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SYNTHESIS AND CHARACTERIZATION OF TITANIUM DIOXIDE NANO-PARTICLES AND THEIR APPLICATIONS TO TEXTILES FOR MICROBE RESISTANCE

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ABSTRACT

The challenge of any textile company today to position itself in the global market lies in the development of value added products. Nanotechnology is the art and science of manipulating matter at the nanoscale to create new and unique materials and products. This research work attempted synthesis and characterization of titanium dioxide nano particle and their application on woven and knitted fabrics (100% cotton & 45/55% polyester/cotton) for antibacterial activity.

The titanium dioxide (TiO_2) nano particles were prepared with urea as a reacting medium. These nano particles have an average size of 9 nm, which was confirmed by Transmission Electron Microscope (TEM). 1% of the nano TiO_2 were applied on both woven and knitted fabrics. The presence of nano particles on the fabric surface was confirmed by Scanning Electron Microscope (SEM). The nano TiO_2 impregnated woven and knitted (100% cotton & 45/55% polyester/cotton) fabrics showed excellent antibacterial activity against two representative bacteria, Staphylococcus aureus (Gram positive) and Klebsiella Pneumoniae (Gram Negative).The antibacterial property of untreated fabrics showed no reductions whereas the titanium dioxide nano particles treated 100% cotton woven shows 85% and 64% reduction, 45/55% polyester/cotton woven shows 93% and 78% reductions, 100% cotton knitted fabrics showed 75% and 58%, and 45/55% polyester/cotton knitted fabrics showed 79% and 74% reductions against Staphylococcus aureus and Klebsiella Pneumoniae bacteria, respectively. Mechanical properties such as tensile strength, elongation, crease recovery, air permeability of nano TiO_2 treated fabrics did not change considerably than the untreated fabrics.

Keywords: Microbe resistance, Nanotextiles, TiO_2 synthesis, Tio_2 characterization, Nanoparticles

1. Introduction

Nanotechnology is regarded as a key technology which will not only influence technological development in the near future, but will also have economic, social and ecological implications.

Nanotechnology deals with the science and technology at dimensions of roughly 1 to 100 nanometers (1 Billion Nanometers = 1 Meter), although 100 nanometers presently is the practically attainable dimension for textile products and applications [1]. The technology can be used in engineering desired textile attributes, such as fabric softness, durability, breathability and in developing advanced performance characteristics, namely, water repellency, fire retardancy, antimicrobial resistance, etc., in fibers, yarns and fabrics [2].With the advent of Nano technology, a new area has developed in the realm of textile finishing. Nano coating on the surface of textiles and clothing enhance the material for UV blocking, antimicrobial and self-cleaning properties [3].

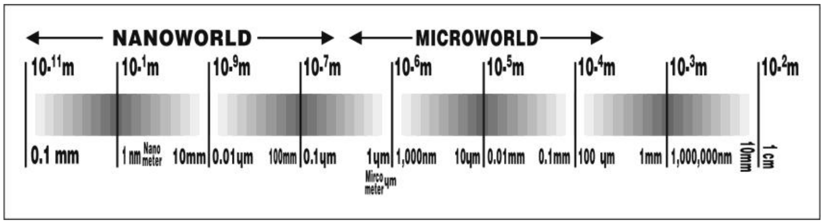

Figure 1.1: Nano scale

The inherent properties of the textile fibers provide room for the growth of micro-organisms. Besides, the structure of the substrates and the chemical processes may induce the growth of microbes. Humid and warm environment still aggravate the problem. Infestation by microbes cause cross infection by pathogens and development odor where the fabric is worn next to skin. In addition, the staining and loss of the performance properties of textile substrates are the results of microbial attack. Basically, with a view to protect the wearer and the textile substrate itself antimicrobial finish is applied to textile materials [4].It is a well known fact that the growth of bacteria and microorganisms in food or water is prevented when stored in silver vessels due to its antibacterial properties. 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 means of photo catalysis with the metallic ion, thereby dissolving the organic substance to create a sterilizing effect. With the use of nano-sized particles, the number of particles per unit area is increased, and thus anti-bacterial effects can be maximized [5]. Nano-TiO_2 sol and finishing agent was prepared by sol-gel method, during which tetrabutyl titanate was used as precursor and ethanol was used as solvent. The agent was penetrated into polyester fabric through a padding method, the anti-ultraviolet performance of the fabric was analyzed and the external morphology was carefully studied afterwards [6].

The spacers are attached on the cotton by formation of an ester-bond. The TiO_2 binds to the cotton by chemical means and the textiles present self-cleaning properties. The deposition of TiO_2 on the cotton textile surface is non-homogeneous due to the irregular surface of the cotton fabrics used [7]. Li et al has done the research about the treatment of cotton fabric using the Nanometer Titanium Dioxide, improving the performance of the anti-ultraviolet property [8]. Durable antibacterial cellulose and its blend fabrics were prepared by incorporating a zinc peroxide polymer [9]. Antibacterial properties can be attained by either physically or chemically incorporating antibacterial agents into fibers or fabrics. The antimicrobial agents can be antibiotics, formaldehyde, heavy metal ions (silver, copper), quaternary ammonium salts with long hydrocarbon chains [10].

2. OBJECTIVES

The main objectives of this research work are Synthesization and Characterization of Titanium dioxide nanoparticles, its application on Woven and Knitted fabrics and testing of finished fabrics for antimicrobial property.

3. METHODOLOGY

3.1. Materials

Two kinds of woven and Knitted fabrics were made and the technical specifications of the two fabrics are shown in Table 3.1.

Table 3.1: Specifications woven and knitted fabrics

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

3.2. Synthesis of Titanium dioxide nano particles

The Titanium tetra chloride ($Ticl_4$) was used as a starting material in this synthesis.50 ml of $TiCl_4$ was slowly added to the 200 ml distilled water in an ice cool bath. The beaker was taken from the ice bath to Room temperature. The beaker was kept in magnetic stirrer to make a homogeneous solution for 30 minutes. Bath temperature was raised to 150˚c and kept in the same temperature till the process of nano particle was completed.

In another vessel 26 grams of Urea was dissolved in 250 ml of distilled water. From the vessel 150 ml of urea solution was added to beaker under constant stirring, drop by drop touching the walls of the beaker. The solution turned into white colloid without any precipitation. After the complete reaction, the solution was allowed to settle and the solution was washed with distilled water for 5 times.

3.3. Characterization of Nano particles

Characterization of the nanoparticles was done by three tests such as X-ray Diffraction Method (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) and transmission electron microscope.

The crystallinity was determined by XRD using a Bruker D8 Advance X rays Diffractometer equipped with a Cu Kα (λ = 1.54 A °) source (applied voltage 40 kV, current 40 mA). About 0.5 g of the dried particles were deposited as a randomly oriented powder onto a Plexiglas sample container, and the XRD patterns were recorded at angles between 20˚ and 80˚, with a scan rate of 1.5˚/min.

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

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

Where λ is the wavelength of the incident X-ray beam (1.54 A˚ for the Cu Kα), O is the

Bragg's diffraction angle, ΔW the width of the X-ray pattern line at half peak-height in radians. The chemical composition of the synthesized materials was checked by FTIR spectroscopy with a Bio-Rad FTS-40 spectrometer. The shape and size of the nano particles were obtained through TEM, using a Philips EM201C apparatus operating at 80kv. The samples for TEM measurements were placed on carbon-coated copper grids. The samples for TEM measurements were prepared from much diluted dispersions of the particles in 2-propanol. Surface area measurements were determined from BET on a Coulter SA 3100 surface area analyzer, under N2 flow.

3.4. Finishing Process

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

3.4.1. Procedure

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

3.5. Characterization of nano finished fabric

- Scanning Electron Microscope

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

3.6. Functional testing of finished fabric samples

To investigate the antibacterial activity of woven and knitted fabrics impregnation was done with titanium dioxide nano particles separately. Antibacterial test AATCC 100-2004 was carried out against Staphylococcus Aureus (Gram positive organism) and Klebsiella Pneumoniae (Gram negative organism).The percentage reduction of bacteria by the 100%cotton and 45/55% polyester/cotton fabrics is reported as R,

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

Where R = % reduction

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

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

4. RESULTS AND DISCUSSIONS

4.1. Characterization of nano particles using XRD, FTIR and TEM

The results shown in figure 4.1 & 4.2 indicate that the experimental conditions greatly affect the morphology and size of the articles, prepared with the different condition. In fact, increasing the reaction temperature, results in a significant lowering of the nanoparticles size and of their agglomeration number D, calculated as

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

TEM size distributions were obtained for nano particles, as shown in Figure 4.3. The mean crystallite size is presented in Table 4.1.

Table 4.1: Diameter of nano particle

Metal oxide Nano particle	Solvent	Temperature C	Average Diameter (nm)
Titanium dioxide	Urea	160’	9

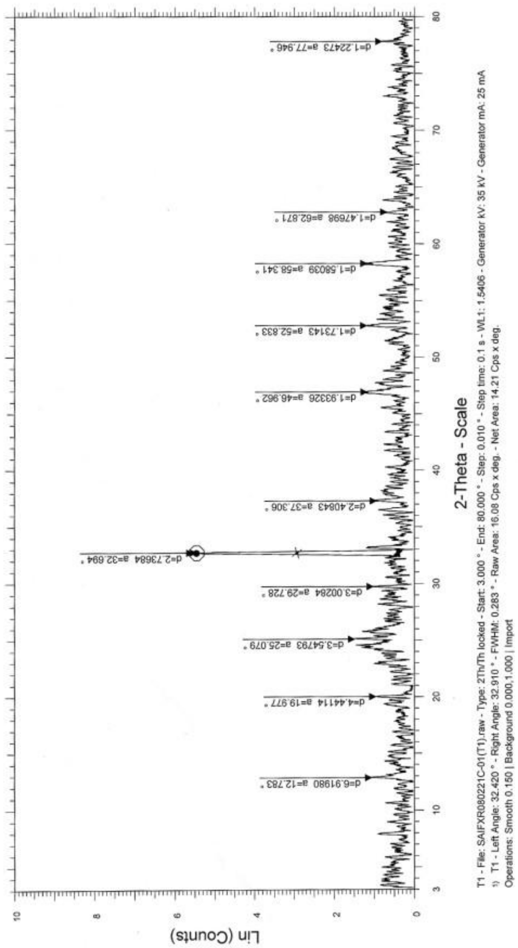

Figure 4.1: X ray diffraction report of Titanium dioxide nano particle

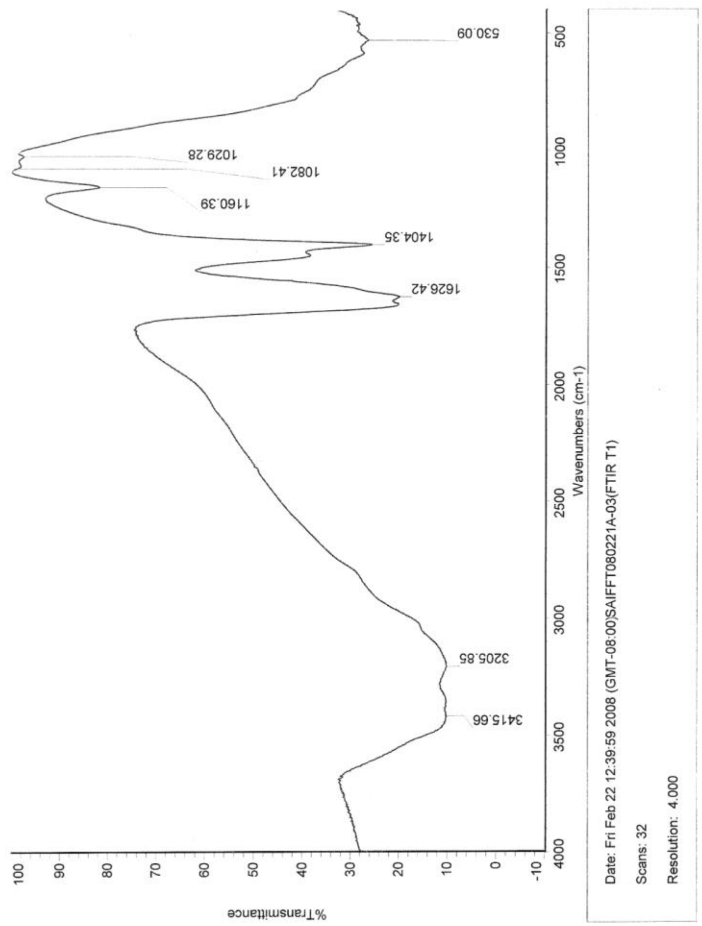

Figure 4.2: Fourier Transform Infrared Spectroscopy of Titanium dioxide nano particle

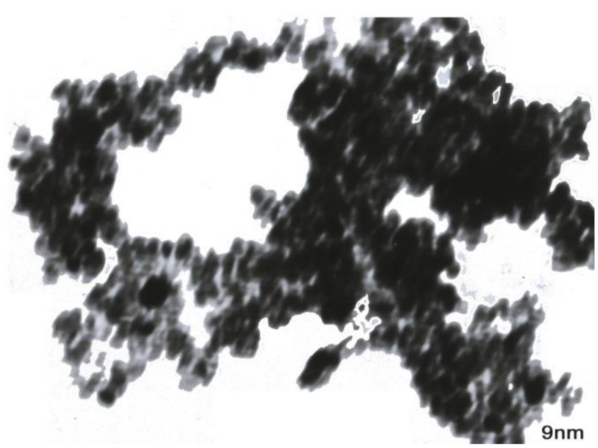

Figure 4.3: TEM image of Titanium dioxide nano particle

4.2. Analysis of TiO_2 finished fabrics using SEM

The surfaces of the treated fabrics were observed by SEM microscopy. In Figure 4.5, 4.6 and 4.7 SEM micrographs show the nano scaled TiO_2 particles by urea medium on 45/55% Polyester/cotton samples. The nanoparticles are well dispersed on the fiber surface in both cases, although some aggregated nano particles are still visible. The particles size plays a primary role in determining their adhesion to the fibers. It is reasonable to expect that the largest particle agglomerates will be easily removed from the fiber surface, while the smaller particles will penetrate deeper and adhere strongly into the fabric matrix.

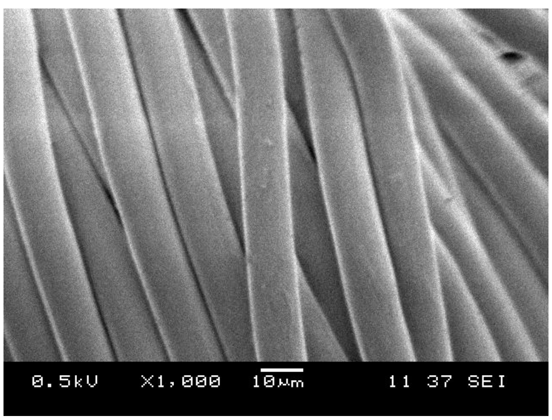

Figure 4.4: SEM image of the Untreated 45/55% Polyester/cotton fabric

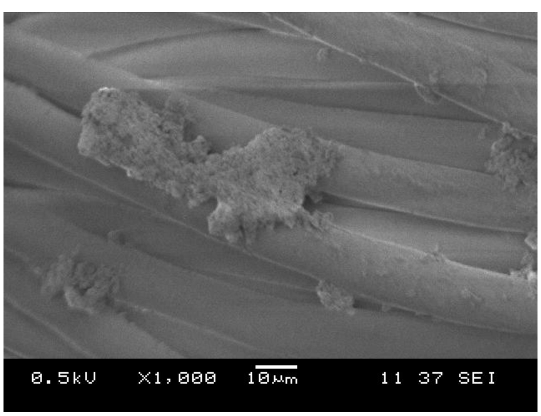

Figure 4.5: SEM image of 45/55% Polyester/cotton Woven fabric treated with 1% titanium dioxide nano particle

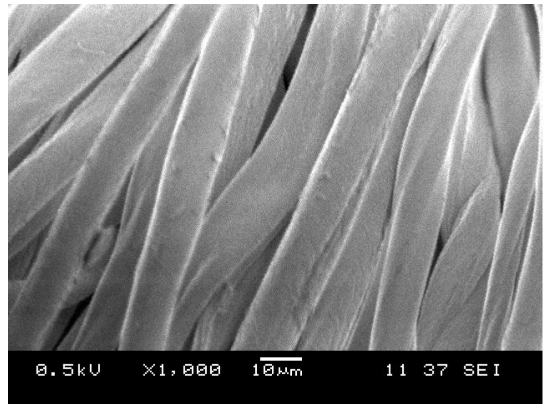

Figure 4.6: SEM image of the Untreated 100% cotton knitted fabric

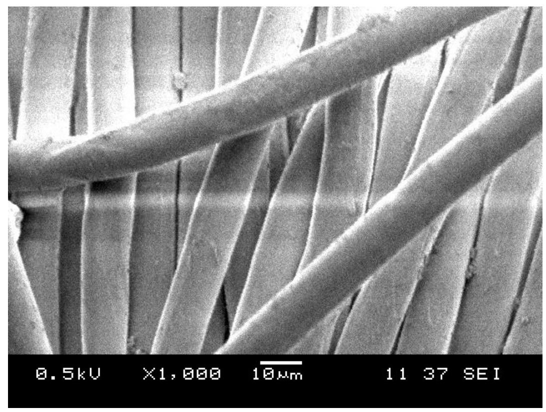

Figure 4.7: SEM image of 100% cotton knitted fabric treated with 1% titanium dioxide nano particle

4.3. Evaluation of TiO_2 treated 100% cotton and 45/55% Polyester/Cotton for Antibacterial activity

TiO_2 are preferable to other inorganic forms of titanium because of its higher efficiency in preventing infection. In the control fabric the growth of both staphylococcus aureus and Klebsiella pneumoniae was found on the fabric as well as surrounding the fabric sample**.** In TiO_2 mated fabric there was no bacterial growth on the fabric**,** but it was found surrounding the fabric sample. The woven fabrics treated with Titanium dioxide exhibit better reduction than the knitted fabrics because of their construction. Among the composition 45/55% polyester/cotton blend shows better reduction than the 100% cotton because the resistance property of polyester.

Table 4.2: Antibacterial activity of Titanium dioxide nano particle treated fabrics

Fabric samples	Staphylococcus aureus	Klebsiella Pneumoniae
Untreated	No reduction	No reduction
Woven 100% cotton	85%	64%
Woven 45/55%Polyester/Cotton	93%	78%
Knitted 100% Cotton	75%	58%
Knitted 45/55%Polyester/Cotton	79%	74%

5. CONCLUSIONS

Tio_2 nano particles were synthesized and characterized using XRD, FTIR, TEM and SEM. It is shown that the nano-Tio_2 impregnated onto woven and knitted textiles showed excellent antibacterial activity against two representative bacteria, Staphylococcus aureus and Klebsiella pneumoniae. This work provides a simple method for aqueous preparation of Tio_2 nano composites and their application onto 100% cotton & 45/55% polyester/cotton fabrics to impart antibacterial function.

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