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**FUNCTIONAL FINISHING OF VISCOSE
POLYESTER BLEND USING
TITANIUMDIOXIDE NANOPARTICLES**

A PROJECT REPORT

SUBMITTED BY

- 1. N.SAMPATH KUMAR (71205212031)**
- 2. S.SATHESH KUMAR (71205212035)**
- 3. K.SOWMYA (71205212045)**
- 4. M.UMA MAGESHWARI (71205212051)**

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**BACHELOR OF TECHNOLOGY
IN
TEXTILE TECHNOLOGY**

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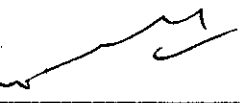
ANNA UNIVERSITY: CHENNAI 600025

APRIL 2009

ANNA UNIVERSITY: CHENNAI 600 025

BONAFIDE CERTIFICATE

certified that this project report "STUDY OF FUNCTIONAL FINISHING ON VISCOSE POLYESTER BLEND USING TITANIUM DIOXIDE NANOPARTICLES" is the bonafide work of N.SAMPATH KUMAR, S.SATHESH KUMAR, K.SOWMYA, M.UMA RAMESHWARI" who Carried out the project work under supervision.



SIGNATURE

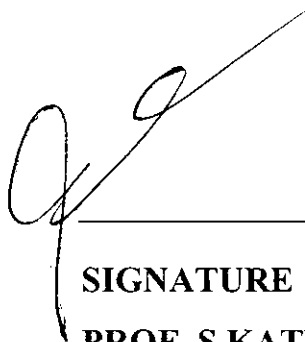
DR. LOUIS D'SOUZA

PROFESSOR AND HEAD

DEPARTMENT OF TECHNOLOGY

KUMARAGURU COLLEGE OF TECHNOLOGY

COIMBATORE - 641 006



SIGNATURE

PROF. S.KATHIRVELU

SUPERVISOR

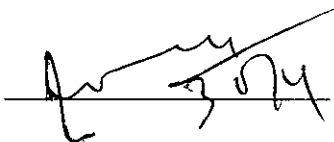
Asst. PROFESSOR

DEPARTMENT OF TEXTILE TECHNOLOGY

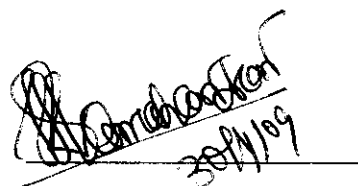
KUMARAGURU COLLEGE OF TECHNOLOGY

COIMBATORE - 641 006

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ABSTRACT

The project work was done in an attempt to study the functional finishing of textile fabrics using metal oxide nano particles. The textile fabrics chosen for the study were woven and knitted fabrics made of POLYESTER VISCOSE blends(65/35 AND 35/65). TiO_2 nano particles were synthesized using SOFT CHEMISTRY. The four types of fabric samples were impregnated with the nano particles of TiO_2 using SPRAYING technique .The finished fabric was done characterization test using SEM photo micro graphs. The nano particles synthesized were characterized using FTIR & XRD techniques. The finished fabrics were tested for their ultra-violet protection characteristics using UV- Vis spectrophotometer method. The various test results were recorded, studied,analyse and reported using the functional testing results, the efficiency of the functional finishing was studied and reported. The results of the various tests were analysed and discussed. Finally, we have arrived at conclusions of this study. The further scope of the project have also been reported.

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SYMBOLS AND ABBREVIATIONS

TiO_2	- Titanium dioxide
TiCl_4	- Titanium Tetrachloride
HNO_3	- Nitric acid
O_2	- oxygen
NaOH	- Sodium Hydroxide
SPF	- Solar Protection Factor
HPC	- Hydroxy Propyl Cellulose
TTIP	- Tetra Isopropoxide
PPI	- Picks per inch
EPI	- Ends per inch
TEOT	- Tetraethylorthotitanate
HT	- High Temperature
FSP	- Flame Spray Pyrolysis
UPS	- Ultra Violet Protection Factors
PEG	- Polyethylene Glycol
FTIR	- Fourier Transform Infrared Spectroscopy
XRD	- X-ray diffraction
SEM	- Scanning Electron Microscopy
μ	- Micron
λ	- Wavelength
θ	- Angle
Dia	- Diameter

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CHAPTER -1

INTRODUCTION

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 emphasized 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 individual'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.

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

production is also less. Thereby, a product that is more economical and eco-friendly can be produced using this method.

NANO FINISHING

The finishing given to clothing should be durable, in order to make the clothes serve its purpose for a longer period of time. This level of durability for special properties can be obtained only using NANOFINISHING. 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 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 fabrics. 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 a SATISFACTORY and HIGHLY DURABLE material.

What is Photocatalysis?

When a semiconductor material is illuminated with ultra band gap light, it becomes a powerful redoxcatalyst capable of killing bacteria, cleaning surfaces, and even splitting water to give hydrogen and oxygen.

Photocatalytic Reaction Mechanisms

Initial reactions



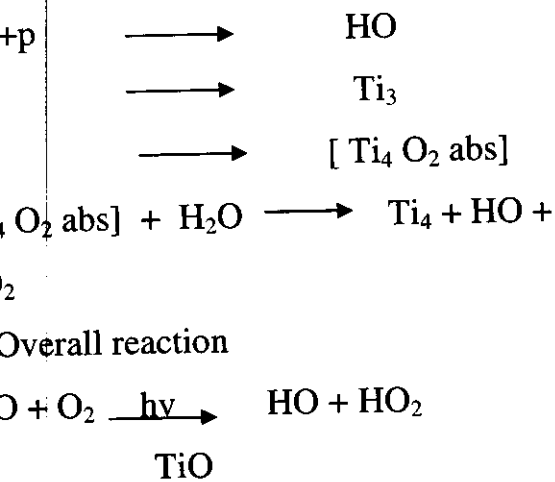
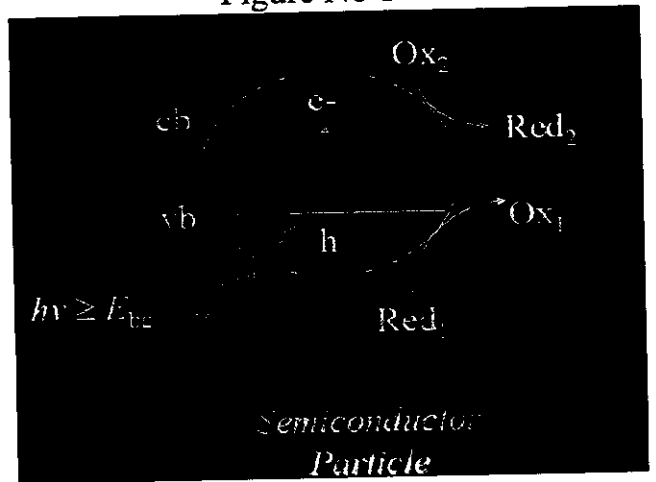


Figure No 1



Titanium dioxide is a photocatalyst, once it is illuminated with energy higher than its band gap, the electrons in TiO_2 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 into $\text{O}_2^{\bullet -}$ - the positive electric holes and water will generate hydroxyl radicals. Since both are unstable chemical substances, when the organic compound adsorbs on the surface of the photocatalyst it will combine with $\text{O}_2^{\bullet -}$ and OH^\bullet respectively, and turn into carbon dioxide (CO_2) and water (H_2O). Through the reaction, the photocatalyst is able to decompose common organic matters in the air such as odour molecules, bacteria and viruses. It was determined that a fabric treated with nano- TiO_2 could provide effective protection against bacteria and the discoloration of stains, due to the photocatalytic activity of nano- TiO_2 . On the other hand, zinc oxide is also a photocatalyst, and the photocatalysis mechanism is similar to that of titanium dioxide; only the band gap is different from titanium dioxide. Nano- ZnO provides effective photocatalytic properties once it is illuminated by light, and so it is employed to impart anti-bacterial properties to

SCHEMATIC DIAGRAM SHOWING PHOTOCATALYTIC REACTION

CHAPTER-2

REVIEW OF LITERATURE

OVERVIEW OF FUNCTIONAL FINISHING OF TEXTILES

ANTI-BACTERIAL FINISHING

Anti-bacterial finishes can be divided into two types based on the mode of action on microbes. One type consists of chemicals that can be considered to act 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 'reservoir' type of anti-microbial can be very effective against microbes on the 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. 'Surface' 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 enzymes, reaction with the cell membrane to the destruction of the cell walls and poisoning the cell from within.

When photocatalytic metal oxides are illuminated by light with energy greater than its band gaps, the electrons 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 into O_2^- the positive electric hole and water will generate hydroxyl radicals. Since both are unstable chemical species, when the organic compound falls on the surface of the photocatalyst, it will combine with O_2^- and OH^- respectively, and turn into carbon dioxide and water.

This cascade reaction is called 'OXIDATION – REDUCTION', and the mechanism is shown below. Through the reaction, the photocatalyst is able to decompose common organic matters in the air such as odour molecules, bacteria and viruses.

SOIL RELEASE FINISHING

Removal of soils from fabrics have been attributed to several mechanisms. They are:

Adsorption of detergent and absorption of water leading to:

Penetration of soil-fibre interface by wash liquid

Solubilisation and emulsification of soils.

Mechanical work leading to:

Hydrodynamic flow carrying away the removed soil

fibre flexing to force soil from between fibres

Surface abrasion to remove soil physically

Swelling of finish to reduce inter-fibre spacing.

Of these mechanisms, solubilisation and emulsification of soils are controlled by detergent composition, hydrodynamic flow is controlled by washing machine design and fibre flexing is controlled by fabric construction. The textile chemist can only influence the mechanisms that involve the fibre surface, i.e. removal of oily soil, penetration of soil-fibre interface, surface soil is removed from fibres by a two-step process.

First, a thin layer of wash liquid penetrates between the particle and the fibre surface, enabling surfactants to adsorb onto the particle surface. Then, the particle becomes solvated and is transported away from the fibre and into the bulk of the wash liquid by mechanical action. Finishes that are hydrophilic (enhancing penetration of the fibre soil interface) with low adhesion to soil under washing conditions should improve particulate soil release. Ablative or sacrificial finishes that leave the fibre surface during washing and take the soil particles along with them can also benefit particulate soil release.

For the most part however, the removal of particulate soils is determined by detergent composition and mechanical action.

A low fibre-wash liquid interfacial energy is desired, that is a hydrophobic finish is preferred for spontaneous oil roll up.

A high fibre-oil interfacial energy is desired, that is the finish should also be

oleophobic.

A low interfacial tension between the oil and the wash liquid will favour oily soil release.

Additional finish characteristics that should facilitate oily soil release include flexibility of the finish to swell during the washing process and flexibility under mechanical action. As in particulate soil release, sacrificial finishes are expected to benefit oily soil release.

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 treatments, 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 No 1

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

Solar protection factors (SPF) of undyed fabrics

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.

$$\text{Solar Protection Factor max} = \frac{1}{1 - \text{cover factor}}$$

Figure illustrates the relationship between the maximum SPF and the 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, 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 in a deep shade can achieve SPF values of 50 or higher just from the presence of

ne dye. Since fashion and comfort often dictate the use of lightly coloured fabrics to provide the desired SPF values in light shades. 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 particle are more effective in scattering of UV rays than bulk particles, by which an enhanced UV protection can be provided

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).

➤ 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 OVERVIEW OF SYNTHESIS OF TiO_2 NANO PARTICLES

2.1. SYNTHESIS OF TITANIUM DIOXIDE NANO PARTICLES

Titanium dioxide (TiO_2) is one of the most popular and promising materials in photo catalytic application due to its strong oxidizing power, high photo stability and redox selectivity. TiO_2 is commercially available and easy to prepare in the laboratory. Titania (TiO_2) has three main polymorphs viz. ANATASE, RUTILE and BROOKITE. Among the three kinds of crystal structure of TiO_2 , commercially available anatase TiO_2 nano particles are the most active for photo catalysis. The anatase and brookite phases are thermodynamically metastable and can be transformed exothermally and irreversibly to the rutile phase at higher temperatures. The transition temperatures reported in the literature range from 450 to 1200⁰ C. The transformation temperature depends on the nature and structure of the precursor and the preparation conditions. Thus, rutile is the most thermodynamically stable phase. Due to its chemical stability, biocompatibility, photo catalytic reactivity, strong oxidizing power and low cost, titania has extensive applications as photo catalysts, gas sensors, self cleaning surfaces, water and air purification components and pigments. In addition, rutile structure has a band gap energy of about 3.0 eV, high dielectric constant, electrical resistivity and refractive index which makes it a relevant choice for dye-sensitized solar cells (DSCs) and capacitors.

Nano crystalline materials are currently receiving much attention by virtue of their special chemical, physical, and mechanical properties. Of these materials, nano sized titanium dioxide has been of great interest because it exhibits many modified electronic and optical properties as well as extensive applications. Nano materials with an average grain size less than 100 nm have been attracting much attention because they exhibit unique and improved mechanical, electrical, optical, and chemical properties compared with conventional polycrystalline materials.

Physical properties should be fully controlled. Titanium oxide (TiO_2) nanoparticles are important because of their outstanding chemical and physical properties, which are useful in many applications such as photovoltaic cell, gas sensors, catalysts, photo catalysts and pigments, etc. The titania particles must fulfill a wide range of requirements such as particle size, size distribution, morphology, crystallinity and phase, etc. so as to become useful in applications. The synthesis of nano-size TiO_2 particles with well-defined physical and chemical properties is a crucial step in nano technology area.

A variety of methods have been developed for the synthesis of TiO_2 nanoparticles, for example, sol-gel process, hydrothermal methods, solvothermal methods and emulsion precipitation.

Many novel methodologies for synthesizing nano sized TiO_2 have been investigated, such as oxidation of titanium tetrachloride in a modified diffusion flame reactor, crystallization in reverse micelles or in supercritical CO_2 , photo assisted sol-gel method and polymer templating method. The different synthetic

processes usually result in the as-prepared nano sized TiO_2 with distinct physiochemical properties. Some of these novel methods using wet-chemistry have also been developed for the preparation of powders with spherical shape and uniform size. Sol-gel process, emulsion and pyrolysis have been used to prepare mono-dispersed spherical titania powders.

Although TiO_2 nanoparticles have been prepared successfully via these methods, most of the nanoparticles synthesized via traditional routes are poorly crystalline and/or exhibit a broad size distribution. For instance, sol-gel derived TiO_2 nanoparticles are induced crystallization. However, the heat treatment frequently leads to particle agglomeration and size change, which is harmful to nano sized TiO_2 , is mainly by

the sol-gel method, it has a main disadvantage that costly organic solvents are required. Several aqueous -based methods using metal salts as a precursor material, such as forced hydrolysis method and homogeneous precipitation method, have several shortcomings: the concentration of reacting species is too low and the reaction time is very long. For synthesis of anatase TiO_2 nanocrystalline, much attention has been paid to hydrothermal methods using amorphous TiO_2 , TiCl_4 or TiOCl_2 aqueous solution, and sol-gel methods using titanium alkoxides, TiO_2 can be also obtained by hydrolysis of titanium compounds, such as titanium tetrachloride or titanium alkoxides, in solution.

HT

As for anatase TiO_2 nanocrystalline, much attention has been paid to hydrothermal methods using amorphous TiO_2 , TiCl_4 , or TiCl_2 aqueous solution, and sol-gel methods using titanium alkoxides, TiO_2 can be also obtained by hydrolysis of titanium compounds, such as titanium tetrachloride or titanium alkoxides, in solution.

The different synthetic processes usually result in the as prepared nanosized TiO_2 with distinct physiochemical properties. Organic modifiers, such as hydroxyl group-containing, carboxylate group -containing, and amine group -containing organics are widely used in synthesis of nanosized TiO_2 to regulate their final morphology and hence their properties. The basis of such methods is the employment of organics with different types of functional groups to produce a amorphous and further calcinations are generally required to desired product with the required shapes and interface structures of organic modifiers with crystal surface

ANATASE CA

A variety of methodologies for synthesizing nanosized TiO_2 have been investigated, such as oxidation of titanium tetrachloride in a modified diffusion flame reactor, crystallization in reverse micelles or in supercritical CO_2 , photo-assisted sol-gel method, polymer templating method, and hydrothermal method. Titanium iso-propoxide, titanium tetrachloride, titanium(IV) sulfate, and amorphous titanium dioxide are commonly used as starting materials in the above-mentioned synthetic processes. The different synthetic processes usually result in the as-prepared nanosized TiO_2 with distinct physiochemical properties.

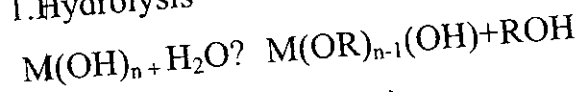
Nanocrystalline anatase titanium oxide (TiO_2) particles are important because of their outstanding chemical and physical properties, which are useful in many applications such as photovoltaic cell, gas sensors, catalysts, photo catalysts and pigments, etc.

The titania particles must fulfill a wide range of requirements such as particle size, size distribution, morphology, crystallinity and phase, etc. so as to become useful in applications. The synthesis of nano-size TiO_2 particles with well-defined physical and chemical properties is a crucial step in nano technology area. However, the next most important aspect is how to arrange them into well defined assemblies/structures or porous aggregates possessing unique properties like pore size and its distribution, thermal stability or structures and their reproducibility, etc. In self-assembly processes, interactions between the primary building blocks are controlled in such a way that at ambient conditions, nanostructures with useful and unique properties are formed. The additional advantage is that these structures have novel number or reports are available on the synthesis or nanoparticles of TiO_2 -based materials.

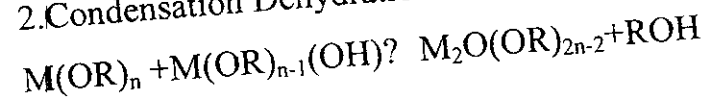
Sol-gel method

Sol-gel is one of the most successful techniques for preparing nanosized metallic oxide materials with high photo catalytic activities. By tailoring the chemical structure or primary precursor and carefully controlling the processing variables, nanocrystal line products with very high level of chemical purity can be achieved. In sol-gel processes, TiO_2 is usually prepared by the reactions of hydrolysis and poly condensation of titanium alkoxides, $\text{Ti}(\text{OR})_n$ to form oxopolymers, which will then transformed into an oxide network. The reaction scheme is usually written as follows:

1. Hydrolysis



2. Condensation Dehydration



To obtain homogeneous titanium oxide networks for application, control of hydrolysis is essential. Therefore, in addition to the precursor for titanium source, some of the chelating reagents, such as diol, carboxylic acid or diketone compounds are added. The condensation is usually accomplished by gelization and calcination. Condensation pulls together the constitute particles of the gel into a compact mass, thus building up the metal oxide crystal. Calcination temperature, on the other hand, is especially important for removing the organic molecules from the final products and completing the crystallization. However, very high calcinations temperature results in aggregation and/or phase transformation, and affects the microstructures as well as the properties of TiO_2 nanoparticles. It has been established that the photocatalytic activity of TiO_2 strongly depends all its metal structure. For instance, anatase -phase TiO_2 crystallites are generally found to be more active

anatase in few examples, such as photodecomposition of H_2S and photo-oxidation of H_2O with Fe^{3+} . Recently, it was found that anatase/rutile mixture (7/3) made the best photocatalyst for the oxidation of organic materials in the wastewater treatment. Thus, the microstructure of the TiO_2 plays crucial role in determining its photocatalytic activity.

Suetal had reported the various aspects of sol-gel method in a detailed way. A solution of titanium(IV) n-butoxide ($Ti(O-Bu)_4$) in isopropyl alcohol (i-PrOH), was used as molecular precursor of TiO_2 . In order to control the reaction kinetics, acetylacetonc was used as a chemical additive to moderate the reaction rate. Water was deionized. The water used for hydrolysis in solution with i-PrOH was added gradually under mechanical stirring. The reaction condition was carefully controlled to obtain white precipitate of titanium oxyhydroxide, which was then washed with water for several times. The molar ratio of these reactants was: $Ti(OBu)_4:H_2O:i-PropOH:acac = 1:100:2:0.01$. The final solution was peptized by adding HNO_3 , followed by refluxing at for R h to give a sol or pH 2.5.

During optimization of processing conditions in the early stage or this work, the important role of the pH value in the control of the size the TiO_2 particles was revealed. A portion of as-prepared titania hydrosol was retained I'm further analysis and comparison. The other portion was gelled by drying at $100^\circ C$ for 3 h, then calcinated in vacuum oven at various temperatures ($400 - 700^\circ C$) to give TiO_2 powder

b.Synthesis of TiO_2 nanoparticles with pure brookite at low temperature by hydrolysis of $TiCl_4$ using HNO_3 solution:

The rutile and anatase phases are well known and many studies on their synthesis and application have been reported. However, it has been reported that anatase phase is difficult to

prepare and the difficulty in preparing brookite having high purity and large surface area is probably one of the reasons for its limited application. Recently, pure brookite-type TiO_2 particles have been synthesized by hydrothermal method, in which the autoclave with high temperature and pressure as well as complexing agents are required. Other researchers reported that brookite is sometimes observed as a by-product when the precipitation is carried out in an acidic medium at low temperatures. J.H. Lee and Y.S. Yang described a relatively simple method that leads to synthesis of pure brookite -type TiO_2 nanoparticles without adding complexing agents at low temperature.

In this method, TiCl_4 used as a starting material was hydrolyzed using HNO_3 so that TiO_2 nanoparticles could be obtained by merely heating it at temperatures ranging from 60°C to 150°C , for the formation of pure brookite -type TiO_2 particles from aqueous TiCl_4 solution. A typical procedure for making nanocrystalline TiO_2 particles was as follows. TiCl_4 was hydrolyzed by adding 1.0M HNO_3 drop wise to prepare a stock solution, in which the concentration of titanium was 5.45M. During the reaction, the yellow cakes of $\text{TiO}(\text{OH})_2$ were formed first, which were then dissolved with added HNO_3 solution to form an aqueous TiCl_4 solution. This stock solution remained in a stable state with out precipitation even after 6 months at room temperature. Finally, HNO_3 solution with concentration of 1.0-8.0 M was added to the stock solution to prepare transparent aqueous TiCl_4 solutions with various concentrations of Ti^{4+} for precipitation. This solution was poured into reactor and placed in the oven at the temperature of 60 150°C for precipitation. TiO_2 precipitates were repeatedly cleaned by distilled water and dried at 70°C for 48 hr or more to obtain the final particles. It was found that the concentration of HNO_3 in the solution play an important role in determining the crystal structure of TiO_2 precipitates.

Synthesis of high-purity anatase TiO₂ nanoparticles via a low-temperature sol-gel route

Among the three well-known crystallographic phases of TiO₂, anatase has been proved to have excellent chemical and physical properties for environmental purification and many other uses. The fundamental requirement for the technological applications of anatase is the preparation of suspensions or substrate supported transparent thin films. For example, titania suspensions have been demonstrated to possess high available surface areas, which are beneficial for aqueous photocatalytic reactions. TiO₂ colloidal suspensions with excellent dispersibility and long-term stability are also the basis of the low-temperature formation of homogeneous titania thin films. However, the existence of agglomeration of the nanoparticles challenges the formation of stable titania suspensions. Surface modification with organic surfactants may help to disperse TiO₂ nanoparticles in solvents by steric stabilization, which is, however, at the expense of deterioration of the material properties.

Therefore, it is urgent to find a surfactant free methodology to prepare anatase TiO₂ nanoparticle suspensions with less agglomeration.

1. Xu et al. reported on the synthesis of high-purity anatase TiO₂ nanoparticles via a low-temperature sol-gel route. In their synthesis, 5 g of TiCl₄(99%) was dissolved in 50 ml of ethanol(98%) with continuous stirring to form a transparent yellowish sol at 0^o C in an ice-water bath. After being stirred for 2 h, the transparent sol was placed in an oven and baked at 80^o C for 3 days. The obtained off-white powder was washed carefully using a centrifuge at a speed of 10,000 rpm to remove the organic species and Cl⁻ that were adsorbed onto the sample surfaces. The white product thus obtained was dried at 50^o C in air.

Room temperature synthesis of mesoporous aggregates of anatase TiO_2 nanoparticles

The sol-gel chemistry performed in water rich solvents always produces amorphous titania and requires higher temperature for its crystallization, which affects/destroys previously existing nanostructures. It has been reported that sol-gel reactions in non-aqueous media give nanocrystalline titania at ambient conditions. Also, sol-gel method allows better control from the molecular precursor to the final product, which makes possible the low temperature tailoring of TiO_2 with useful properties. S.B. Deshpande et al. have described a simple sol-gel route for the synthesis of 4-5 nm sized anatase nanoparticles under mild conditions in non-aqueous media followed by aging/drying process that helps to develop crystallinity and their self assembly leading to mesoporous aggregate structures.

Titanium oxide nanoparticles are obtained through room temperature hydrolysis of titanium tetrabutoxide (BTM) in presence of acetic acid followed by aging process at ambient conditions. TiO_2 powders were synthesized by carrying out controlled hydrolysis of BTM in acidic media by maintaining different volume ratios at room temperature.

The controlled amount of water necessary for hydrolyzing alkoxide groups was added during processing. The as-dried precipitate was aged for different time duration ranging from 120 to 360 days. Aged precursor heat-treated at various temperatures (400°C and $560^\circ\text{C}/2\text{ h}$) in air. In the sol-gel process, two simultaneous reactions: hydrolysis and condensation take place when $\text{Ti}(\text{OC}_4\text{H}_9)_4$ reacts with water. The amount of water determines the degree of hydrolysis and type of initial species formed, thus influencing the condensation reactions that involve the polymerization or hydrolysed metal alkoxides in alcoholic solution. The acetic acid is probably adsorbed on the surface of TiO_2 particles on specific sites that helps to control the

particle size and porous nature of the aggregates.

Size-controlled synthesis of anatase TiO_2 nanoparticles by carboxylic acid group-containing organics

Titanium isopropoxide, titanium tetrachloride, titanium(IV) sulfate, and amorphous titanium dioxide are commonly used as starting materials in the above-mentioned synthetic processes. The different synthetic processes usually result in the as-prepared nanosized TiO_2 with distinct physiochemical properties. Organic modifiers, such as hydroxyl group-containing, carboxylate group-containing, and amine group-containing organics or polymers, are widely used in synthesis of nanosized TiO_2 to regulate their final morphology and hence their properties.

B. Jiang used Carboxylic acid group- and hydroxyl group-containing organics with different number of functional groups and different nonpolar parts as modifiers in the synthesis of phase-pure anatase nanosized TiO_2 using metatitanic acid as a starting material.

TiO_2 nanoparticles can be conveniently synthesized starting from a small amount of anatase crystal seed-containing metatitanic acid and using carboxylic acids as modifiers. The 127.3 g of metatitanic acid (TiO_2 , 51 wt.%), which contained 2 wt.% of anatase crystal seeds with particle sizes in a range of 2-5 nm, was dispersed in 500 ml of distilled water, pH of the metatitanic acid suspension was ca. 2.2. Then, a 2.1 M Na_2CO_3 aqueous solution (95 ml) was added drop wise into the metatitanic acid suspension, giving the suspension of pH 10. The addition of Na_2CO_3 is beneficial to disperse metatitanic acid well by decreasing the hydrogen bonding among metatitanic acid molecules. After neutralization, a certain amount of organic modifier was added into the suspension under stirring, and then the modifier-containing TiO_2 aqueous suspension was peptized at 60°C for 1 h. The precipitate was washed with distilled water until the tilt rate conductance was decreased to 20mSm⁻¹. The metatitanic acid treated as above was added nitric acid (60

%) solution and distilled water to a total volume of 250 ml at pH of 0.1. After
ng at room temperature for 3 h, the suspension was autoclaved in a Teflon -
ed autoclave (300 ml) at 150 C for 18 h.

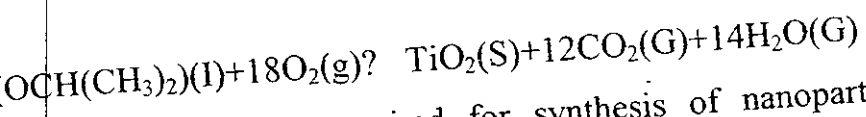
Flame spray pyrolysis

A lot of methods to fabricate TiO_2 nanoparticles such as a sol-gel, a flame-
based or an electrical furnace assisted gas phase process have been reported. One
of the process disadvantages of the sol-gel process for the synthesis of TiO_2
nanoparticles, is that it requires subsequent calcination to obtain crystals of anatase
and rutile. Flame-based methods are of potential commercial importance because
of their high production rates and relatively low cost. Furthermore, they can be
operated as continuous processes. Hence this technique has been used a lot to
produce fine, pure and single -phase particles in the as-prepared state.

H. Chang described a method to synthesise TiO_2 nanoparticles having
different phase compositions and particle sizes were prepared by flame spray
pyrolysis.

Flame spray pyrolysis (FSP), a flame assisted liquid droplet to - particle
conversion process, is an attractive method to obtain multi component
nanoparticles. The particles prepared by FSP exhibit high purity, controlled
stoichiometry and clear crystallinity because the flame temperature can be
maintained high enough to complete thermal decomposition through intense
oxidation chemistries.

The precursor solution for the synthesis of TiO_2 nanoparticles was prepared
by dissolving titanium tetraisopropoxide (TTIP). The overall chemical reaction for



The equipments required for synthesis of nanoparticles are ultrasonic homogenizer, diffusion flame burner, and thermophoretic sampler. The ultrasonic homogenizer was operated at 1.7MHz to generate liquid droplets of precursor solution. The generated droplets were then carried by argon gas into the diffusion flame burner, in which the evaporation of solvent, the precipitation of solute, decomposition and oxidation occurred.

Hydrogen gas was used as a fuel while oxygen and air were used as oxidants for the diffusion flame. The burner (36mm in outside diameter and 300mm in length) consisted of five concentric stainless tubes. Droplets were supplied to the high temperature flame zone through the central tube with an argon gas flow rate of 2 l/min. In order to keep the straight pathway of the precursor droplets in the flame region, argon gas was flowed at 1 l/min through the second tube from central one. Hydrogen, oxygen, and air passed through the outer tubes with flow rates of 5, 6, and 15 l/min, respectively. A stainless tube of 50mm in diameter and 100mm in length was also installed at the burner exit to facilitate stable burning. Particles synthesized in the flame were thermophoretically collected on the surface of a cold glass tube (100mm in diameter and 300mm in length) which was installed at the location of 150mm above the burner exit and maintained at 12°C by flowing cooling water through the inside of the glass tube. The particle size depended on the precursor concentration and flame temperature.

g. Preparation of TiO₂ nanoparticles in glycerol-containing solutions

The formation of nanoparticles by the sol-gel method has been reported to be strongly dependent on the choice of a solvent and a foreign stabilizing surfactant such as polyethylene glycol (PEG) and 2-(2-methoxyethoxy) ethanol. An attempt was made to find a new stabilizer and a solvent system to obtain

T. Trung reported synthesis of TiO_2 nanoparticles of anatase, structure were prepared from titanium isopropoxide by using a new solvent system that is different from other conventional stabilizer-free solvents such as water and different types of alcohol and acid, in which a solution containing glycerol is used. TiO_2 nanoparticles were prepared by hydrolyzing titanium isopropoxide, $\text{Ti}(\text{i-Pro})_4$ (purity 99.999%), in different solvent systems with or without glycerol (purity 99.5%, spectrophotometric grade).

h. one component solution system

A novel one-pot synthesis route was proposed to prepare nanometric anatase TiO_2 using trichloroethylene as reaction medium, which may have a great advantage over multicomponent solution systems when TiO_2 is used as a reinforcing filler for polymers dissolved in trichloroethylene' by T. Trung, C. -So Ha.

Two milliliters of titanium isopropoxide was mixed with 60 ml of trichloroethylene with traces of water, under rigorous stirring, at room temperature. Then the hydrolysis and condensation reactions occurred. The as-prepared powders were washed by ethanol and acetone for several times and dried at 100°C in vacuum oven for 3 days, then heated at 400°C for 10h in air.

i. Hydrothermal synthesis of titanium dioxide from peroxotitanate solution

TiO_2 can be also obtained by hydrolysis of titanium compounds, such as titanium tetrachloride or titanium alkoxides, in solution. However, the use of starting materials containing of chlorine ligand usually resulted in the residue of chlorine in the as-prepared powder, which would increase the environmental problem. Therefore, chlorine-free route for preparation of high-purity TiO_2 nanoparticle is preferable for the practical application. Y.B. Ryu et al. prepared

which were precipitated from an aqueous peroxotitanate solution using different amine group-containing organics.

Peroxotitanate solution was prepared from H_2TiO_3 (80%) dissolved in the presence of H_2O_2 (30%) and different amine group-containing organics (NH_4OH , tetramethylammonium hydroxide (TMAOH, 25 wt%), tetraethylammonium hydroxide (TEAOH, 35 wt%), tetrabutylammonium hydroxide (TBAOH, 45 wt%), in appropriate quantities. Specifically, 2 g of H_2TiO_3 was added to an ice-cooled solvent containing 20 ml of H_2O_2 and 3 ml of amine group containing organics. After stirring for 90 min, a homogeneous pale yellow-green solution was obtained, which can be stable for several weeks at temperature of less than 5°C . Fresh aqueous peroxotitanate solution was diluted by deionized water to 100 ml and then was transferred to a 250 ml Teflon container held in a stainless steel vessel. After the vessel was tightly sealed, it was heated at $120\text{-}200^\circ\text{C}$ for 5 h. After hydrothermal treatment, the TiO_2 particles were separated in a centrifuge at 10,000 rpm for 3 min and were then washed in distilled water. The particles were dried at 105°C for 12 h and then were calcined at $200\text{-}900^\circ\text{C}$ for 3 hrs. The TiO_2 particles calcined at 400°C were shown to have a stable anatase phase which has no organic compounds.

j. Synthesis of nano sized TiO_2 particles in reverse micelle systems

Reverse micelle systems (or water-in-oil microemulsions) have been used as microreactors to synthesize ultrafine particles with a narrow distribution of particle size by controlling the growth process. However, TiO_2 nanoparticles prepared by the reverse micelle method do not show photocatalytic activity because they are amorphous. In order to show photocatalytic activity, amorphous TiO_2 nanoparticles should be calcined at rather higher temperature. At the same time, the organic contaminant remaining in the prepared TiO_2 using reverse micelle

a high temperature results in sintering, agglomeration, and phase transition from anatase to rutile.

R. Inaba et al. attempted to produce monodispersed TiO_2 nanoparticles of high purity using a reverse micelle (RM) system composed of water, Triton X 100 (TX-100) (tert-octylphenoxy polyethoxy ethanol) and isooctane and by hydrothermal treatment. Nanosized pure TiO_2 particles with high crystallinity, large surface area and high thermostability were prepared by hydrolysis of tetrabutyl titanate in water(TX -100)isooctane reverse micelle solutions as reaction media followed by hydrothermal treatment for improvement of crystallinity. The size of TiO_2 nanoparticles can be controlled by changing the water content of the reverse micelle solution.

For the preparation of TiO_2 nanoparticles in the RM system, TX -100 was used as the surfactant and isooctane was used as the continuous oil phase. Tetrabutyl titanate was used as a titanium dioxide source. Preparation of titanium dioxide was carried out as follows.

The RM solution was prepared by dispersing the aqueous phase into the TX-100 (0.64 g, 2.5×10^{-3} mol)/isooctane (50 ml) mixture with vigorous stirring. During this process, the water content of the solution, W_o ($=[\text{H}_2\text{O}]/[\text{TX-100}]$), was controlled.

In order to allow tetrabutyl titanate to react with water completely during hydrolysis and polycondensation, the molar ratio R ($=[\text{I-bO}]/[\text{TTBJ}]$) was adjusted to 2. The solution was aged under mild stirring for 24 h. The hydrolysis of tetrabutyl titanate results in the formation of TiO_2 nanoparticles inside the cores of the reverse micelles. The temperature of the solution during the process was kept at 30°C . Thus, the particles prepared inside the constrained micro reactors were expected to be relatively dispersed in the order of nanometers. Then the solution was charged into a 100 ml Teflon -lined stainless- steel autoclave. The autoclave was kept for 24h. After the

autoclave was cooled down to room temperature, the precipitate generated at the bottom of the autoclave was separated in a centrifuge at 6000 rpm for 20 min. The residue was then washed with methanol and water several times to remove the organic contaminants and the surfactant, respectively. The product was then dried under reduced pressure at 60° C for 5h.

1. An improved continuous process

In general, the synthesis of particles has been developed by a batch or semi-batch process. In addition, Kim et al. have already prepared TiO₂ nanoparticles by hydrolysis of TEOT (tetraethylorthotitanate) in a semi batch/ batch two stages mixed process. However, on an industrial scale, the batch and semi batch process are not suitable; a continuous process is required for mass production.

K.D. Kim and H.T. Kim attempted an improved continuous process in which a small reactor for controlling the nucleation rate is connected in series with an aging tube for controlling the growth of particles. The hydrolysis rate is controlled by the vaporization of H₂O and the feed rate of TEOT (tetraethylorthotitanate) solution, respectively. In addition, by controlling the parameters affecting to the particle size in the aging tube, nanoparticles can be obtained.

The apparatus consists of the following sections; (1) small reactor (100 ml) for two feed reagents (vaporized water and TEOT) with an ethanol solvent, (2) a syringe pump (Kd scientific, Model 100) to supply the vaporized H₂O, Et(OH) solutions with a constant feed rate (0.040 -0.306 ml/min) and a micro feed pump (EYELA, MP-3) to supply the TEOTI Et(OH) solutions with a constant feed rate (0.66-1.10 ml/min), (3) a heating tape for vaporizing the H₂O, and (4) a long silicon tube (diameter: 3.0 mm) for aging of the particles. The aging tube is placed in a water bath at a constant temperature and can be changed for desired length and valve to control the flow rate

solutions after mixing of TEOT and vaporized H_2O . The two feeds are continuously supplied to the small reactor. Primary nuclei of titanium dioxide are generated by the polymerization of the hydrolysis product while passing through the small reactor. A part of them is grown to fine particles while passing through the aging tube. In this continuous reactor system, two methods for preparing nanoparticles with narrow size distribution were used. One is the vaporized $H_2O/Et(OH)$ solution by which slower hydrolysis rate of TEOT is induced, resulting in prevention of agglomeration of particles. The possibility of an increase in particle size and agglomeration can be minimized by using this intermittent flow. The ways for changing the flow type in the aging tube are N_2 gas injection and valve handling. Firstly, at the entrance of the aging tube, there is another inlet to feed the N_2 gas. So, the feed liquids and the injected N_2 gas form an intermittent flow in the aging tube. Secondly, the flow type of solution in the aging tube can be controlled by adjusting the valve placed in the middle of the small reactor and the entrance of the aging tube. By using these two methods, the continuous flow in the aging tube can be separated into small portions.

1. Sonochemical preparation of titanium dioxide nano particles

H. Arami et al, had shown that the dissolution of the TiO_2 raw powder in NaOH solution before ultrasonic treatment resulted in the formation of titania nanopowders instead of nanotubes.

Rutile nanoparticles with average crystallite size of about 15 nm, mean particle size of 20 nm and specific surface area of $78.88 \text{ m}^2/\text{g}$, were prepared through a sonochemical method. It has been shown that solution treatment before ultrasonic irradiation culminated in the formulation of rutile nanoparticles.

dissolved into 30 ml NaOH

solution (10 M) under vigorous stirring at room temperature for 2 h. Then the yellowish solution was irradiated in an ultrasonic bath (Power Sonic 405, 40 kHz and 350 W) for 2 h in ambient temperature. The resultant precipitates were then centrifuged, washed and decanted with deionized water several times and dried at 60°C for 24 h.

n. Preparation of titania particles by thermal hydrolysis of TiCl_4 in n-propanol solution

C. Fang, Y. - W. Chen reported about the preparation of uniform dispersed spherical titania powders by thermal hydrolysis of TiCl_4 in a mixed solvent of n-propanol and water. Titanium tetrachloride (99% TiCl_4) was dissolved in distilled water. The concentration of titanium was 1M. The aqueous solution was stirred for 6 h at 10°C. The aqueous solution was mixed with n-propanol to alter the RH ratio to 0, 1, 2, 3 and 4, respectively. Hydroxypropyl cellulose (HPC) (150-400 cps,) was then added to the solution as a steric dispersant. The final concentration of titanium in the mixed solution was 0.05, 0.10, and 0.20 mol/l respectively.

Six hours later, the solution were heated and aged at 70°C in an oven for a few minutes to carry out hydrothermal hydrolysis and white precipitate was formed. After precipitation, the solution was neutralized with a 3N NH_4OH solution to remove chlorine ion. The precipitate was then separated by centrifuging and washing twice with distilled water and finally rinsed with n-propanol. The obtained precipitate was dried in an oven at 60°C for 24 h.

The spherical titania with narrow size distribution was synthesized by hydrolysis of TiCl_4 in a mixed solvent of n-propanol and water. The morphology of titania powder was affected by the volume ratio of n-propanol particle size increased with an increase of RH ratio. The TiO_2 particles were spherical and

nia particles was obtained by adding HPC in mixed solution. The particle size increased with an increase of the concentration of TiO_2 , in the solution

preparation of titanium dioxide in the W/C micro emulsions

Water-in-oil (W/O) micro emulsions has been successfully employed to obtain ultra fine particles with controlled sizes for a variety of materials. However, this method gives rise to environmental problems caused by solvents. Thus, an improved method using carbon dioxide instead of oil has been developed. Here, we report the use of water-in-carbon dioxide (W/C) micro emulsions to achieve particle size control for titanium dioxide. Carbon dioxide is an attractive alternative to organic solvents because it is non toxic, nonflammable, highly volatile, inexpensive, and environmentally benign.

S.-S. Hong et al. investigated the formation of TiO_2 nanoparticles through direct reactions of titanium tetraisopropoxide (TTIP) with water solubilized in W/O microemulsions stabilized by polymeric surfactant poly(dimethyl aminoethyl methacrylate-block-1 H, 1 H,2H,2H-perfluorooctylmethacrylate) (PDMAEMA - b-PFOMA). The crystallite size can be controlled by the water-to-surfactant mole ratio, W_o , which influences the hydrolysis rate, droplet size and intermicellar interactions.

A variable volume view cell was used as a reaction vessel to prepare TiO_2 particles. The cell was immersed in a water bath and the temperature of the cell was controlled by the water bath. Pressure was controlled using a high pressure generator. An external magnetic stirrer was used with a PTFE-coated magnetic bar. The W/C microemulsions were prepared by adding the PDMAEMA - b-PFOMA surfactant to the cell, followed by the addition of water to achieve the desired water-to-surfactant ratio (W_o) and then the cell was loaded with carbon

th continuous stirring for 30 min. Finally, an adequate amount of titanium
raisopropoxide (TTIP) was added and then the pressure of the cell was
ntrolled to 4000 psi for 30 min to prepare titanium hydroxide. The prepared
nosized particles were washed by ethanol and dried at 105° C for 1 day and then
alcined at 500°C for 3 hours.

METHODS OF IMPARTING THE NANO PARTICLES OF METAL OXIDES ONTO THE TEXTILE SUBSTRATES

1. PAD DRY CURE METHOD

The most widely used method to impart nano particles onto the textile substrate. The textile substrate, cut to the size of 30 x 30 cm was immersed in the solution containing nanoparticles (2%) and acrylic binder (1 %) for 5 min and then was passed through a padding mangle, which was running at a speed of 150 rpm with a pressure of 15 l(gf/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^o C. The fabric was then immersed for 5 min in 2% of sodium lauryl sulfate to remove unbound nanoparticles. Then the fabric was rinsed at least 10 times to completely take out all the soap solution. The fabric then 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^o C). The fabric sample (10 cm x 10 cm) was soaked for 10 min in a dispersion of nanoparticles (5(10 w/w), under gentle magnetic stirring. The fabric was then squeezed to remove the excess dispersion, and dried in a oven at 130^o C for 15 min at atmospheric pressure (dry heat). The drying step was carried out on a horizontal flat surface.

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 nanoparticles can be minimized. A dispersion of nanoparticles (2%) was filled in a spray gun. The textile substrate was fixed on a vertical board. The nanoparticle 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 AND SCOPE

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

CHAPTER-4

METHODOLOGY

1.1 YARN PROCUREMENT

The following yarns were procured for the fabric manufacture.

POLYESTER VISCOSE BLEND (65/35)

POLYESTER VISCOSE BLEND (35/65)

1.2 FABRIC MANUFACTURE

Fabrics with two commonly used structures WOVEN, KNITTED were manufactured using a sample loom. The type and specification of fabrics are as follows,

Table No 2

WOVEN STRUCTURE	
REED	72 EPI
PICK	80 PPI
WIDTH	50 inches
GSM	135
STRUCTURE	PLAIN

Table No 3

KNITTED STRUCTURE	
GAUGE	24
DIA	24 inches
GSM	130
STRUCTURE	PIQUE

The gsm and structure of the fabric were selected based on commonly used configuration for apparels. In woven a gsm of 135 and plain weave is selected, as it is the commonly used pattern for shirting materials. In knitted a gsm of 130 and pique pattern for Polo t-shirts.

SYNTHESIS OF NANO METAL OXIDES

SYNTHESIS OF TITANIUM DIOXIDE NANOPARTICLES

- Hydrolysis of TiCl_4 containing 0.8M Ti^{4+} ions
- Using HNO_3 of 5M concentration
- Treated in oven at $70\text{-}80^\circ\text{C}$ for 20 Hrs
- Pure Brookite is obtained with a size less than 20 nm .

3.4. APPLICATION OF NANOPARTICLES 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 nanoparticles (2% , M:L ratio $1:20$) was filled in a hand-held spray gun. The textile substrate ($10\text{cm} \times 10\text{cm}$) was fixed on a vertical board. The nanoparticle 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/cm^2 . The fabric was then dried at room temperature on a flat surface.

TESTING

The testing involved two types of tests namely,

CHARACTERIZATION TEST

FUNCTIONAL TEST.

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

- Fourier transformed infrared spectroscopy (FTIR)
- X-ray powder diffractometry (XRD)
- 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 ($k = 1.54 \text{ \AA}$) source (applied voltage 35 kV, current 25 mA). About 0.5 g of the dried particles were deposited as a randomly oriented powder onto a Plexiglass sample container, and the XRD patterns were recorded at angles between 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

$$D = \frac{0.89 \lambda}{W \cos \Theta}$$

Where,

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

Θ is the Bragg's diffraction angle,

W 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 analyzed through SEM, using a Stereoscan S360 Oxford-Cambridge.

Table No 4

TEST	TEST CENTRE
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,

$E(k)$ is the relative erythematological spectral effectiveness, $S(k)$ is the solar spectral radiance 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.

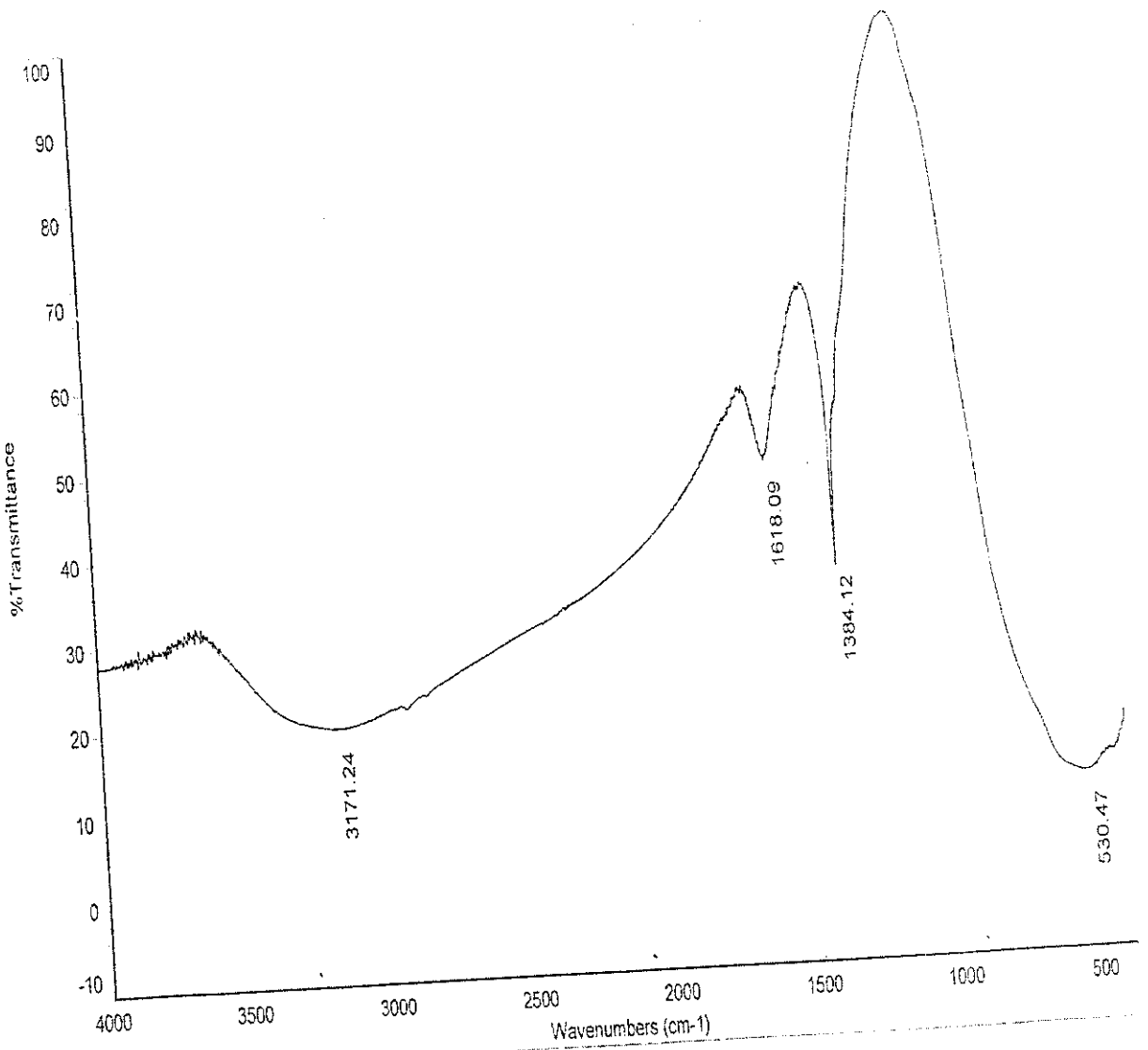
CHAPTER-5

RESULTS

CHARACTERIZATION TESTS

1.1. FTIR

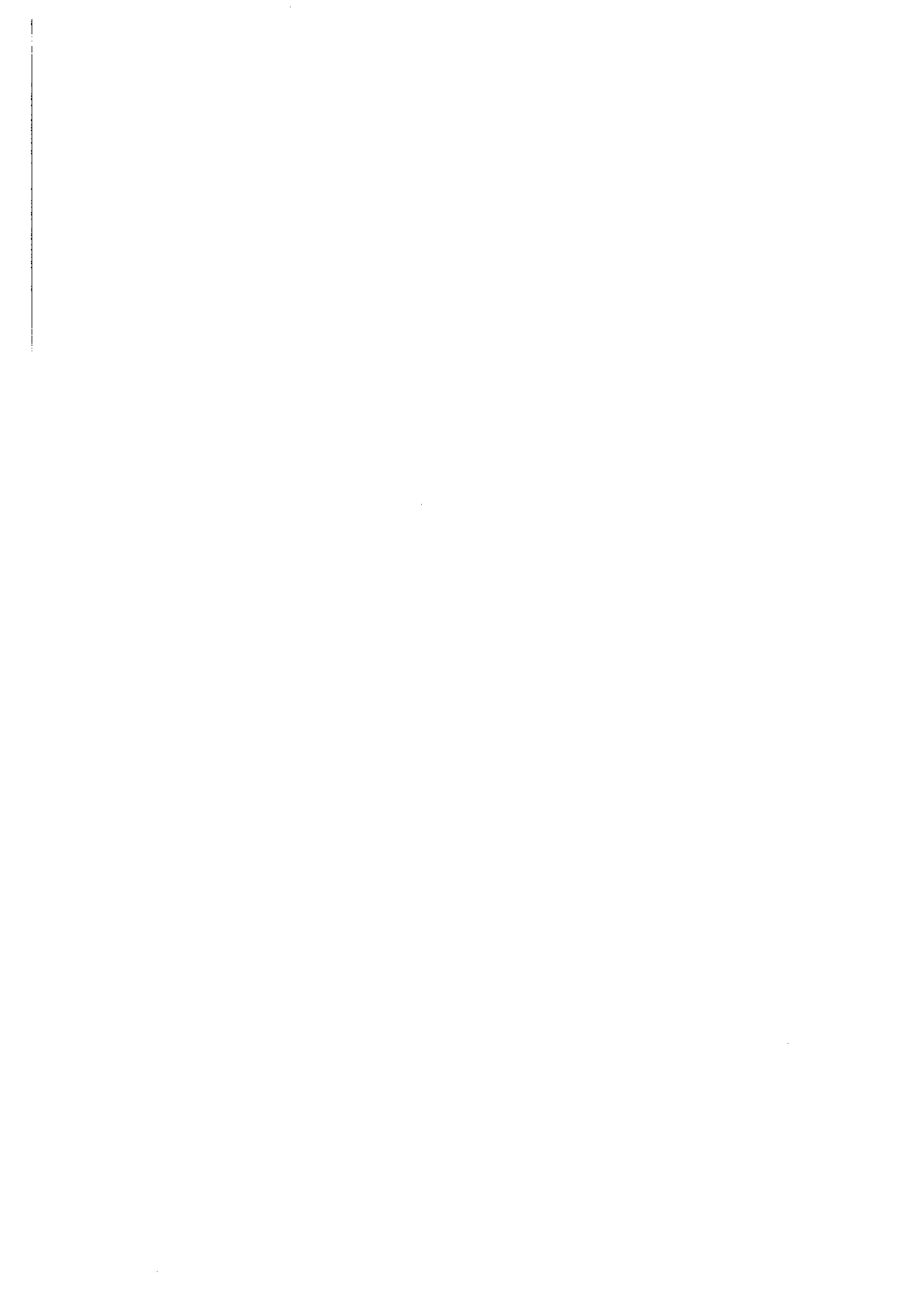
Figure No 2



Date: Thu Mar 12 14:45:16 2009 (GMT-08:01SAIFFT090228E-01(KCT TiO2))

Scans: 32

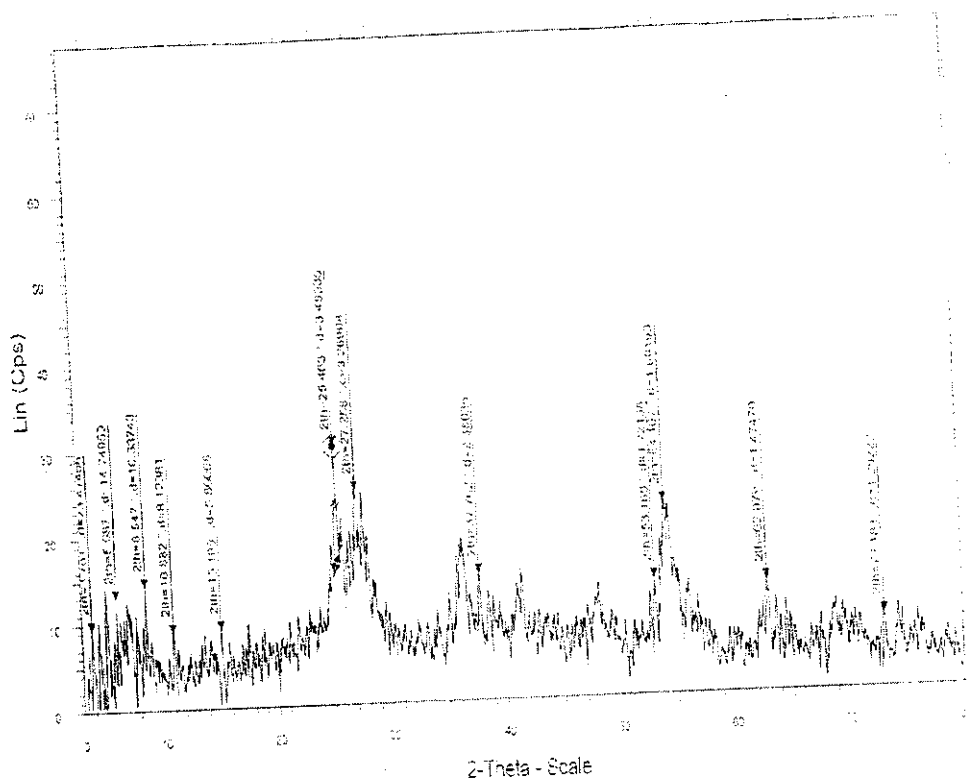
Resolution: 4.000



1.2 XRD

Figure No 3

KCT-TiO2-1



KCT-TiO2-1 - File: 24\FAP\502780\KCT-TiO2-1 - 2 - Step: 6.000 - 2-Theta: 3.000 - Tube: 1.000 - 142 Pat: 0.0 - Generator: 45.00
KCT-TiO2-1 - Scan Area: 25.223 - Right Angle: 20.000 - 1.0 (Cps) - 0.4 (0.01) - Total: 1.170 - Net Area: 5.740 Cps x cm
Conditions: Step: 0.100 | Background: 1.000 | 1.000

FIGURE 3 Shows the XRD pattern of TiO₂ Nanocrystals. The diffraction peaks indicate the Nanocrystalline nature. These peaks at a scattering angle (2θ) of 9.87, 8.597, 25.463, 27.258, 37.762, 54.167 and 62.979 corresponds to reflections.

SCHERRER'S EQUATION

Crystalline Domain Diameter

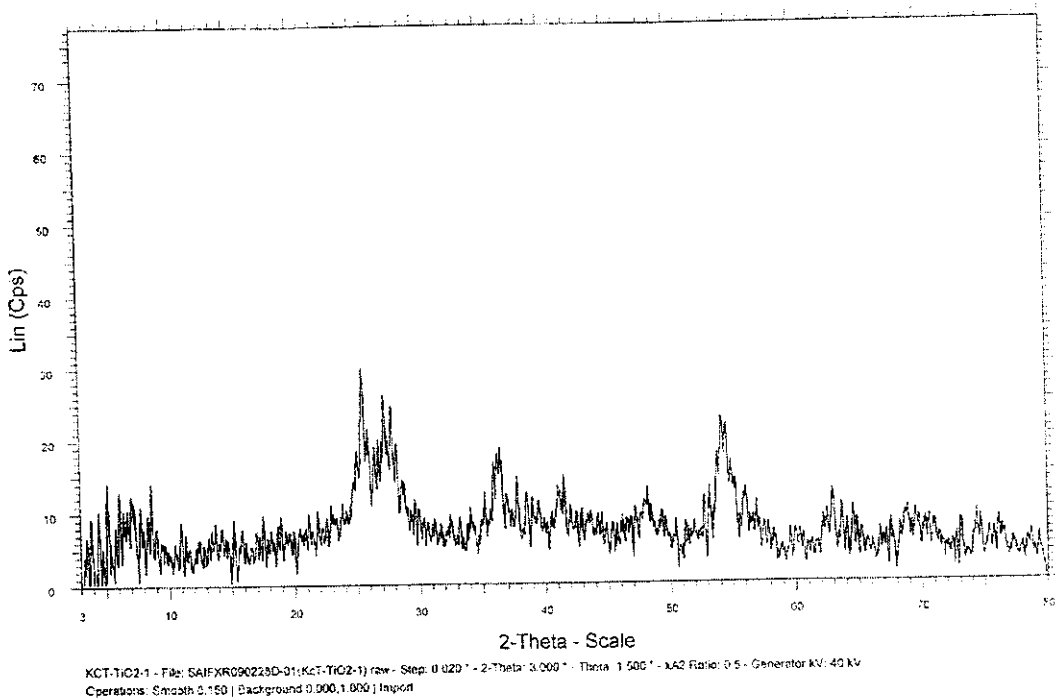
$$D = \frac{0.89\lambda}{\Delta 2\theta \cos \theta}$$

Thus from the data obtained from the XRD graph and using the formula the diameter of particle vary from 40-50nm.

KRD

Figure No 4

KCT-TiO2-1



Using scherrer's formula the diameter of nano particles were calculated,
Titanium dioxide - 40-50nm

5.1.3. SEM

Four samples were sent for SEM testing in PSG college of Technology, coimbatore.

1. Polyester viscose blend(65/35) knitted.
2. Polyester viscose blend(65/35)woven.
3. Polyester viscose blend(35/65)knitted.
4. Polyester viscose blend(35/65)woven.

POLYESTER VISCOSE BLEND(65/35) KNITTED

Figure No 5

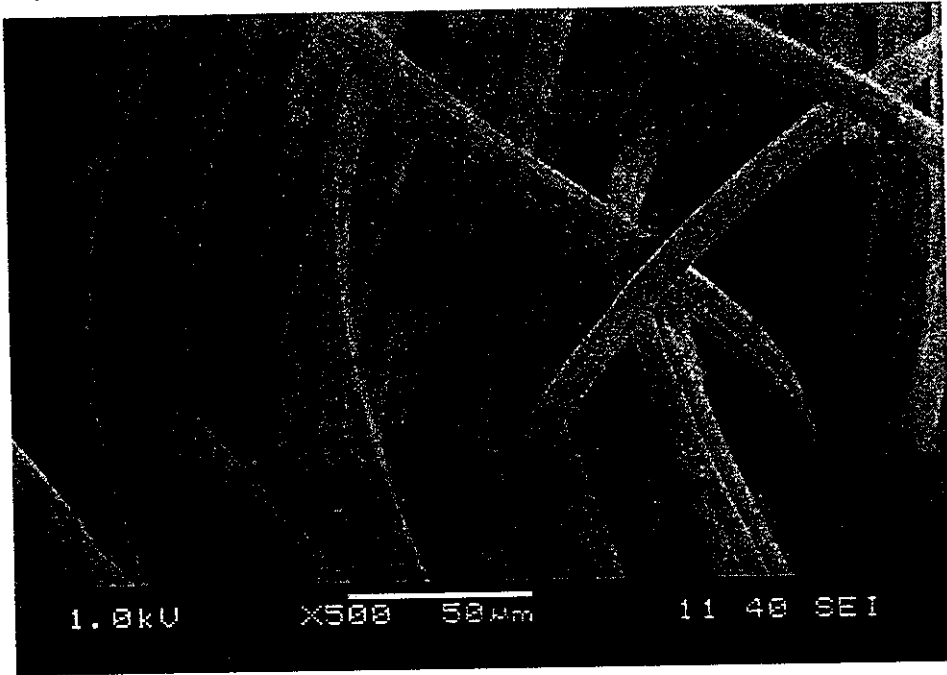
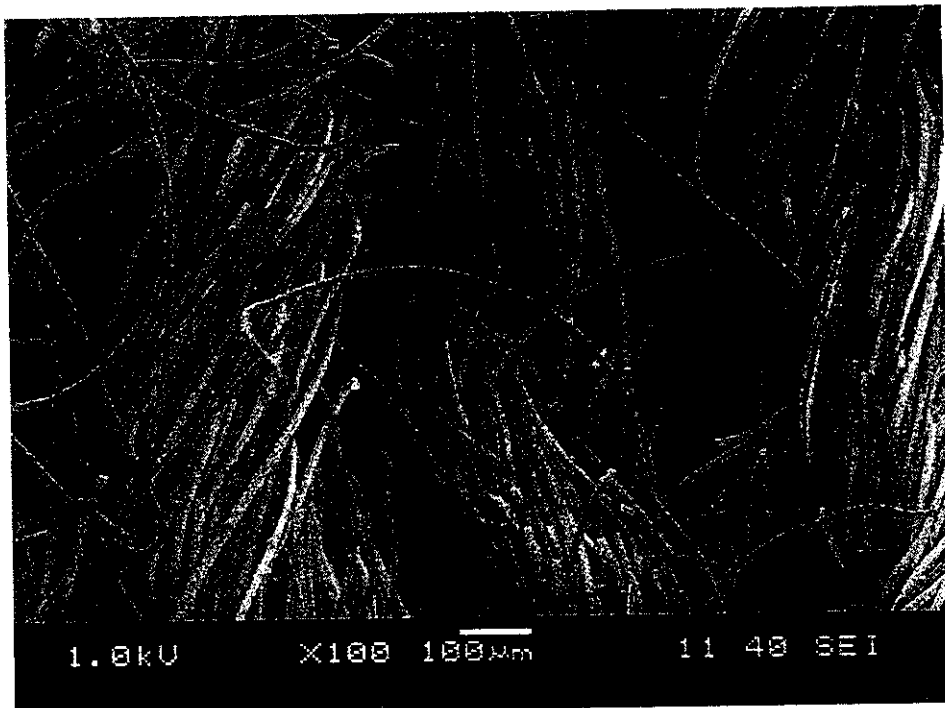


Figure No 6



POLYESTER VISCOSE BLEND(65/35) WOVEN

Figure No 7

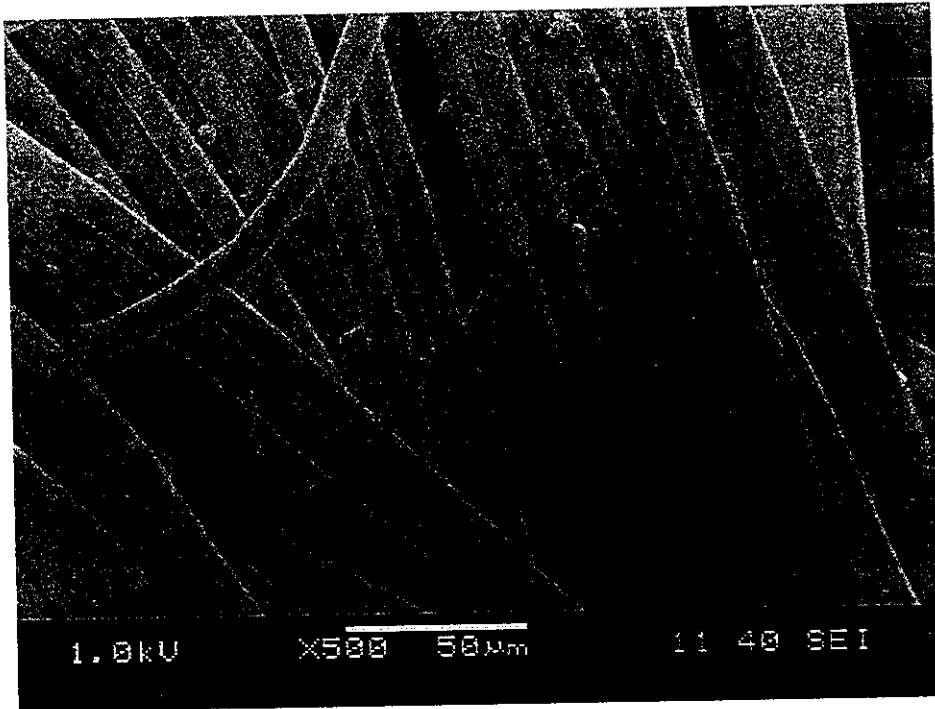
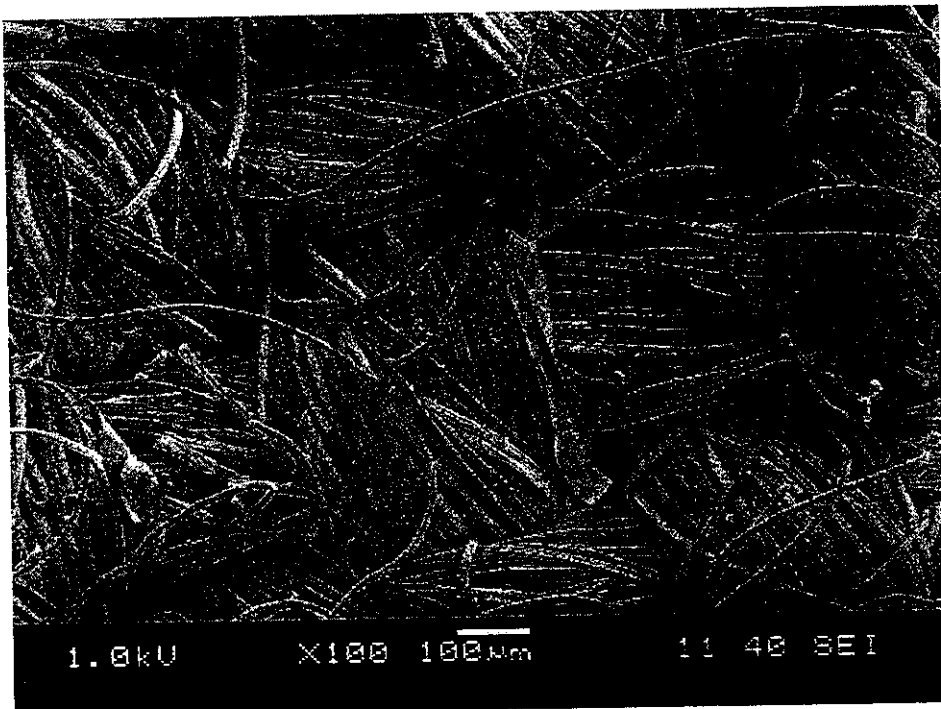


Figure No 8



POLESTER VISCOSE BLEND(35/65) KNITTED

Figure No 9

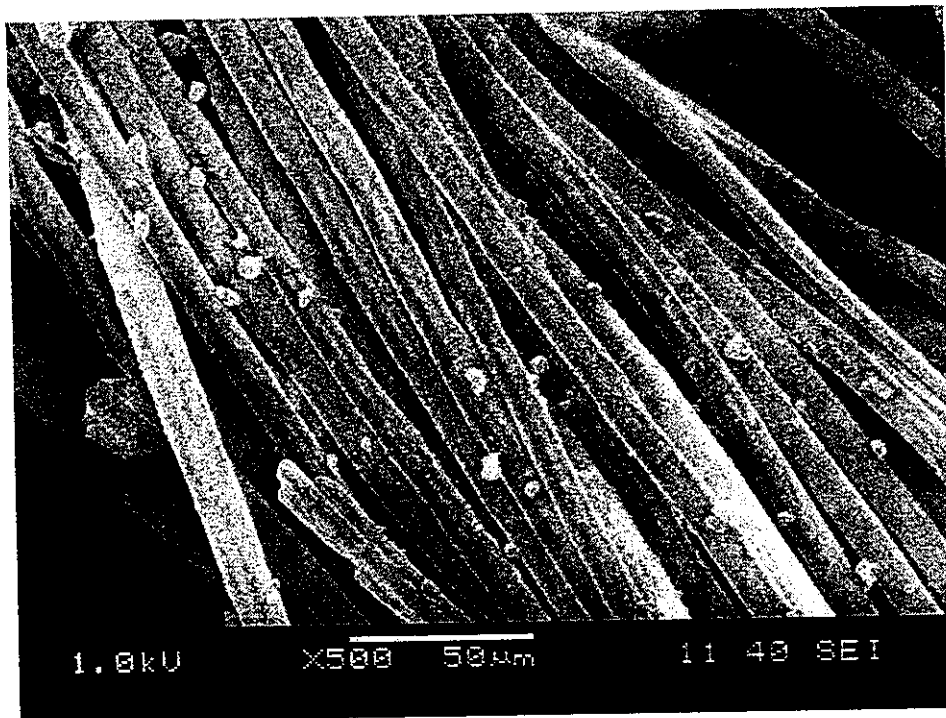


Figure No 10



POLYSETSER VISCOSE BLEND(35/65) WOVEN

Figure No 11

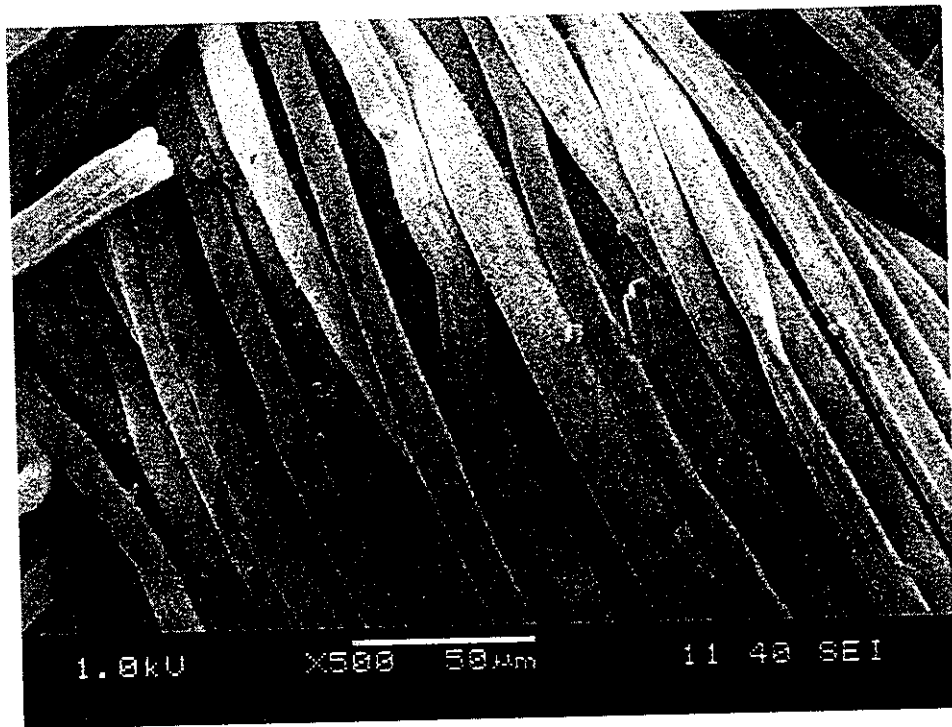


Figure No 12

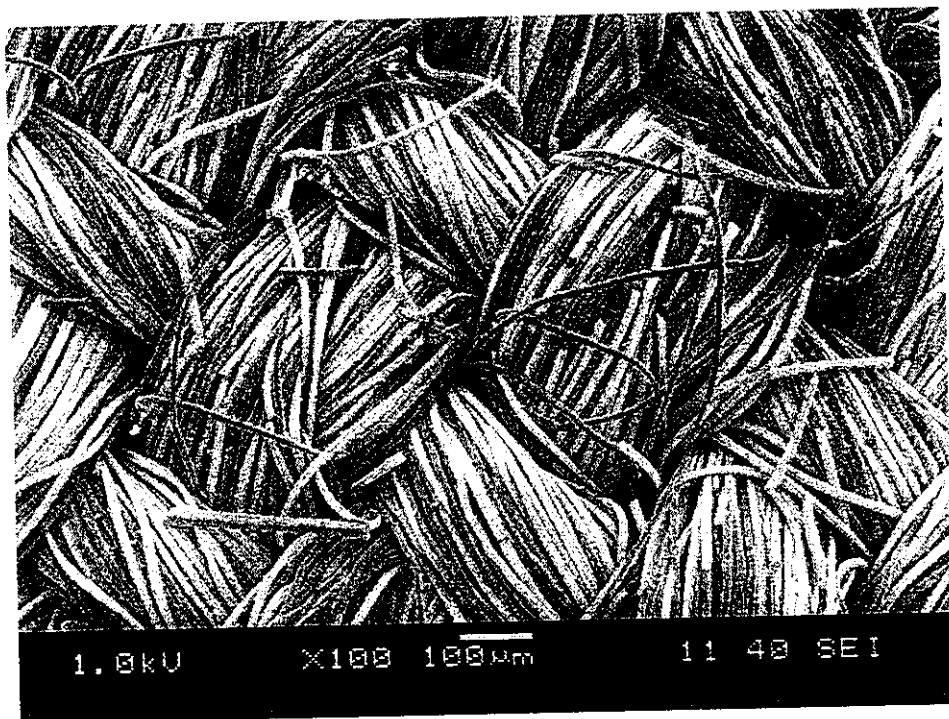


Figure 5,6,7,8,9,10,11 and 12 indicates the presence of nano particle in surface

5.2 FUNCTIONAL TESTING

ANTIMICROBIAL TEST

- Prepare nutrient broth in two sets of conical flask.
- Place the treated and untreated samples inside the flask
- Allow the micro organisms to grow in the media
- Measure optical density value at 660nm

OPTICAL DENSITY VALUE

POLYESTER VISCOSE BLEND(35/65)

Table No 4

Fabric Samples	Optical Density Value
Untreated Woven	0.57
Treated Woven	0.45
Untreated Knitted	0.63
Treated Knitted	0.53

POLYESTER VISCOSE BLEND(65/35)

Table No 5

Fabric Samples	Optical density Value
Untreated Woven	0.58
Treated Woven	0.46
Untreated Knitted	0.54
Treated Knitted	0.40

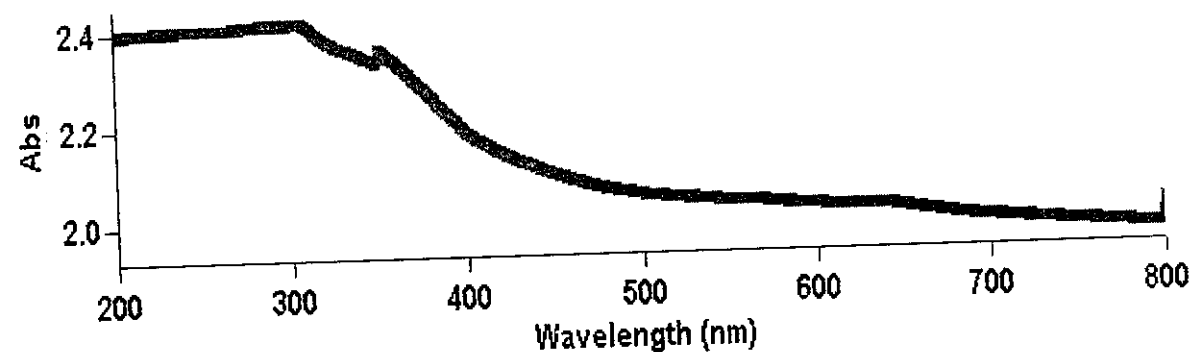
INFERENCE

The amount of microbes present in fabric is indicated by optical density value. As we see in the tables are the optical density value of two blends of Polyester and Viscose. Clearly the values indicate the growth of microbes is prevented in both woven and knitted treated fabrics.

UV TESTS

POLYESTER VISCOSE BLEND(65/35) WOVEN

Figure No 13



Sample Name: SAIFUV090331A-01(Woven1)abs

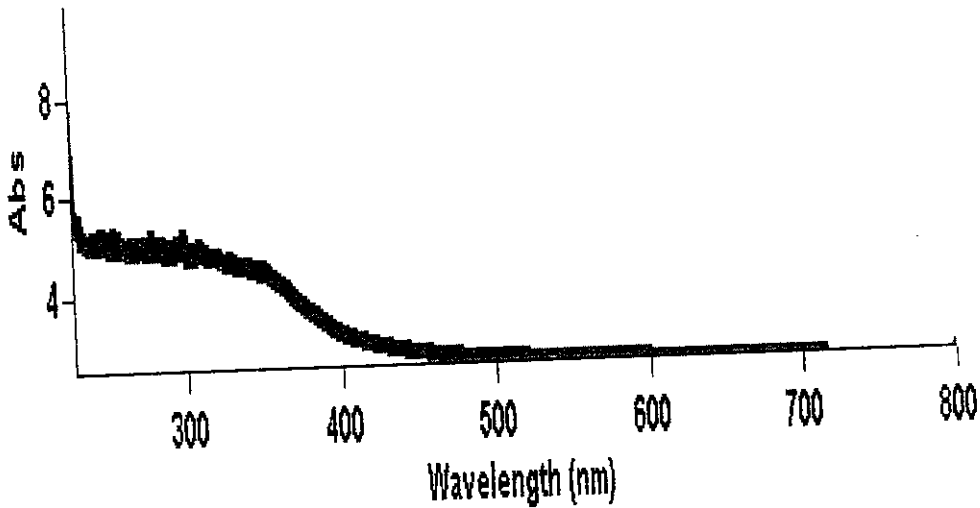
Collection Time 4/3/09 11:58:39 AM

Peak Table
Peak Style Peaks
Peak Threshold 0.0100
Range 800.000nm to 200.000nm

Wavelength (nm)	Abs
350.000	2.356
306.000	2.415

POLYESTER VISCOSE BLEND(35/65) WOVEN

Figure No 14



Sample Name: SAIFUV090331A-02(WovenI)abs

Collection Time

4/3/09 12:03:16 PM

Peak Table

Peak Style

Peak Threshold

Range

Peaks

0.0100

800.000nm to 200.000nm

Wavelength (nm)

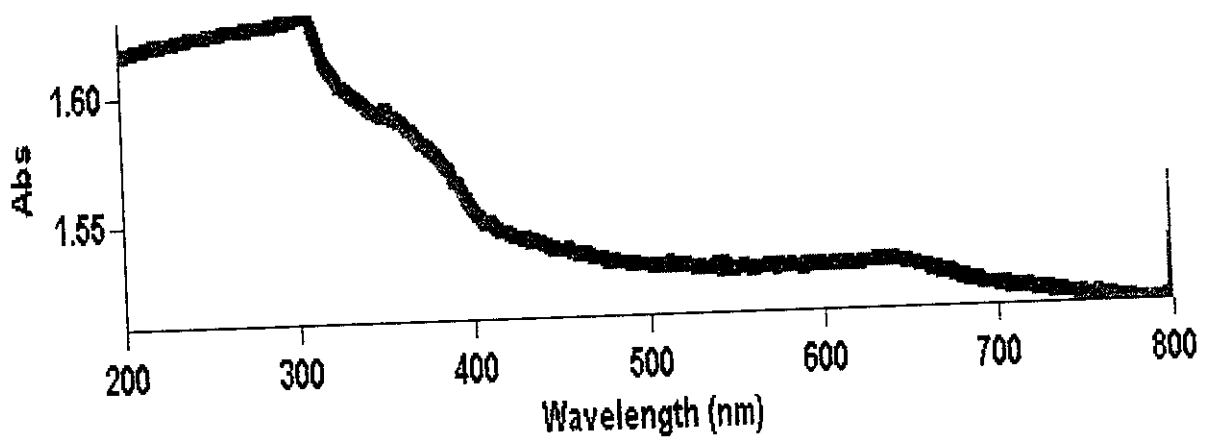
Abs

350.000

4.356

POLYESTER VISCOSE BLEND(65/35) KNITTED

Figure No 15



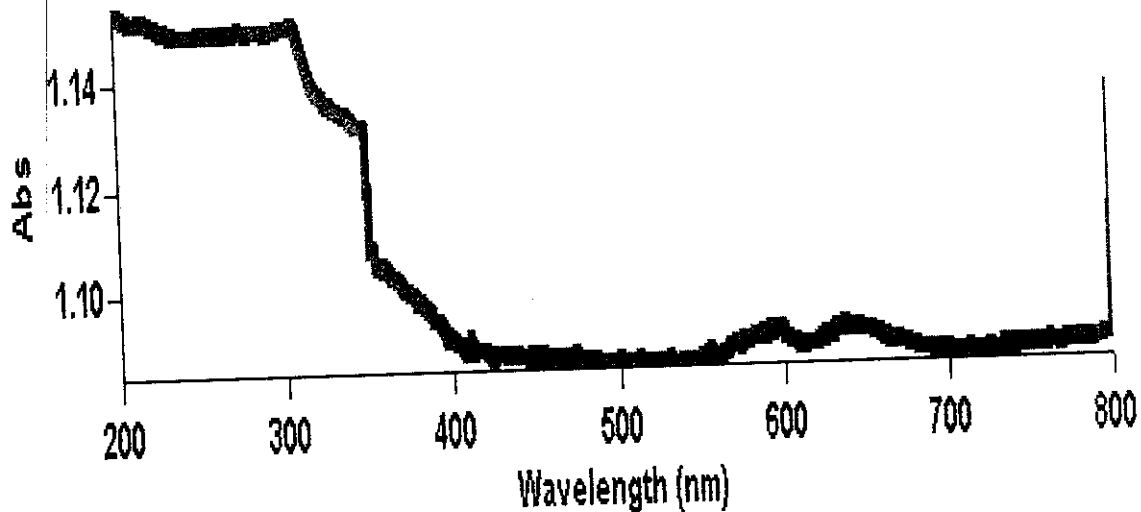
Sample Name: SAIFUV090331A-03(Knitted I)abs
Collection Time 4/3/09 12:07:49 PM

Peak Table
Peak Style Peaks
Peak Threshold 0.0100
Range 800.000nm to 200.000nm

Wavelength (nm)	Abs
306.000	1.630

POLYESTER VISCOSE BLEND(35/65) KNITTED

Figure No 16



Sample Name: SAIFUV090331A-04(Knitted IV)abs1

Collection Time 4/3/09 12:12:28 PM

Peak Table
Peak Style Peaks
Peak Threshold 0.0100
Range 800.000nm to 200.000nm

Wavelength (nm)	Abs
306.000	1.165

CHAPTER-6

CONCLUSION

- Woven structure exhibit better UV protection than knitted due to its compactness and high cover factor.
- Blended fabrics have high UPF than cotton fabrics as Polyester possess higher UV absorption than cotton.
- Although the calculated UPF are significantly lower than the standard values required for classifying the clothing as "excellent" in t JV -shielding, however these results confirm the protection against UV radiation produced by the treatment with nano metal oxide particles on the fabrics. This effect can be enhanced by:
 - Selecting a more advanced method of application.
 - Selecting another kind of textile material (wool, viscose, etc.).
 - Using a different procedure for drying.

Thus, our project work shows that polyester viscose blend with a woven fabric structure treated with Titanium Dioxide nano particles exhibit high ultra-violet protection antimicrobial protection by using spray method for application of nano particles.

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