasma and formation of ZnO is possible. The slice of zinc rod (99.99 %, Spec-pure Johnson Mathey) was placed on the bottom of glass vessel containing 10ml, 0.05 M aqueous solution of SDS. The piece Nd:YAG laser (Spectra Physics inc., USA) operating at 10Hz, using a convex lens to focus to 100 μm spot for 60 min. The position of metal plate was continuously translated mechanically so that each laser pulse falls on the fresh surface and ablates the target surface homogeneously. Upon irradiation with laser beam solution becomes milky-white, showing the formation of colloidal zinc oxide nanoparticles. Larger particles and free SDS, appearing as residues were separated from colloidal solution using 5000g centrifugation. Vacuum evaporation in oven was used to convert ZnO nano particles from solution to powder phase at 60 degree celcius temperature . This physical technique has several advantages over other processes, including the ability to produce materials with complex stoichiometry, narrower particles size distribution , reduced porosity, and control the level of impurities and defects. Particles produced by this method have chemical contamination free surfaces, which are important for further functionalization of bulky groups and application in bioscience. This method provides an alternative, pollution free way for the synthesis of oxides and oxyhydroxide nanoparticles of other metals. Nano particles synthesized by this method have chemical contamination free surfaces, which can be used for biological applications.

The work of Moroni et al. addressed the synthesis and characterization of ZnO nano particles obtained through a homogeneous phase reaction between zinc chloride and sodium hydroxide at high temperature. ZnCl_2 (5.5 g) was dissolved in 200 ml of water at 90°C in an oil bath. 16 ml of 5 M NaOH aqueous solution were added drop wise to the zinc chloride solution, with a

gentle stirring over a period of 10 min at 90°C. The particles were separated from the supernatant dispersion by sedimentation. The supernatant solution was discarded, and the remaining suspension washed five times with distilled water to lower the concentration of NaCl below 10^{-6} M. Each time, the dilution ratio between the concentrated suspension and washing solution was about 1:10. The complete removal of NaCl from the suspension was checked with a solution of AgNO_3 . The purified particles were then peptized with 2-propanol in an ultrasonic bath for 10 min at room temperature. The peptization process is necessary to disrupt the micro agglomerates and release the nano units of zinc oxide. The particles were then collected by centrifugation at 6,000 rpm for 15 min. The washing procedure was repeated three times. Thermal treatments of the particles at 250°C for 5 h lead to the formation of ZnO. The synthesis in 1,2-ethanediol (ED) was carried out in the same way, but at 150°C. The noteworthy feature of the reaction in 1,2-ethanediol at 150°C is that it results in the formation of smaller nano particles with respect to the reaction carried out in water at 90°C. In both cases, the nano particles appear to be nearly spherical and with a quite narrow size range.

n. Solvothermal oxidation route

Chen et al. reported a novel solvothermal oxidation process to prepare hydroxyl-free nano crystalline ZnO using dry solvent under nitrogen atmosphere. The reactions between Zn powder and oxygen source/oxidants here provide a novel solvothermal route to the hydroxyl-free ZnO particles. ZnO nano crystals were prepared by the reactions of zinc powder with trimethylamine N-oxide or 4-picoline N-oxide in different organic solvents [toluene,

ethylenediamine(EDA) or N,N,N',N' -tetramethylethylenediamine(TMEDA)]at 180 degree celcius in an autoclave. In a typical procedure to give 1-10, 2.0 mmol of Zn powder and 4.0 mmol of trimethylamine N- oxide or 4- picoline n- oxide, were placed in a 30.0 ml, Teflonlined stainless steel autoclave in a dry N₂. A solvent [toluene, EDA or TMEDA] was then added upto 70% of the total volume of the autoclave in a glove-box. The sealed autoclave was heated at 180°C for 24, 84, 91 or 100 h, respectively. After cooling gradually to room temperature, a brown precipitate was collected and washed in an ultrasonic bath several times with acetone. The solid was dried in vacuum at 60°C for 3 h.

o. Sonochemical synthesis

Kandjani et al reported wherein ZnO nano particles were synthesized by sonochemical method starting from ZnCl₂ as the source of Zn, reacting with a solution of KOH as the source of oxygen. In this study, the effects of reaction temperature and sonication output power was studied on the purity; particle size and morphology of thr ZnO nano particles were done.

An aqueous ZnCl₂ solution (1M, 100 ml) was added drop-wise to an aqueous KOH solution (2 M, 100 ml) within about 30 min ($[Zn^{2+}]/[OH]=1/2$) in a beaker placed in athermostatic cooling-heating water bath. The resulting solution was kept at different constant temperatures under different constant sonication output powers for 2 h. different ZnO samples were prepared by this simple procedure under different applied conditions. Finally, precipitated water and methanol and then dried at 35°C under vacuum. In theis work, it was shown that the morphology of the particles and their sizes are highly dependent to the reaction temperature and sonication output power. In this study, the effects of reaction temperature and sonication output

power lead to ZnO nano particles with spherical morphology as the principal morphology of the particles.

q. Chemical route

Wu et al. reported about the successful fabrication of ZnO nano particles using a simple chemical approach. It was claimed that this method produced a large quantity of ZnO nano crystals (yield is about 90%) at relatively high purity and very low cost. To prepare ZnO nano particles, the typical synthesis is as follows: the alkali solution of zinc was prepared by dissolving 14.87 g of zinc nitrate $[\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}]$ and 4 g of NaOH in deionized water to form a 100 ml solution $[\text{Zn}^{2+} = 0.5, \text{OH}^- = 1.0 \text{ M}]$. then the NaOH solution was heated to certain temperature . under constant stirring, the zinc nitrate solution was added slowly (drop wise for 30 min) to the above alkali solution. After 2 h reaction, the white precipitate deposited in the bottom of the flask was collected and washed several times with absolute ethanol and distilled water. Finally, the ZnO samples were obtained by centrifugation and dehydration of the precipitate in a vacuum at 60-70°C. It was observed that the morphology of ZnO nano particles seems to change from rod like to short prism- like with increasing reaction temperature. Moreover the ZnO nano particles have a very strong photoluminescence (PL) band at ultraviolet wavelength range.

r. Microwave- Assisted method

Ma et al. reported a simple one- step microwave-assisted aqueous solution method for the synthesis of ZnO with various morphologies using pyridine

at different concentrations. In a typical experiment, 0.297 g of $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (purity >99.0%) and 5 ml of pyridine ($\text{C}_5\text{H}_5\text{N}$, purity > 99.5%) were dissolved in deionized water (30 ml) in a 50ml round-bottomed flask under magnetic stirring. The solution was heated to 90°C and kept at this temperature for 10 min by microwave heating. The microwave oven used was a focused single-mode microwave synthesis system (2.45 GHz, Discover, CEM, USA), which was equipped with the magnetic stirring system and a water-cooled condenser outside the microwave cavity. The product was separated by centrifugation, washed with ethanol several times, and dried at 69°C in vacuum. This method is simple, fast, surfactant-free and low-cost for the preparation of ZnO with various morphologies. It was reported that the basic additive (pyridine, aniline and triethanolamine) has a significant influence on the morphology of ZnO.

s. Levitational gas condensation

The levitational gas condensation (LGC) method is one of the physical approaches to fabricate nano particles. It is a simple one step process for synthesizing nano particles with uniform size distribution. Uhm et al. reported about the Zinc oxide (ZnO) nano particles synthesized by the newly modified LGC and phase evolution. High-purity ZnO powders were synthesized by the LGC method. The apparatus consists of high frequency induction generator of 2.5 KW, levitation and evaporation chamber, and concentration control unit. The wire feeding velocity (V_{Zn}) and mixed Air and O_2 gas pressure in chamber was 50 mm/min and 18 K Pa respectively.

2.3 METHODS OF IMPARTING THE NANOPARTICLES OF METAL OXIDES ONTO THE TEXTILE SUBSTRATES

2.3.1. Pad-dry-cure method

The most widely used method to impart nanoparticles onto the textile substrate. The textile substrate, cut to the size of 30×30 cm was immersed in the solution containing NANOPARTICLES (2%) and ACRYLIC BINDER (1%) for 5 min and then it was passed through a padding mangle, which was running at a *speed* of 15 m/min with a pressure of 15 kgf/cm² to remove excess solution. A 100% *wet pick-up* was maintained for all of the treatments. After padding, the fabric was air-dried and then cured for 3 min at 140°C. The fabric was then immersed for 5 min in 2 g/l of *sodium lauryl sulphate* to remove unbound nanoparticles. Then the fabric was rinsed at least 10 times to completely take out all the soap solution. The fabric thus washed was *air-dried*. Similar method was followed for bulk-ZnO coating also.

2.3.2. Dip-dry method

The textile substrate was conditioned at constant *relative humidity* (33%) and *temperature* (20°C). The fabric sample (10 cm × 10 cm) was soaked for 10 min in a dispersion of *nanoparticles* (5% w/w), under gentle magnetic stirring. The fabric was then squeezed to remove the excess dispersion, and *dried* in a oven at 130°C for 15 min at atmospheric pressure (dry heat). The drying step was carried out on a horizontal flat surface.

2.3.3. Spraying

The nano particles are imparted onto the textile substrate using a hand-held spray gun. This method involves application of nano particles only on the right side of the fabric, whereby excess usage of nano particles can be minimized. A dispersion of nano particles (2%) was filled in a spray gun. The textile substrate was fixed on a vertical board. The nano particle solution was evenly sprayed over the substrate by maintaining a constant distance between the substrate and the spray gun nozzle. The excess dispersion was squeezed using a padding mangle running at a speed of 3 m/min. The fabric was then dried at room temperature on a flat surface.

CHAPTER - 3

AIM & SCOPE

- To impart MULTI-FUNCTIONAL finishing using NANO PARTICLES to produce DURABLE & VERSATILE product.
 - To determine the effect of nature and structure of textile substrate on functional properties.
 - To determine the change in fabric properties on application of nanoparticles.
 - To determine the substrate and nanoparticle duo that exhibits high functional property.
-
- ✓ Multi-functional finishes have a wide scope as they reduce water usage, chemicals and process involved.
 - ✓ Nano technology involves a **bottom up** approach which reduces pollution and energy saving.
 - ✓ Nano particles possess very high surface energy making it more durable than bulk particles.
 - ✓ In future, nano particles play a vital role in durable finishing.
 - ✓ Nano technology is a eco-friendly process.

CHAPTER - 4

METHODOLOGY

4.1 YARN PROCUREMENT

The 100% mulberry silk yarn was procured for the fabric manufacture. Then the yarn was degummed and further processed to make the yarn suitable for fabric manufacture.

4.2 FABRIC MANUFACTURE

Fabrics with two commonly used structures WOVEN, KNITTED were manufactured using sample weaving machine and 30 feeder knitting machine respectively. The type and specification of fabric are as follows,

Table:2: Woven structure

| WOVEN STRUCTURE | |
|-----------------|-----|
| Ends/inch | 120 |
| Picks/inch | 96 |
| Width | 24" |
| Gsm | 55 |
| | |

Table:3: Knitted structure

| KNITTED STRUCTURE | |
|--------------------------|---------------|
| Gauge | 28 |
| Dia | 26" |
| Gsm | 65 |
| Structure | Single jersey |

The Gsm and structure of the fabric were selected based on commonly used configuration for silk apparels.

4.3. SYNTHESIS OF NANO ZINC OXIDES

The zinc oxide (ZnO) nano particles were prepared by wet chemical method using zinc nitrate and sodium hydroxide as precursors and soluble starch as stabilizing agent.

- Initially soluble starch(0.5%) was dissolved in 500 ml of distilled water by using microwave oven.
- Next, add Zinc nitrate 14.874(0.1 mol) to above solution
- Then the solution was kept under constant stirring using magnetic stirrer to completely dissolve the zinc nitrate.
- After complete dissolution of zinc nitrate, 0.2 mol of sodium hydroxide solution was added under constant stirring, drop by drop touching the walls of the vessel.
- The reaction was allowed to proceed for 2 h after complete addition of sodium hydroxide.
- After the completion of reaction, the solution was allowed to settle for overnight and the supernatant solution was then discarded carefully .
- The remaining solution was centrifuged at 10,000 x g for 10 min and the supernatant was discarded.
- Thus obtained nano particles were washed three times using distilled water.
- Washing was carried out to remove the byproducts and the excessive starch that were bound with the nano particles.
- After washing, the nano particles were dried at 80°C for overnight.
- During drying, complete conversion of Zn (OH)₂ into ZnO takes place.

4.4. APPLICATION OF NANO PARTICLES ONTO TEXTILE SUBSTRATE

Spraying method was used to apply the nano particles onto the textile substrate. This method involves application of nano particles only on the right side of the substrate. As the functional properties exhibited by the nano particles involve only surface phenomena, it is not needed to apply nano particles even on rear side of the fabric. Hence, spray technique proves to be more effective in producing functional effect using less quantity of nano particles.

A dispersion of nano particles (2%, M:L ratio 1: 20) was filled in a hand-held spray gun. The textile substrate (10cm × 10cm) was fixed on a vertical board. The nano particle solution was evenly sprayed over the substrate by maintaining a constant distance between the substrate and the spray gun nozzle. The excess dispersion was squeezed using a padding mangle running at a speed of 7 m/min at 150°C. The air pressure was maintained at 7 kg/cm². The fabric was then dried at room temperature on a flat surface.

4.5. TESTING

The testing involved two types of tests namely,

- a. CHARACTERISATION TEST,
- b. FUNCTIONAL TEST.

The characterization test is used to characterize the nano particles composition, their shape, size and crystallinity. It involves following tests,

- Fourier transformed infrared spectroscopy(FTIR)

- Scanning electron microscopy(SEM)
- Transmission electron microscopy(TEM)

The CHEMICAL COMPOSITION of the synthesized materials was checked by FTIR spectroscopy with a Biorad FTS-40 spectrometer.

The CRYSTALLINITY was determined by XRD using a Bruker D8 Advance Xrays Diffractometer equipped with a Cu Ka ($k = 1.50 \text{ \AA}$) source (applied voltage 40 kV, current 25 mA). About 0.5 g of the dried particles were deposited as a randomly oriented powder onto a Plexi glass sample container, and the XRD patterns were recorded at angles between 3° and 80° , with a scan rate of $6^\circ/\text{min}$. The crystalline domain diameters (D) were obtained from XRD peaks according to the **Scherrer's** equation

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

Where,

k is the wavelength of the incident X-ray beam (1.54 \AA for the Cu Ka),

h is the Bragg's diffraction angle,

ΔW is the width of the X-ray pattern line at half peak-height in radians.

The SHAPE & SIZE of the particles were obtained through TEM, using a Philips EM201C apparatus operating at 80 kV. 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.

The DISTRIBUTION of nano particles on the surface of substrate was

Table : 4: Test centers

| TEST | TEST CENTER |
|-------------|---|
| FTIR,XRD | CUSAT,COCHIN |
| SEM | PSG COLLEGE OF TECHNOLOGY,COIMBATORE |
| TEM | NORTH EASTERN HILL UNIVERSITY,SHILLONG |

The UV-Screen properties (functional testing) of the treated fabrics were investigated by absorption spectroscopy using a **uv-vis spectrophotometer** (Perkin-Elmer Lambda, equipped with a 60-mm integrating sphere). The blank reference was air. The UV profiles of the untreated samples were compared to the spectra collected from the same fabrics treated with nano particles, and the effectiveness in shielding UV radiation was evaluated by measuring the UV absorption, transmission and reflection. Each measurement is the average of four scans obtained by rotating the sample by 90°. The transmission data were used to calculate the UPF (ultra violet protection factor) and the percent UV transmission, according to the following equations,

$$UPF = \frac{\int_{\lambda_1}^{\lambda_2} E(\lambda).S(\lambda)d\lambda}{\int_{\lambda_1}^{\lambda_2} E(\lambda).S(\lambda).T(\lambda)d\lambda}$$

$E(k)$ is the relative erythemal spectral effectiveness, $S(k)$ is the solar spectral irradiance in $W m^{-2} nm^{-1}$ and $T(k)$ is the spectral transmission of the specimen obtained from the UV spectrophotometric experiments. The values of $E(k)$ and $S(k)$ were obtained from the National Oceanic and Atmospheric Administration database (NOAA). The UPF value was calculated for UV-A in the range 315-400 nm, and for UV-B in between 295 and 315 nm. The percent UV transmission, was determined for UV-A and UV-B radiation from the transmission spectra of the fabric samples.

FUNCTIONAL TESTS

ANTIMICROBIAL TEST

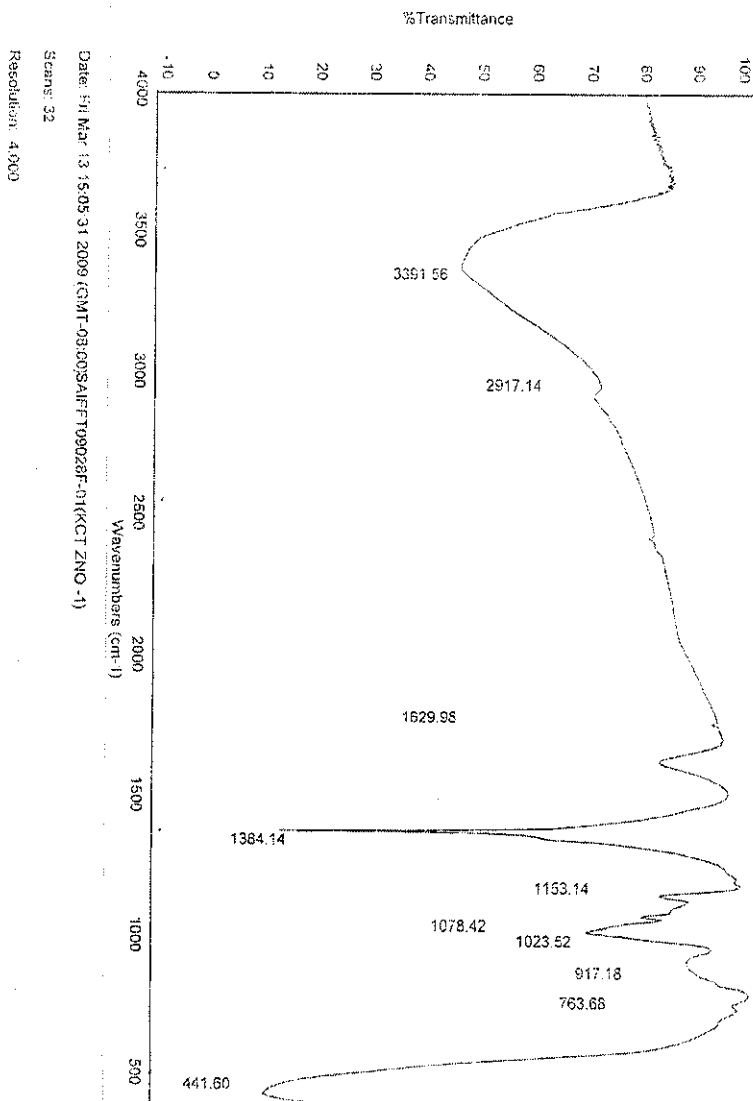
- ✓ Prepare nutrient broth in two sets of conical flasks.
- ✓ Place the treated and untreated samples in each flask.
- ✓ Keep the flasks for overnight in shaker to allow the micro organism to grow in media.
- ✓ Then the optical density reading is taken at 660 nm.
- ✓ These readings are noted to find the growth of micro organism.

CHAPTER - 5

RESULTS AND DISCUSSION

FTIR

Figure :1: FTIR Graph



- In the spectrum of ZnO nanopowders, the absorption band at 441 cm^{-1} indicated the presence to Zn-O
- The broad peak between 3391.56 cm^{-1} was due to the stretching vibrations of the -OH group on the surface of ZnO nanoparticles
- As can also be seen in Fig.1, the peaks at 763 cm^{-1} is the special absorption for one hydrogen atom on benzene ring substituted by other function groups
- The absorption peaks at 1629.98 cm^{-1} should belong to $\text{C}=\text{O}$. The peak at 1153.14 cm^{-1} was assigned to the stretching vibrations of the -C-O-C- asymmetric group

XRD

Figure : 2: XRD Graph

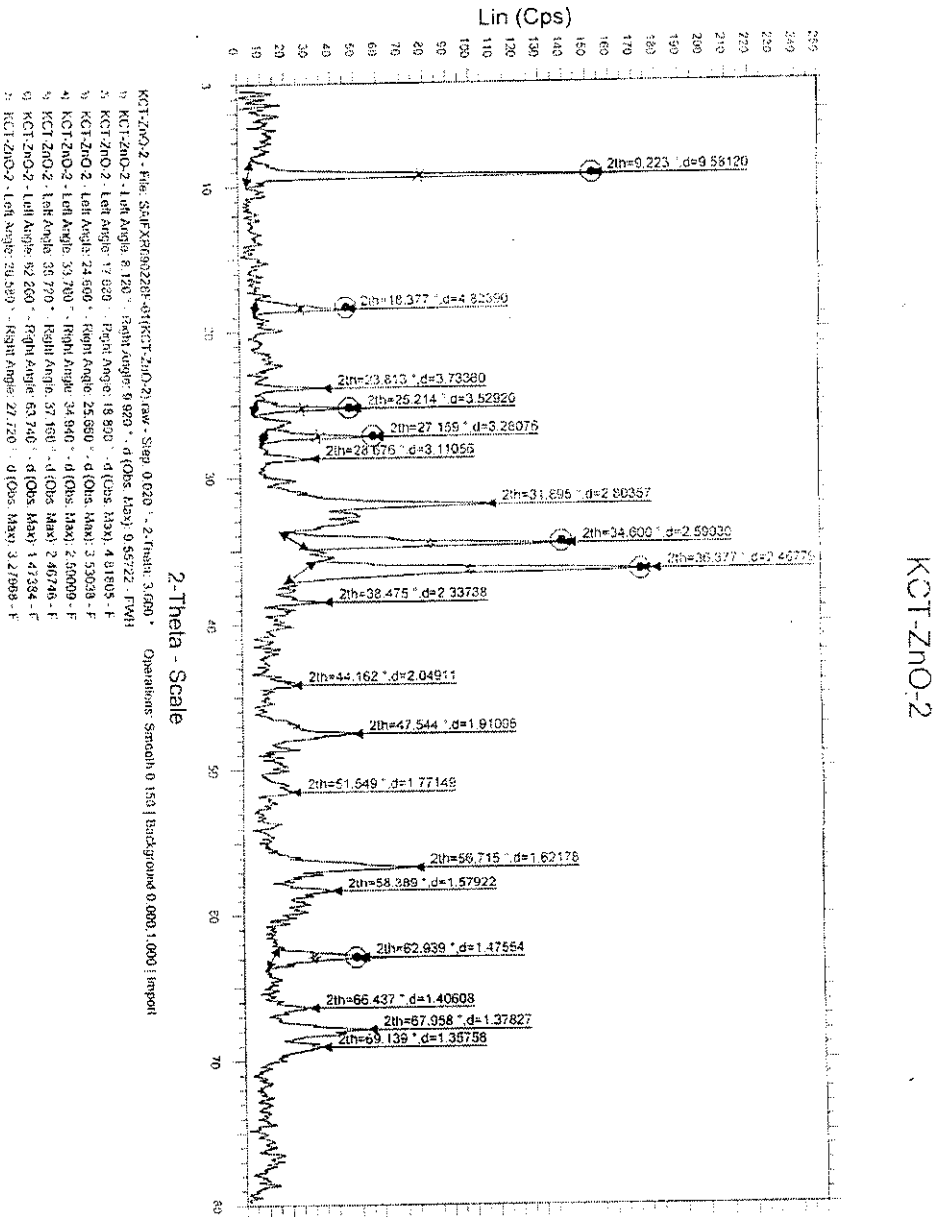


Figure 2 Shows the XRD pattern of ZnO nanocrystals. The diffraction peaks indicate the nano crystalline nature. These peaks at scattering angles (2θ) of 31.890, 34.60, 36.37, 47.544, 56.715, 62.939, 67.958, and 69.139 correspond to the reflection.

SCHERRER'S EQUATION

Crystalline domain diameter

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

The data required for the above formula are obtained from XRD results

Eg:

$$\lambda = 1.5$$

$$2\theta = 36.377^\circ$$

$$\Delta W = 3.9 \text{ radians}$$

From XRD result of nano zinc oxide its size is calculated as **~50nm**

TREATED SAMPLES

WOVEN

Figure:5: SEM Treated woven silk fabric 500x

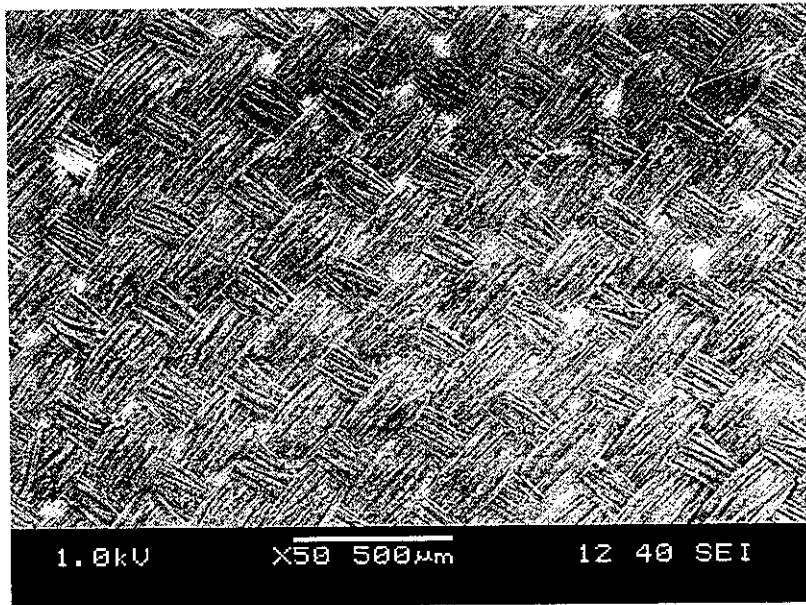
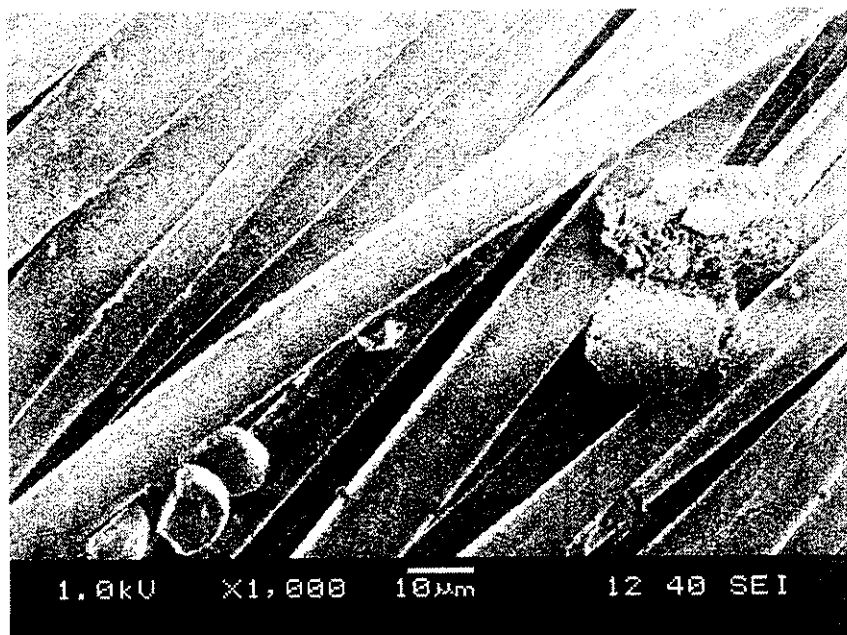


Figure:6: SEM Treated woven silk fabric 1000x



KNITTED

Figure:7:SEM Treated knitted silk fabric 1000x

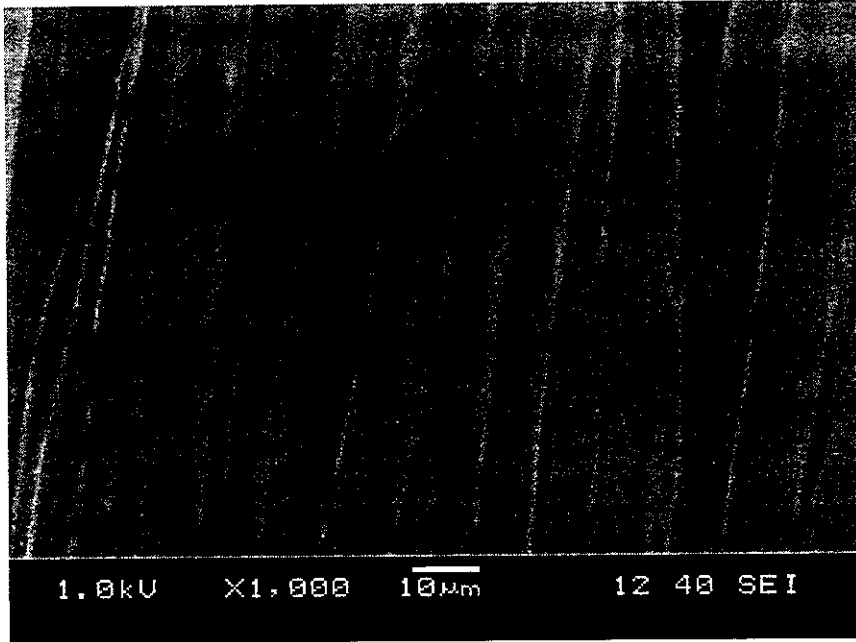


Figure:8:SEM Treated knitted silk fabric 500x

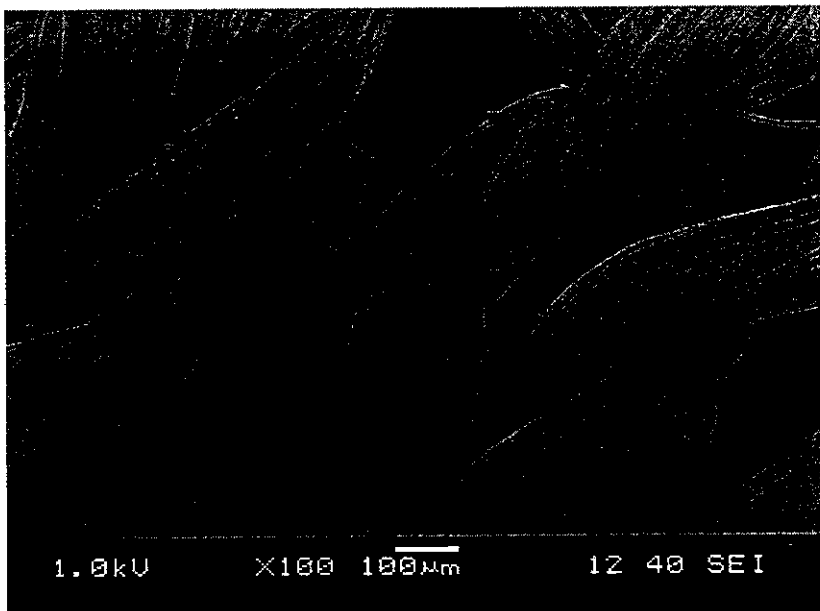


Figure 7 and 8 shows the presence of nano particles on the surface of knitted

FUNCTIONAL FINISH RESULTS

ANTIMICROBIAL

Table : 5: Optical density value

| Fabric sample | Optical density Value |
|----------------------|------------------------------|
| Untreated woven | 0.28 |
| Treated woven | 0.15 |
| Untreated knitted | 0.26 |
| Treated knitted | 0.11 |

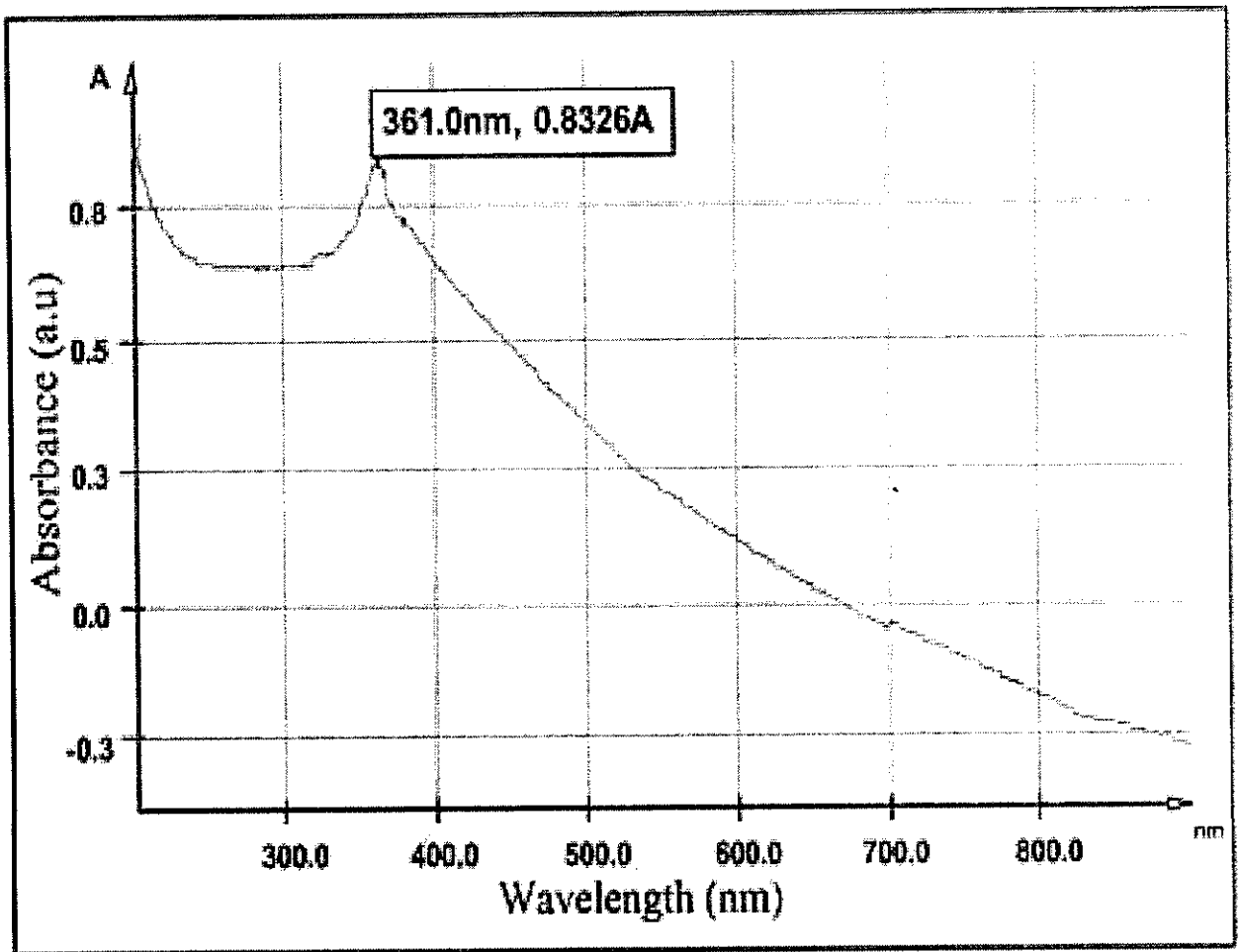
INFERENCE

The microbes growth in fabric is indicated by using Optical density value. The above table values shows that the growth of microbes are controlled more than 50% in both the woven and knitted fabrics.

UV-VIS SPECTROMETRY RESULTS

SYNTHESIED NANO ZINC OXIDE

Figure:9: UV absorbance graph of nano zinc oxide



This graph shows the absorbance value of synthesized nano particles. The peak value indicates that the UV absorbance is high at 361nm.

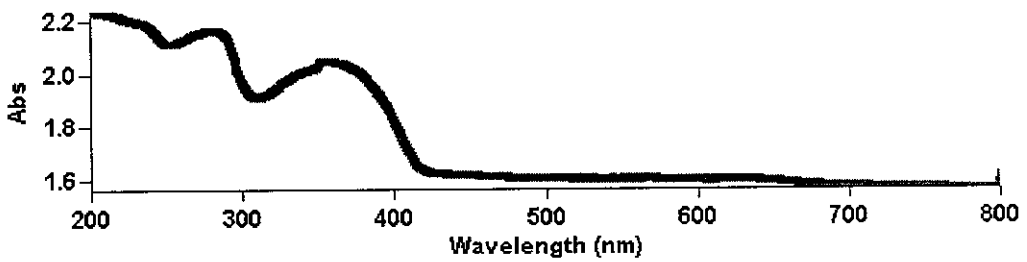
TREATED WOVEN FABRIC

Sample Name: SAIFUV090330A-01(ZnO1) abs

Collection Time 4/3/09 11:31:22 AM
Peak Table
Peak Style Peaks
Peak Threshold 0.0100
Range 800.000nm to 200.000nm

| Wavelength (nm) | Abs |
|-----------------|-------|
| 352.000 | 2.048 |
| 280.000 | 2.167 |

Figure: 10:UV absorbance of treated woven fabric



TREATED KNITTED FABRIC

Sample Name: SAIFUV090330A-02(ZnO₂) abs

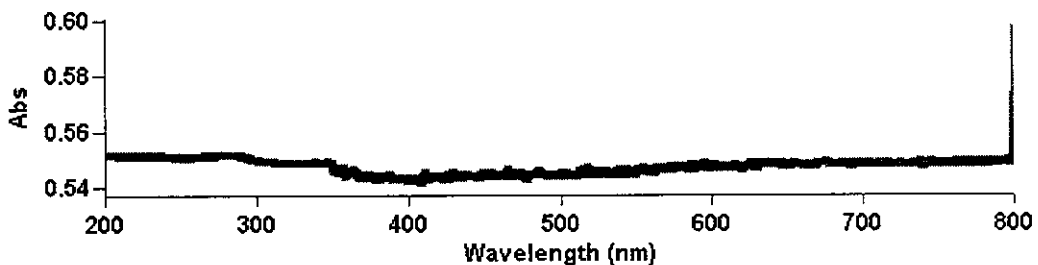
Collection Time 4/3/09 11:38:29 AM

Peak Table

| | |
|----------------|------------------------|
| Peak Style | Peaks |
| Peak Threshold | 0.0100 |
| Range | 800.000nm to 200.000nm |

No peak found above threshold

Figure:11: UV absorbance of treated knitted fabric



INFERENCE

The figures 10&11 shows the UV absorption of treated woven and knitted fabric respectively. From the above graphs, its clear that the UV absorption of ZnO treated fabric is increased. Woven fabric haven shown more UV absorption than the knitted one.

CHAPTER - 6

CONCLUSIONS

- ❖ Knitted structure exhibit better control over the microbes growth.
- ❖ Woven fabrics show more UV absorption than knitted structure due to its compactness and high cover factor.
- ❖ SEM analysis shows the nano particles are well seated on the surface of woven fabrics than knitted fabrics.
- ❖ Although the nano ZnO finishes doesnt give excellent functional properties,the results confirm that the UV protection and antimicrobial properties are improved to an accountable extent. These effect can be enhanced by selecting a more advanced methods like computerised machine spraying techniques.
- ❖ As the nano zinc oxide has good results over microbes control, it may also have the property of self cleaning as they both work on principle of photo catalytic oxidation
- ❖ The nano zno coating may enhance the fabric better strength properties, air permeability and anti-friction property.

Thus, our project work shows that silk fabric treated with nano Zinc Oxide exhibit good ultra-violet protection and anti microbial property, by using spray method for application of nano particles.

CHAPTER - 7

REFERENCES

- Yadav A, Prasad V, Kathe AA, Raj S, Yadav D, Sundaramoorthy C, Vigneshwaran N (2006) “ **Functional finishing in cotton fabrics using zinc oxide nanoparticles**” , Bull Mater Science
- Alessio Becheri, Maximilian Durr , Pierandrea Lo Nostro , Piero Baglioni (2007) “**Synthesis and characterization of zinc oxide nanoparticles: Application to textiles as UV-absorbers**”, Springer Science Business Media.
- Yadav A, Prasad V, Kathe AA, Raj S, Yadav D, Sundaramoorthy C, Vigneshwaran N (2006) “oxide nanoparticles **Functional finishing in cotton fabrics using zinc**” , Bull Mater Science
- Tang E, Cheng G, Ma X, Pang X, Zhao Q (2006) “**Surface modification of zinc oxide nanoparticle by PMAA and its dispersion in aqueous system**” , Application Surface Science
- Y.Li, N.H.Lee, E.G.Lee , J.S.Song, S.J.Kim,(2004) Chem.Phys.Lett.
- C.S.Fang, Y.W.Chen(2003) “Materials Chemistry and Physics”
- R. Inaba, T.Fukahori, M.Hamamoto, T. Ohno (2006) “ **Journal of Molecular Catalysis A: Chemical**”
- H.Changa, S.J. Kima, H.D.Jang, J.W.Choi “**Colloids and Surface A: Physicochem.Eng.Aspects**”
- A new dimension to textiles/garments – Pradeep kaira.
- Selected Applications of Nanotechnology in textiles, Y.W.H.Wong et al.. Autex Research Journal, Vol 6,No 1, March 2006.
- Nanotechnology in Textile Finishing. State of Art and Future Prospects, Jan

- Self cleaning suits may be future , Mark T.Sampson.
- http://www.snaimpex.com/a_new_dimension_to_textiles.htm.
- http://www.nees.uni-bonn.de/lotus/en/faq_html.html
- <http://www.voyle.net/nano%20textiles/textiles-2004-2006.htm>
- http://www.itc.gov.hk/innotecl/IFT_R&D_centre_Conference.pdf
- <http://www.nanomat.de/pdf/nanovision-beringer.pdf>