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# DEVELOPMENT OF SELF CLEANING APPARELS

A PROJECT REPORT

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**KUMARAGURU COLLEGE OF TECHNOLOGY**

**(An Autonomous Institution affiliated to Anna University of Technology,  
Coimbatore)**

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INTERNAL EXAMINER

  
EXTERNAL EXAMINER

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## **ABSTRACT:**

Water and soil repellency has been one of the major targets for fibre and textile scientist and manufacturers. Surface modification is also an important element of a textile manufacturing on the basis of lotus leaf concept; scientist developed a new concept-self cleaning textile. Nano technology has proven its importance in almost all the fields including textile field. Nano materials defined as particle ranging from 1 to 100nm dia. have been widely utilized because of their properties. Nano science is used for imparting self cleaning capability to fabric which makes the finish more superior than other type functional finishes. The final properties of a textile material are critical in determining how they perform for their given use. Among those nano particles  $\text{TiO}_2$  is a frequently used one. This project work uses nano technology for obtaining the self-cleaning property. A low temperature of nano  $\text{TiO}_2$  using sol-gel method was used to prepare nano titanium dioxide which is applied on the fabric using pad-dry-cure method. The self cleaning properties of the treatment solution and treated cotton fabrics obtaining via dip padding process where evaluated. SEM & XRD pattern shows that the nano  $\text{TiO}_2$  produced is a nature phase. The fabrics were then characterized using SEM (scanning electron microscope), XRD (X-ray diffraction) to evaluate the nano crystalline nature. Aqueous nano dispersion of the nano  $\text{TiO}_2$  exhibits positive result as a self cleaning finishing agent for cotton fabrics.

Plasma is the fourth state of matter, the plasma treatment can be done by using the atmospheric air by vacuum, or other gases depending on the required end result. Surface modification of cotton fabric with self-cleaning property is attempted through plasma treatment. The gas used in the plasma process should possess the same property as that of the chemical used ( $\text{TiCl}_4$ ). Hence Argon (Ar) which has the same property is used in the plasma treatment.

Comparing the two results of chemical treated and plasma treated samples, the plasma treated sample is the more efficient one. Comparatively it is costlier when compared to chemical process and the time consumption is very less. Now-a-days people prefer the less time consumption and the easiest one even it is a bit costlier.

## CHAPTER - 1 INTRODUCTION

### 1.1 Chemical process:

As new material nano sized  $\text{TiO}_2$  is of great interest of many scientists in the recent years. Its small size and large specific surface area allow for certain unique and unusual physico-chemical properties more over due to its high chemical stability, non toxicity and good heat resistance. It is highly promising to be used in electronic photo catalysis water purification as well as anti bacterial and self cleaning materials. Nano  $\text{TiO}_2$  has three crystalline structures Anatase, Brookite and Rutile. Anatase structure has higher stability and is particularly more suitable to be used as a self-cleaning materials.

“Lotus leaves” is the best example of self cleaning surface the concept of self cleaning textiles is based on the lotus plant whose leaves are well known for their ability to “Self Clean” by repelling water and dirt. Now-a-day’s people are very busy in their work that they do not have time for the clean their daily wear clothes and also people who are working in kitchens having headache to wash their garments also military peoples have to survive in such drastic condition that they can’t wash their cloths. Nano technology provides a new concept self cleaning textiles which gives self cleaning as well as fresh cloth everyday this not only technically benefited but techno economically also benefitted

The field of self cleaning coating is dividing into two categories:

- Hydrophobic and,
- Hydrophilic.

These two types of coating both clean themselves through the action of water, by rolling droplets and the latter by water that carry out dirt.

Among the various ways of nano sized  $\text{TiO}_2$  synthesis the sol gel method is the most common method used synthesis of Anatase crystal type of nano  $\text{TiO}_2$  by sol-gel method at low temperature (room temperature) was reported. Finally its application in self cleaning finishing textiles.

## **1.2 Plasma:**

The technology of plasma has been developing rapidly recently. The process is more expensive when compared to chemical process and however it takes much less time than chemical process and there no effluents in the process. The field of plasma process can be divided into two types namely:

- using the gas,
- without using the gas (vacuum)

In this project work Argon gas is used to inhibit the hydrophobic nature to the surface of the cotton fabric which is otherwise is known for its hydrophilicity.

## CHAPTER -2 REVIEW OF LITERATURE

### 2.1 Self cleaning mechanism:

Nano care fabrics are created by modifying the cylindrical structure of the cotton fibres making the fabric. At the nano scale, cotton fabrics look like tree trunks. Using nano techniques, these trunks are covered in a fuzz of minute whiskers which creates a cushion of air around the fibre. When water hits the fabric, it beads on the point of the whiskers, the beads compress the air in the cavities between the whiskers coating extra buoyancy. In technical the fabric has been rendered super non wet ability or super hydrophobic.(8)

The whiskers also create fewer points of contact for dirt, when water is applied to solid fabrics, the droplet on an inclined super hydrophobic surface does not slide off; it rolls off(9). When the droplet rolls over a contamination, the particle is removed from the surface if the force of absorption of the particle is higher than the static friction force between the particle and the surface. Usually the force needed to remove a particle is very low due to the minimized contact area between the particles and surface. As the result, the droplet cleans fabric by rolling off the surface.

The self cleaning fabric work using the photo catalytic properties of  $\text{TiO}_2$ . The fabric is coated with a thin layer of  $\text{TiO}_2$  particle that measures only 17nm in dia. When this semi conductive layer is exposed to light, photons with energy equal to or greater than the band gap of the  $\text{TiO}_2$  electrons up to the conduction band(4).

#### 2.1.1 Types of self cleaning: (1)

- Using hydrophobic coatings –lotus effect.
- Using hydrophilic photocatalytic coating.
- Using silver nano particles.
- Using easy to clean finishing.

### **2.1.2 Methods of self cleaning:**

- Using photo catalyst.
- Using microwaves.
- Using carbon nano tubes.
- Using metal oxide colloidal.
- Using silver nano particles.
- Using chlorine halamine.

### **2.2 Nano finish:**

The concept of nano finish is not new it was started over 40 years ago. According to the National Nano Technology Institute, NNI, nano technology is defined as the utilization of structures with at least one dimension at nano meter size for the construction of materials, devices or systems with significantly improving properties due to their nano size(11). Nano technology not only produces small structure, but also an anticipated manufacturing technology which can give through inexpensive control of the small structure of matter. Nano finish can best be described and activities at the level of atoms and molecules that have applications in the real world. Nano particles used in commercial products are in the range of 1-100nm.

Nano finish also has real commercial potential for the textile industry. This is mainly due to the facts that conventional methods used to impart different properties to fabrics often don't lead to permanent effects, and will lose their functions after few laundering or wearing(12). Nano finished fabrics has greater durability because nano particles have a surface area- to- volume ratio and high surface energy, thus presenting better affinity for fabrics and leading to an increase in durability of the function. In addition a coating of nano particles on fabric will not affect their breathability or hand feel or harm full to skin.

The first work on nano technology in textiles was undertaken by Nano-Tex a subsidiary of the US based Wellington Industries, later more and more textile companies began to investing in the development of nano technology. (13)

Several methods can be used to apply coating onto the fabrics which includes chemical processing like spraying, transfer printing, washing, finishing and padding, of these methods padding is the most commonly used.

### **2.3 Identifying, Types and Nature of Stains: (25)**

Stains can be categorized as very light, light, moderate, severe, and very severe.

Stains have been classified in few ways:

- Organic and inorganic
- water based and oil based

And have been fitted into five sub categories:

- **ORGANIC:** Food, fruits, coffee, tea, beverages, cosmetics and tobacco.
- **OIL BASED:** Greasy foods, cooking oils, grease, tar, and skin and hair oils.
- **INK/DYES:** Felt tip markers, pens, natural and synthetic dyes.
- **BIOLOGICAL:** Vegetarian, mold, mild dew, algae, fungus, etc.,
- **METALLIC:** Iron (rust), copper, bronze, etc.,

#### **2.3.1 Stain color:**

- **Black colour:** Grease, tar, ink, shoe polish, moss, fungus, mildew, animal feces, vegetarian, dirt
- **Brown shades:** Coffee, tea, foods, chocolate, Grease, oil, algae, moss, vegetarian dirt, wood(tannin), Tobacco, urine, animal feces, rust, copper, bronze, dyes, plant fertilizers.
- **Red shades:** Foods, fruit drinks, blood, ink, dyes, rust, plant fertilizer, dirt.
- **Orange shades:** Foods, fruit drinks, rust, dyes.
- **Green shades:** Algae, mildew, foods, ink, dyes, copper, bronze,
- **Yellow shades:** Eggs, musted oil, grease, urine, rust.
- **Amber shades:** Varnish, polyurethane.
- **Blue shades:** Plant fertilizer, ink, dyes.
- **Bluish green:** Copper, bronze.
- **Grey shades:** Aluminum, efflorescence, paper.

- White shades: Aluminum, efflorescence, plant fertilizer, paint.
- Others: Eggs, shellac, lacquer, varnish, urethane, poly urethane, paint, ink, crayon, dyes cement colouring stains, pigmented sealers, coloured past waxes, coloured grouts.

### 2.3.2 Stain removing techniques:

- Organic: mix  $H_2O_2$  with a few drops of ammonia. Pour on the stain and leave until the fizzing stops. Use brush to work in. rinse with water. If it lightness repeat. If this does not work apply the commercial poultice according to the instruction.
- Oil based: apply poultice. Oil stains usually require a few application to be removed.
- Ink/dye: for light coloured marble and limestone...and had about 1" of "DI DI7" with enough water to mix with poultice. For dark marbles wipe with acetone.
- Biological stains: spray with a solution of 3 parts bleached, one part water and a few drops of dish detergent. Scrub with a brush unit gone. Rinse thoroughly with clean water.

## 2.4 What is Plasma?

Plasma is a partially-ionized gas consisting of equal numbers of positive and negative charges and a different number of unionized neutral molecules. When a gas is subjected to a DC or radio frequency (RF) potential at reduced pressure this is usually accompanied by glow, which is known as glow discharge. The words glow discharge and plasma tend to be used synonymously, although glow discharges are not perfect plasma but for the purposes of this text they will not be differentiated. The characteristic glow of these plasmas is due to electronically excited species producing optical emission in the ultraviolet or visible regions of the spectrum and is characteristic of the composition of the glow discharge gas. For example, Argon gives a bright blue colour and air or Nitrogen gives a Pink colour that is due to excited Nitrogen molecules.

### 2.4.1 Classification of plasma: (16)

- On the basis of pressure in plasma chamber Atmospheric Pressure and low pressure plasma.
- On the basis of degree of ionization and the temperature of electrons and ions-Hot and cold plasma.

- On the basis of frequency of the power supply DC and AC plasma (RF, Microwave, GHz Plasma).

Depending upon the electron affinity of the process gases used-Electropositive and electronegative gas plasma.

Any plasma reactor will be a combination of all of the above, e.g. one atmosphere glow discharge cold plasma is based on cold, AC and atmospheric pressure plasma.

#### **2.4.2 The role of plasma treatment in sustainable development (14)**

In light of the application processes discussed in this text, plasma technology holds tremendous potential to develop processes which can limit the environmental impact of textile processing and contribute towards sustainable development. Savings with plasma treatment can be due to a variety of factors but mostly relate to conservation of water and energy as plasma treatment leads to dramatic reductions in the use of both.

Physical etching of textile substrate can be used to create nano sized peak on the surface of the fiber. This coupled with nano-layering of a hydrophobic fluoro carbon compound can be used to create the famous “Lotus effect” on textile which makes surfaces of hydrophilic fibers effectively hydrophobic while still leaving it breathable. Conventionally these treatments are performed by pad/dry/cure treatments which utilize large amounts of water and also require heat to cure the applied chemical.

In contrast plasma treatment can achieve the same effect by applying a gas such as oxygen, Argon for etching, fluoro carbon in gaseous state for nano layering by using comparatively very little electrical power and also water repellency properties.

Though these finishes can't yet produce results of the same scale as conventional wet treatment, research in the field promises that results will improve in the future and will ultimately lead to the replacement of the conventional method of hydrophobization.

## CHAPTER 3      METHODOLOGY

### 3.1 Fabric particulars:

Type of fabric	=	Light weight cotton.
Fabric GSM	=	104 sq.cm
Fabric colour	=	White.
Weave	=	Plain weave.
Ends/inch	=	58.
Picks/inch	=	82.
Warp count	=	28 <sup>s</sup> .
Weft count	=	30 <sup>s</sup> .

### 3.2 Chemical process:

99.9% purity of  $\text{TiCl}_4$  is taken for the conversion of nano form of  $\text{TiO}_2$  by using the sol-gel process for imparting the self cleaning property by dip and nip method.

### 3.3 Plasma treatment:

In this work low pressure plasma machine is used for the plasma treatment fabric by using Argon gas for imparting water repellency property. The Argon gas possesses the same property as that of the nano  $\text{TiO}_2$ .

### 3.4 Tests:

- SEM (scanning electron microscope).
- XRD (X-ray diffraction).
- Stain release (AATCC TM 175).
- Soil release- oily stain release method (AATCC 130:2010).

### 3.5 Purpose of research:

Nature has already developed an elegant approach that combines chemistry and physics to create super repellent surfaces as well as self cleaning surfaces. "Lotus leaves" is the best example of self cleaning surfaces. The concept of self cleaning textiles is based on the lotus plant whose leaves are well-known for their ability to 'self-clean' by repelling water and dirt.



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Figure 1 "LOTUS LEAF EFFECT"

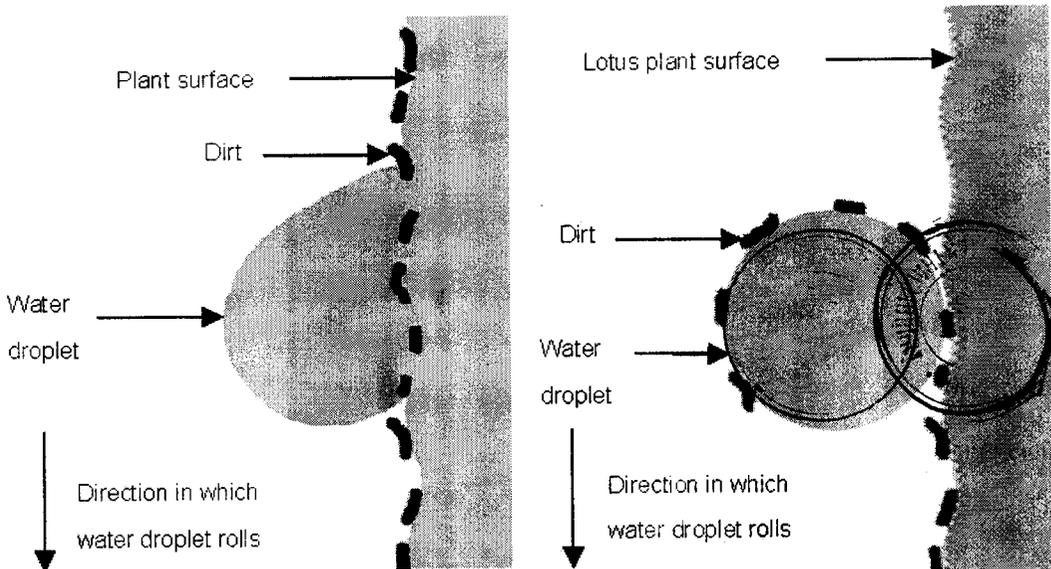
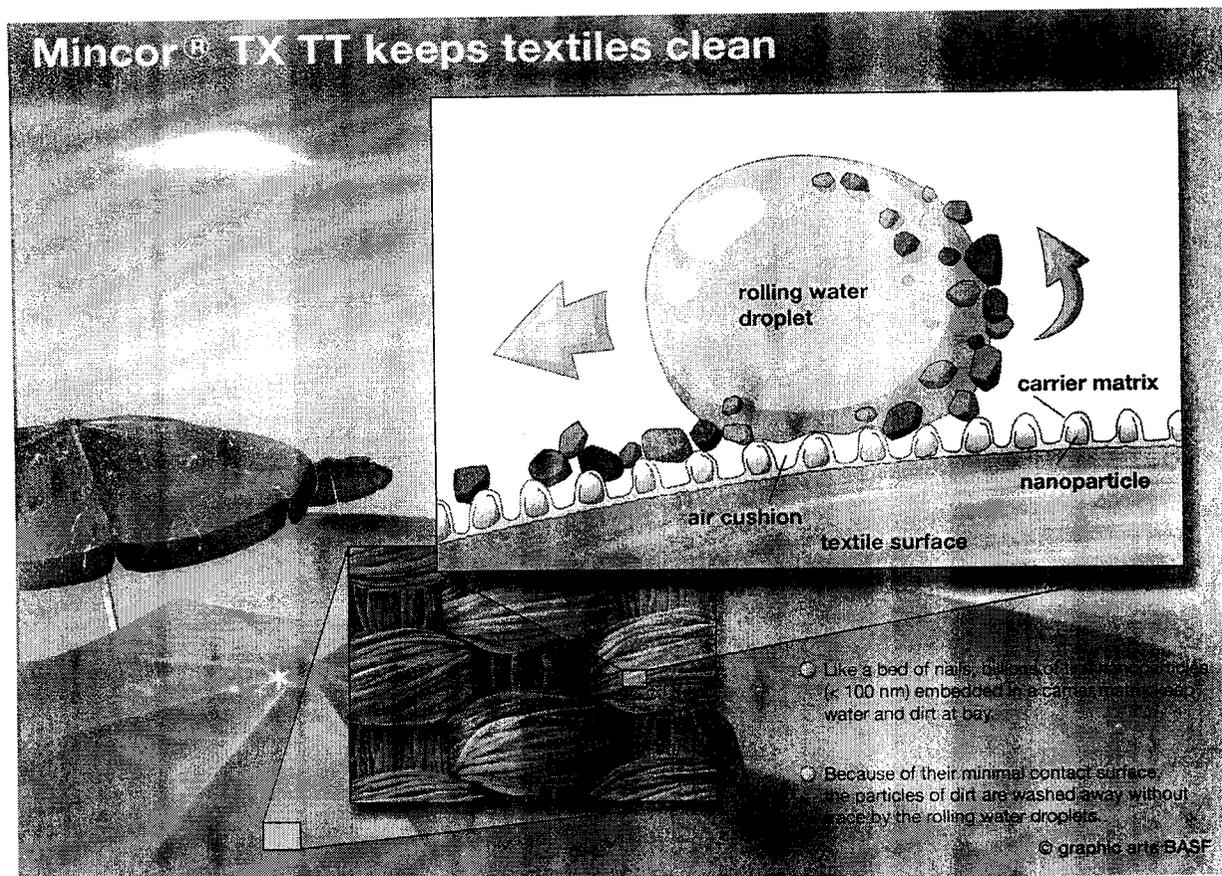
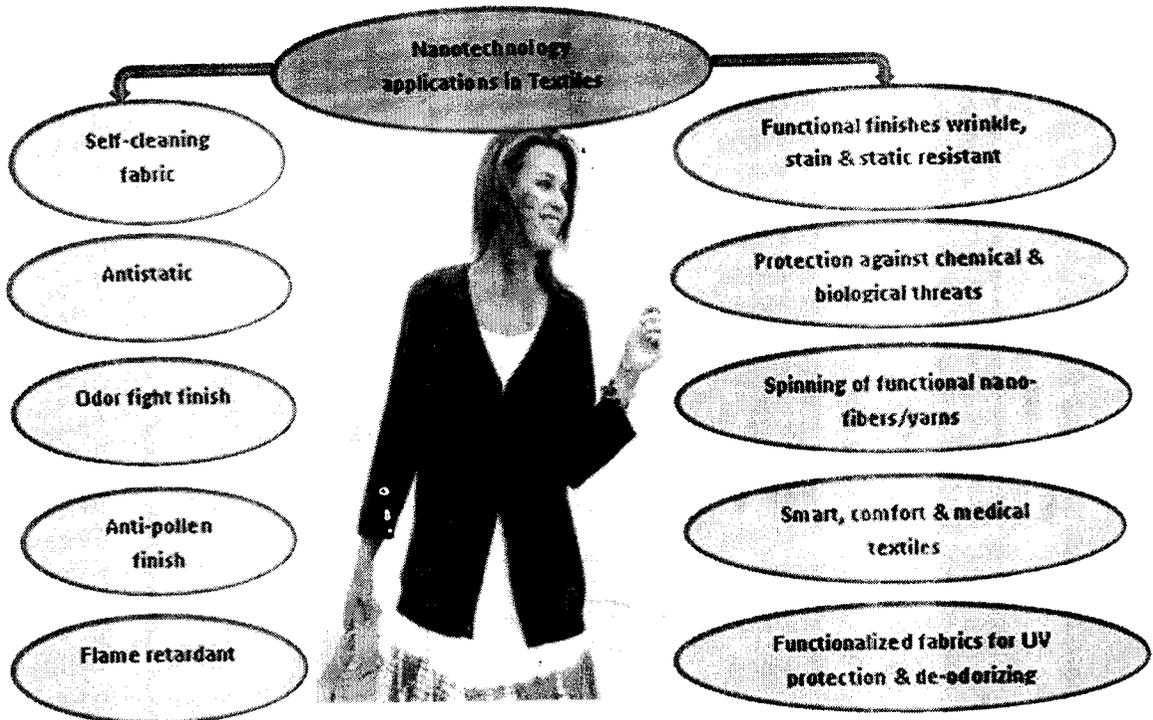


Figure 2 LOTUS LEAF EFFECT 2



Now-a-day's people are very busy in their work that they do not have time for clean their daily wear cloths. People who are working in kitchens have headache to wash their garments. Also military peoples have to survive in such drastic condition that they cannot wash their cloths. Nanotechnology (fig.) provides a new concept of self cleaning textiles which gives self-cleaning as well as fresh cloths every day, this not only technically benefited but techno economically also benefited.

Figure 3 APPLICATION OF NANO TECHNOLOGY IN TEXTILES



There are basically two types of self-cleaning surfaces involving nanotechnology. In the first place extremely water repellent, microscopically rough surfaces: dirt particles can hardly get a hold on them and are, therefore, removed by rain or by a simple rinse in water. The second example is given by photo-catalytic layers: due to a layer of nano crystalline titanium oxide, fouling organic material is destroyed by solar irradiation. Self-cleaning surface having a water contact angle greater than  $150^{\circ}$  and a very low roll off angle. Water through these surfaces easily rolls off and completely cleans the surface in the process. Self-cleaning fabrics not only resist coffee and red wine stains but are also repellent to water, dirt, odor and are antibacterial as well.

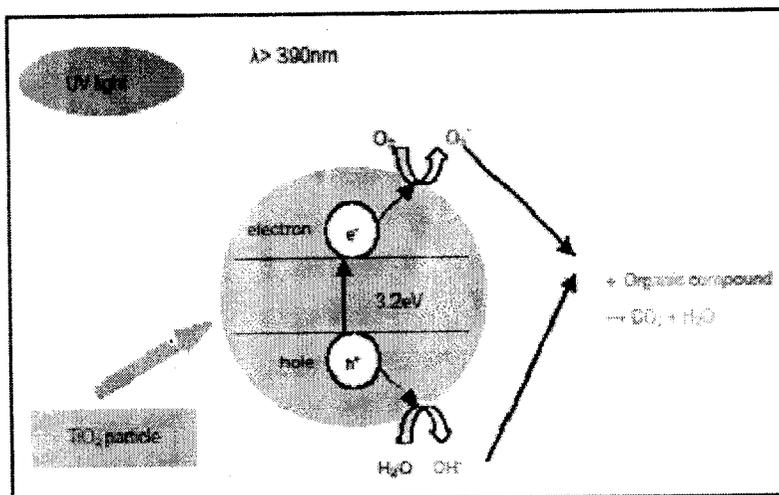
## The manufacturing of self-cleaning textiles uses the following nanotechnology:

- Photo catalyst
- Microwaves
- Carbon nano tubes
- Metal oxide colloidal
- Silver nano particles

### 3.6 Selection of Titanium Dioxide

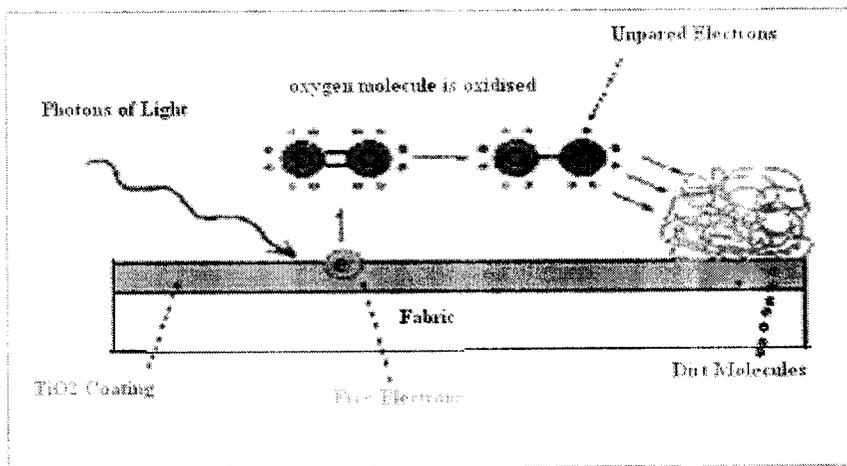
Titanium dioxide is a photo catalyst; when it is illuminated by light of energy higher than its band gap, electrons in  $\text{TiO}_2$  will jump from the valence band to the conduction band, and the electron ( $e^-$ ) and electric hole ( $h^+$ ) pairs will form on the surface of the photocatalyst. The negative electrons and oxygen will combine to form  $\text{O}_2^-$  radical ions, whereas the positive electric holes and water will generate hydroxyl radicals  $\text{OH}^\cdot$ . Since both products are unstable chemical entities, when the organic compound falls on the surface of the photocatalyst, it will combine with  $\text{O}_2^-$  and  $\text{OH}^\cdot$  and turn into carbon dioxide ( $\text{CO}_2$ ) and water ( $\text{H}_2\text{O}$ ). This cascade reaction belongs to the oxidation-reduction class and its action is schematically illustrated in Figure

Figure 4 PHOTOCATALYST



The self-cleaning fabrics work using the photo catalytic properties of titanium dioxide, compound used in many new nanotechnology solar cell applications. The fabric is coated with a thin layer of titanium dioxide particles that measure only 20 nanometers in diameter. When this semi-conductive layer is exposed to light, photons with energy equal to or greater than the band gap of the titanium dioxide excite electrons up to the conduction band. The excited electrons within the crystal structure react with oxygen atoms in the air, creating free-radical oxygen. These oxygen atoms are powerful oxidizing agents, which can break down most carbon-based compounds through oxidation-reduction reactions. In these reactions, the organic compounds (i.e. Dirt, pollutants, and micro organisms) are broken down into substances such as carbon dioxide and water. Since the titanium dioxide only acts as a catalyst to the reactions, it is never used up. This allows the coating to continue breaking down stains over and over as shown in figure.

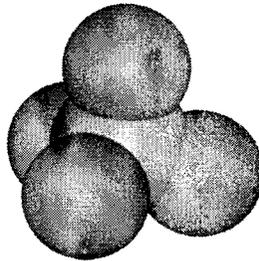
**Figure 5 SELF CLEANING ACTION**



### 3.7 Chemical process:

#### 3.7.1 Properties of $\text{TiCl}_4$ :

Figure 6 Space full model of titanium tetrachloride



Molecular formula:	$\text{TiCl}_4$
Molar mass:	189.71g/mol.
Appearance:	colorless fuming liquid.
Density:	1.726gms/cm <sup>3</sup> .
Melting point:	-24.8 degree Celsius.
Boiling point:	136.4 degree Celsius.
Solubility:	soluble in ethanol.
Viscosity:	$8.27 \times 10^{-4}$ Pascal.
Brand name:	spectra 99.99 %

### 3.7.3 Conversion process of $\text{TiCl}_4$ into nano $\text{TiO}_2$ :

#### Chemicals and apparatus used:

1. Titanium tetra chloride.
2. Ethanol.
3. Urea.
4. Magnetic stirrer.
5. Heater
6. Burette.

$\text{TiO}_2$  is an important material and has many applications; SOL-GEL process has many advantages over conventional properties methods. In the present work, the advantages of SOL-GEL process have been exploited to prepare nano crystalline  $\text{TiO}_2$  powder. Analytical grade precursor chemicals of  $\text{TiCl}_4$  was diluted with ice cooled 120 alcohol under continuous stirring in ice bath and is labeled A. One Mol. Urea was dissolved in 100ml water labeled B. Solutions A and B are mixed under continuous stirring for 6 hours at room temperature and for 1 hour at boiling temperature to remove alcohol. The phase and the structural evaluation of the heat treated gel were monitored by XRD and SEM techniques. The average crystalline size obtained from XRD data is found to be  $\sim 17\text{nm}$ .

### 3.7.4 PROCESS FLOW CHART:

1 litre beaker is placed in trough containing ice cubes placed on magnetic stirrer.



Pour 120ml of ethanol; drop the magnetic palette into the beaker and stirred it for 30 min.



Add 60ml  $\text{TiCl}_4$  (1:2 ratios)



Urea is added at regular intervals for internal cooling purpose using burette.



This process is continued for 6 hours at room temperature.



Then the temperature is raised to  $80^{\circ}\text{C}$  to remove the alcohol for 1 hour.



Thus the nano  $\text{TiO}_2$  is obtained

For testing SEM & XRD, whether the gel is in nano form; the small amount of gel has been converted into powder by heating it in furnace at  $500^{\circ}\text{C}$  for 1 hour.

### 3.7.5 SEM RESULTS OF NANO TiO<sub>2</sub>

Figure 7 SEM RESULTS OF NANO TiO<sub>2</sub>

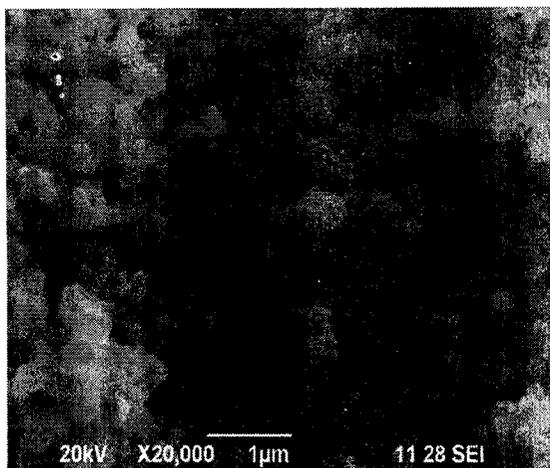
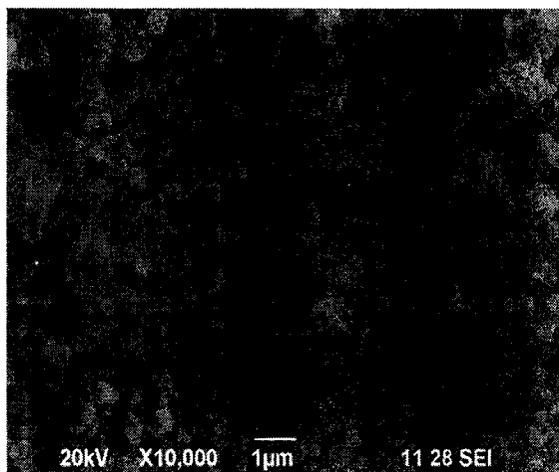


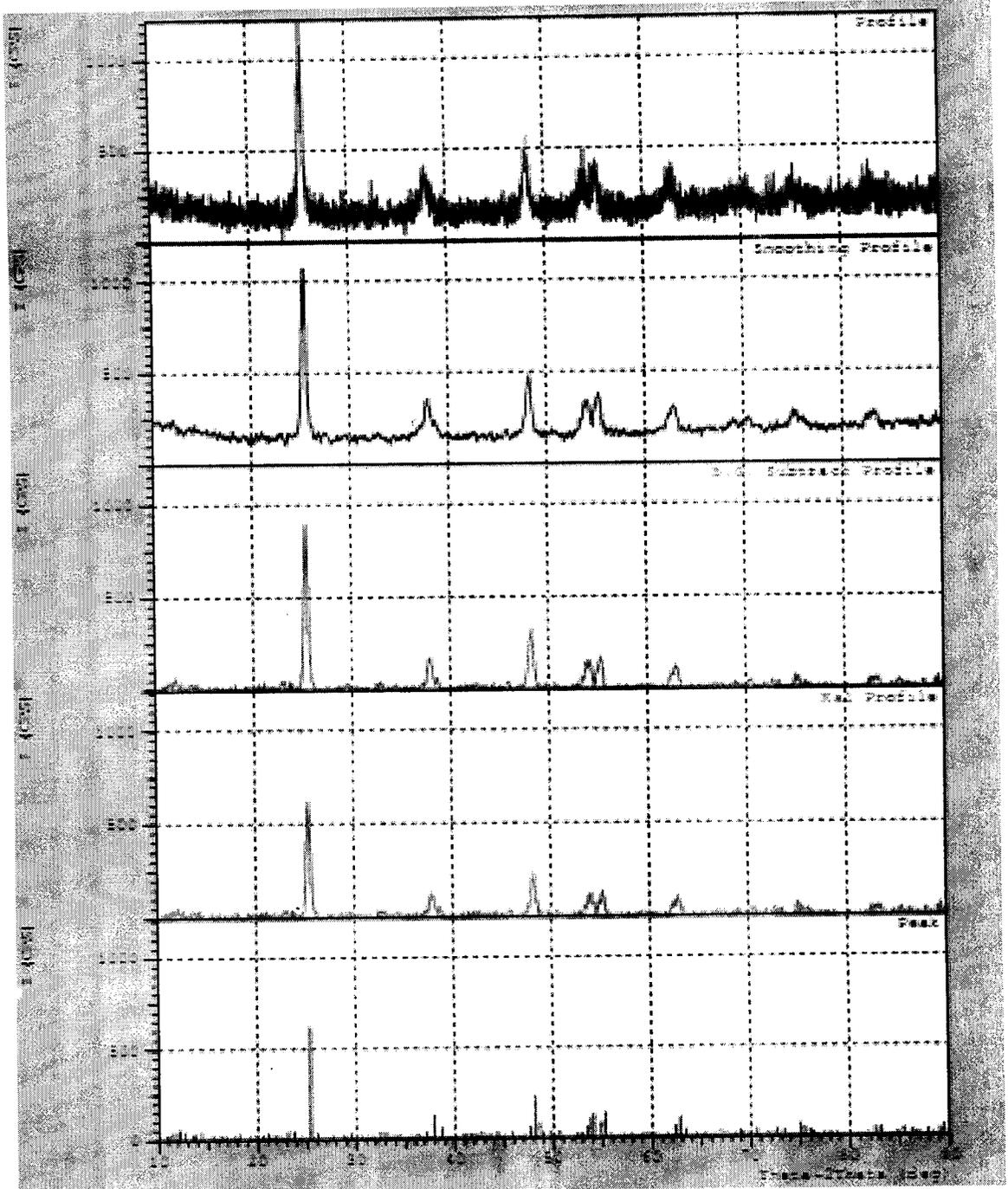
Figure 8 SEM RESULTS OF NANO TiO<sub>2</sub>



### 3.7.6 XRD RESULTS OF NANO TiO<sub>2</sub>:

Figure 9 XRD RESULTS OF NANO TiO<sub>2</sub>

< Group: 002 Data: TIO2XRD >



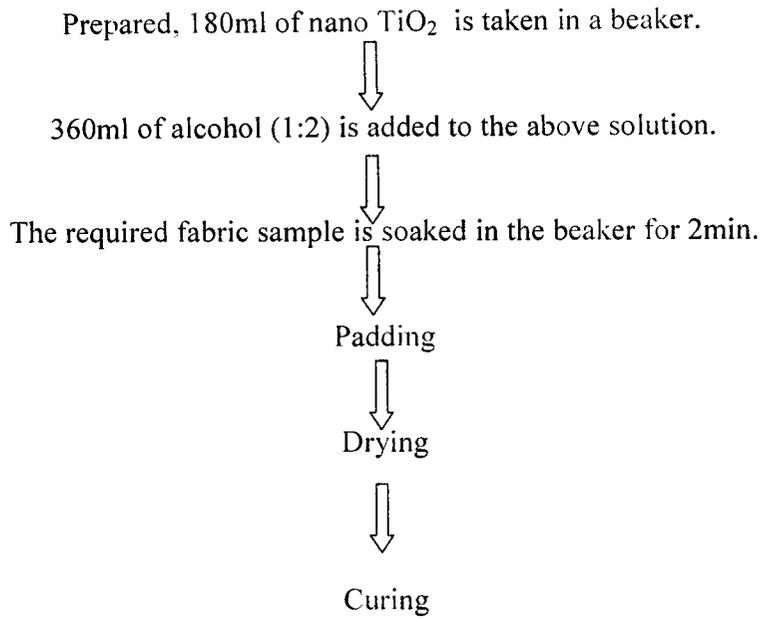
### 3.7.7 Fabric coating techniques with nano TiO<sub>2</sub> particles:

- Thermal evaporation
- Hydrothermal
- Magnetron sputtering.
- Photovoltaic
- Galvanic cell
- Spin-coating
- CVD
- Foil titanium anodize
- Electro deposition
- Electrophoresis
- Sol-gel
- Laser assisted aerosol
- Cold spray
- NPDS
- Flame spray pyrolysis (FSP)
- Spray pyrolysis (SnO<sub>2</sub> & In<sub>2</sub>O<sub>3</sub> & TiO<sub>2</sub>-WO<sub>3</sub>)
- Microwave
- Electrostatic precipitation
- Scattering
- Plasma spray

- LBL technology
- CDC method
- Dip-coating

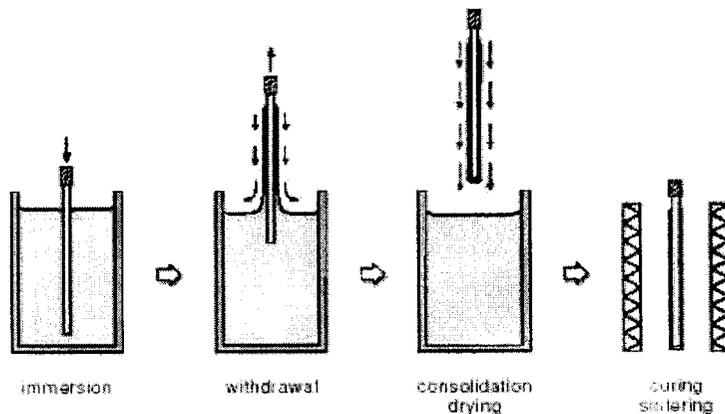
In this project SOL-GEL process is used because it is the easiest conventional method and takes short time too.

### 3.7.8 Process flow chart for coating nano TiO<sub>2</sub>:



### Coating of TiO<sub>2</sub> to the fabric:

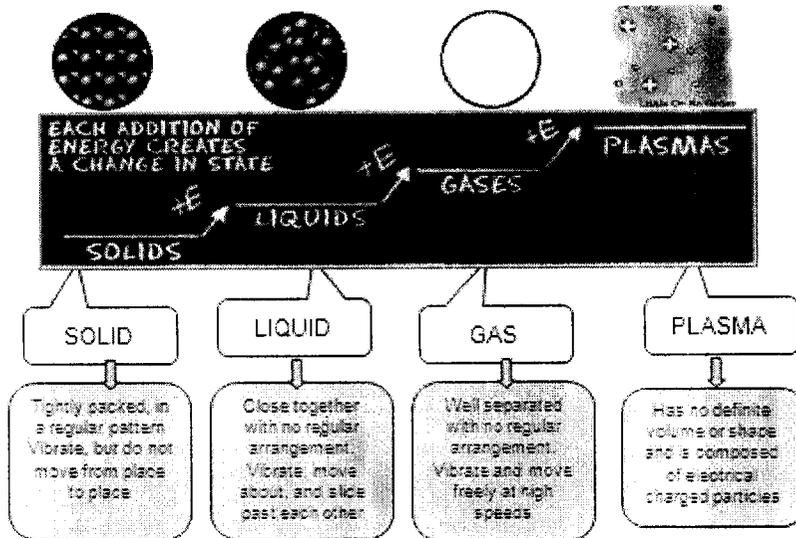
Figure 10 Coating of TiO<sub>2</sub> to the fabric



### 3.8 Plasma:

“A partially ionized gas composed of highly excited atomic, molecular, ionic and radical species, as well as photons and electrons”

Figure 11 Plasma

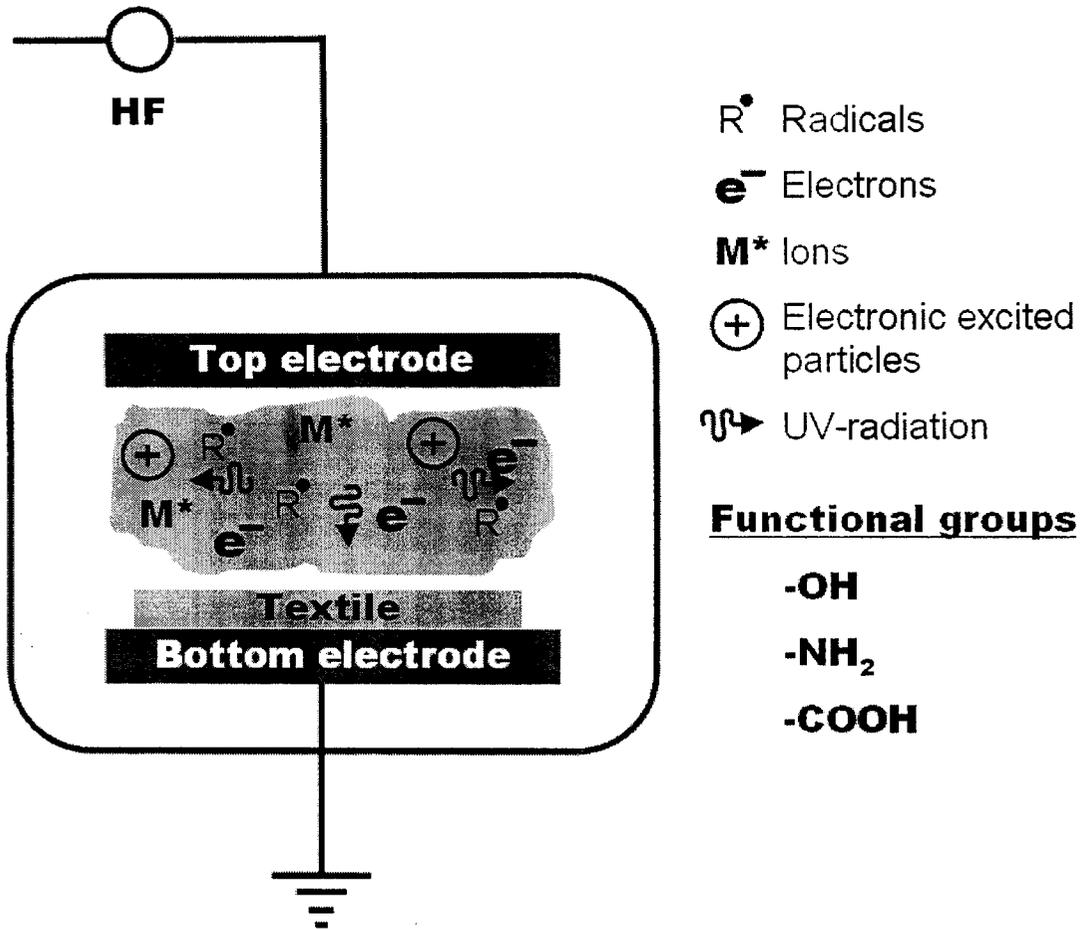


Matter within the universe is most commonly found in the form of plasma rather than as a solid, liquid or gas. Plasma is partially-ionized gas normally generated by an electrical discharge at near-ambient temperatures. Plasma, often considered as the fourth state of matter, is composed of an ionized gas containing a mixture of ions, electron, neutral and excited molecule, and photons.

This state of matter was first identified by Sir William Crookes in 1879, and named 'plasma' by Irving Langmuir in 1928.

The coupling of electromagnetic power into a process gas volume generates the plasma medium comprising a dynamic mix of ions, electrons, neutrons, photons, free radicals, meta-stable excited species and molecular and polymeric fragments, the system overall being at room temperature.

Figure 12 PLASMA PRINCIPLE



Plasma is an ionized gas, i.e. it contains electrons, ions and neutral atoms and or molecules. This allows the surface functionalisation of fibres and textiles without affecting their bulk properties. These species move under electromagnetic fields, diffusion gradients, etc. on the textile substrates placed in or passed through the plasma.

### 3.8.1 Classification of plasma treatment:

- On the basis of pressure in plasma chamber- Atmospheric Pressure and low pressure plasma.
- On the basis of degree of ionization and the temperature of electrons and ions-Hot and cold plasma.
- On the basis of frequency of the power supply DC and AC plasma (RF, Microwave, GHz Plasma).

Depending upon the electron affinity of the process gases used-Electropositive and electronegative gas plasma.

### 3.8.2 Surface modification with polymerizable monomers:

**Table 1 PLASMA TREATMENT AND THEIR EFFECTS**

Plasma Treatment	Effects
Vacuum plasma; gas: Ar monomer: acrylonitrile; on PP fabric.	PPAN (plasma polymerized acrylonitrile) surface grafted PP fabrics exhibit improve water absorption and dyeing properties.
Vacuum of 0.2 mbar; gas: Ar; monomer: Perfluoroacrylate; on cotton/PET fabrics.	Treatment for 1 min gave water repellent properties.
Vacuum; gas: Ar; monomer: acrylic acid; on PET fabric.	Increase in wettability, soiling resistance and colour strength of polyester fabric was better by argon post-plasma polymerization of acrylic acid compared to <i>in situ</i> polymerization
Atmospheric pressure; gases: He/O <sub>2</sub> ; monomer: organosilicone; on PET fabric.	Antireflection layer causes increase in colour intensity of the polymerized PET surfaces.
Vacuum; gas: Ar; monomer: fluoroacrylate in presence of vinyl crosslinking agents; on Nylon-6.	Fire-retardant coating; 50% decrease in peak value of rate of heat release.
Vacuum; pretreatment with Ar/O <sub>2</sub> followed graft polymerization by acrylamide, acrylic acids and acrylates; on celluloses, acetates, and acrylic substrates.	Grafting yield varied depending upon the parameters and monomers used; breaking strength decreased due to etching in the order: cotton > acetate > acrylic

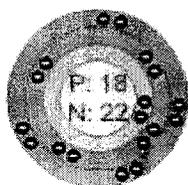
Atmospheric pressure; on PE for 10 s.	Direct grafting of perfluoroalkyltrichlorosilanes. on activated PE occurs much more readily compared to monochloro-substituted silanes.
Vacuum; gases: Ar, O <sub>2</sub> , N <sub>2</sub> and organic solvents; monomers: glycidyl methacrylate, 2,2-diphenyl-1-picrylhydrazyl; on cotton fabric for 60-300 s.	Plasma induced grafting yield with different gases and their mixture is compared.
Vacuum; gases: air, Ar and O <sub>2</sub> ; monomer: acrylic acid; on PET and polyamide fabric for 1-90 min.	In <i>situ</i> plasma polymerization of acrylic acid resulted in improved wettability, dyeability and soil resistance.
Vacuum; gases: air, Ar and O <sub>2</sub> ; monomers: acrylate containing phosphorus; on PAN fabric for 15 min.	The LOI value of PAN (18.5) increases by 4-8 units when treated with the flame retardant phosphate and phosphonate monomers.
Vacuum; gas: Ar; monomer: 1,1,2,2-Tetrahydroperfluoro decyl acrylate; on PAN fabric for 10 min.	Graft-polymerization of monomer in direct contact with the substrate surface required much smaller amounts of fluorinated reactant to achieve water and oil repellency.
Vacuum; gas: O <sub>2</sub> ; monomer: 1,1,3,3-tetramethyldisiloxane acrylate; on N-6 film for 20 min.	Rate of heat release decreased by 30%. Many different properties such as flame retardancy, damping, film deposition or hindering of additive diffusion out of the host matrix can be achieved using this in a single stage, treatment.
Vacuum; gas: Ar; monomer: acrylic acid; on PE for 1-4 min.	Considerable increase in surface free energy and wettability was observe within 1 min of plasma treatment.
Vacuum; gas: oxygen; monomer: acrylic acid; on PP.	Etching + plasma polymer coating provided enhanced electrochemical properties.

The free electron present in the nano titanium dioxide (4 electrons) is same that of the free electrons present in the Argon gas so that the properties will be the same.

According to the table no 1 the water repellency property is obtained by using Argon gas in the plasma treatment.

### 3.8.3 Properties of Argon gas:

Figure 13 ARGON GAS MOLECULE

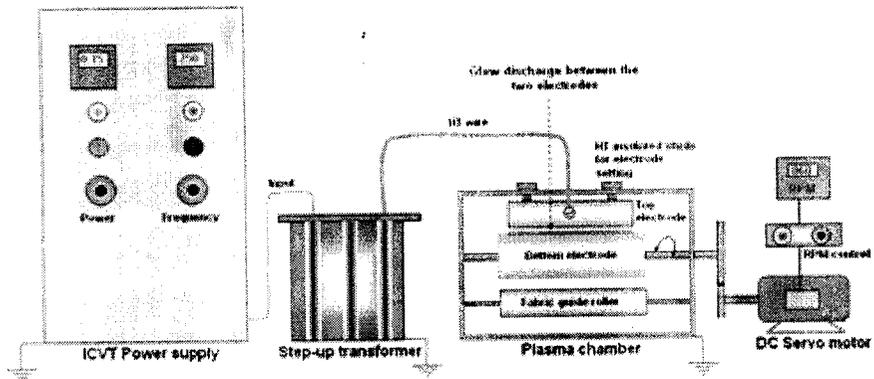


<b>Atomic number</b>	18
<b>Atomic mass</b>	39.948 g.mol <sup>-1</sup>
<b>Electronegativity according to Pauling</b>	unknown
<b>Density</b>	1.78.10 <sup>-3</sup> g.cm <sup>-3</sup> at 0 °C
<b>Melting point</b>	-189 °C
<b>Boiling point</b>	-185.7 °C
<b>Vanderwaals radius</b>	0.192 nm
<b>Ionic radius</b>	unknown
<b>Isotopes</b>	6
<b>Electronic shell</b>	[Ne] 3s <sup>2</sup> 3p <sup>6</sup>
<b>Energy of first ionisation</b>	1520 kJ.mol <sup>-1</sup>
<b>Energy of second ionisation</b>	2665.8 kJ.mol <sup>-1</sup>
<b>Energy of third ionisation</b>	3931 kJ.mol <sup>-1</sup>
<b>Discovered by</b>	Sir Ramsay in 1894

### 3.8.4 Low temperature plasma chamber

Figure 14 Low temperature plasma chamber machine

#### Low Temperature Plasma Chamber



#### Parameters of plasma treatment:

Distance between the fabric and plate:	3cm
Time required for the process:	2 min.
Pressure:	2 bar.

### **3.8.5 Plasma Process:**

The untreated fabric sample of size 50 cm x 55 cm is taken and dried using the drier to remove the moisture content of the fabric. Then the fabric is mounted to the fabric sample holder and placed in between plates (inside the plasma chamber) at the gauge length of 3cm from top and 3cm from bottom, then the door is tightly closed and the vacuum is removed completely for 2 hours till it reaches 0.05. Then the Argon gas is passed inside the chamber for 2 min.

Thus after the process completes the fabric sample is given to SEM to confirm whether the coating has been completely made onto the fabric. Only the right side of the fabric is treated with plasma.

### 3.8.6 Plasma Process flow chart:

The sample of size 50\*55cm is tightly secured on sample holder and dried using drier.



The sample holder is placed in the plasma chamber at the distance of 6cm (3cm from top & 3cm from bottom plates)



Then the chamber is closed tightly, and the vacuum is removed completely for 2 hours till it reaches 0.05.



Argon gas is passed inside the chamber and the process is done for 2 min.



Plasma treated fabric is obtained.

## **CHAPTER 4 RESULTS AND DISCUSSION:**

### **4.1. FABRIC PARTICULARS:**

Ends/inch= 58

Picks/inch= 82

Warp count= 28<sup>s</sup>

Weft count= 30<sup>s</sup>

Fabric GSM = 104sq.cm

## DRAPE:

Small disc weight=2.158g

Large disc weight=5.914g

Table 2 Drape Test:

S no	Sample 1 (Grey fabric)	Sample 2 (chemical treated)	Sample 3 (plasma treated)
01.	5.064g	5.250g	5.210g
02.	5.154 g	5.750g	5.400g
03.	5.125 g	5.512g	5.315g
<b>Average</b>	<b>5.114g</b>	<b>5.504g</b>	<b>5.308g</b>

$$\text{Drape} = \frac{w_s - w_d}{w_\infty - w_d}$$

$w_s$  = weight of projected area of specimen on drape meter.

$w_d$  = weight of small supporting disc.

$w_\infty$  = weight of specimen or large disc.

The drape test results indicate a slight decrement in the drapability of the both chemical treated and plasma treated fabrics.

## ABBRASSION RESISTANCE (constant revolution method):

Rotation=50 revolution

Weight=400grams

**Table 3** abbrasion resistance

S no	Sample 1 (Grey fabric)		Sample 2 (chemical treated)		Sample 3 (plasma treated)	
	Initial weight(g)	Final weight(g)	Initial weight(g)	Final weight(g)	Initial weight(g)	Final weight(g)
01.	0.142g	0.140g	0.145g	0.140g	0.141g	0.139g
02.	0.143g	0.138g	0.143g	0.145g	0.142g	0.137g
03.	0.145g	0.140g	0.143g	0.139g	0.143g	0.141g
04.	0.145g	0.142g	0.145g	0.142g	0.146g	0.140g
<b>Average</b>	<b>0.143g</b>	<b>0.140g</b>	<b>0.144g</b>	<b>0.141g</b>	<b>0.143g</b>	<b>0.139g</b>

**Weight loss %** = (initial weight-final weight)/initial weight x 100

**Abrasion resistance** =100-weight loss %

The abrasion test results does not reflect any significant change in the abrasion resistance property of cotton fabric for both the treatments. Hence it can be inferred that both nano chemical finish and plasma finish does not alter the surface characteristics of the fabric.

## CREASE RECOVERY:

Table 4 crease recovery

s no	sample 1 (Grey fabric)		Sample 2 (chemical treated)		Sample 3 (plasma treated)	
	Warp (degree)	Weft (degree)	Warp (degree)	Weft (degree)	Warp (degree)	Weft (degree)
01.	75 <sup>0</sup>	85 <sup>0</sup>	78 <sup>0</sup>	87 <sup>0</sup>	76 <sup>0</sup>	82 <sup>0</sup>
02.	78 <sup>0</sup>	83 <sup>0</sup>	81 <sup>0</sup>	88 <sup>0</sup>	79 <sup>0</sup>	80 <sup>0</sup>
03.	73 <sup>0</sup>	88 <sup>0</sup>	77 <sup>0</sup>	85 <sup>0</sup>	70 <sup>0</sup>	87 <sup>0</sup>
04.	76 <sup>0</sup>	84 <sup>0</sup>	79 <sup>0</sup>	86 <sup>0</sup>	74 <sup>0</sup>	84 <sup>0</sup>
<b>Average</b>	<b>75.5<sup>0</sup></b>	<b>85<sup>0</sup></b>	<b>78.7<sup>0</sup></b>	<b>86.5<sup>0</sup></b>	<b>74.7<sup>0</sup></b>	<b>83.2<sup>0</sup></b>

Similar trend can be seen in case of crease recovery property of the fabric after treating it with both nano chemical TiO<sub>2</sub> and Plasma treatment. Again it proves that both types of finishes do not affect the physical characteristics of the basic fabric. It can be inferred that both the two types of treatments have no significant influence on the crease recovery property, an important property for the fabric intended for apparel application, when compared to other finishing methods which tend to make the fabric stiffer than the untreated fabric.

### Stiffness tester:

**Table 5 stiffness test**

s no	sample 1 (Grey fabric)		Sample 2 (chemical treated)		Sample 3 (plasma treated)	
	Warp	Weft	Warp	Weft	Warp	Weft
01.	1.95cm	1.75cm	2.10cm	1.80 cm	2.00 cm	1.80 cm
02.	1.95cm	1.80cm	2.15 cm	1.75 cm	2.15 cm	1.75 cm
03.	2.05cm	1.85cm	2.00 cm	1.85 cm	1.95 cm	1.90 cm
04.	2.05cm	1.85cm	2.15 cm	1.80 cm	1.90 cm	1.85 cm
<b>Average</b>	<b>2.015cm</b>	<b>1.82cm</b>	<b>2.10 cm</b>	<b>1.80 cm</b>	<b>2.15 cm</b>	<b>1.82 cm</b>

### Fabric thickness:

**Table 6 fabric thickness**

S no	sample 1 (Grey fabric)	Sample 2 (chemical treated)	Sample 3 (plasma treated)
01.	0.015cm	0.015cm	0.015cm
02.	0.017cm	0.017cm	0.017cm
03.	0.018cm	0.018cm	0.018cm
04.	0.017cm	0.017cm	0.017cm
<b>Average</b>	<b>0.016cm</b>	<b>0.016cm</b>	<b>0.016cm</b>

The above Table clearly indicates that there is not modification to the surface of the fabric and hence there is no significant change in the thickness of the treated and untreated fabrics.

**Flexural rigidity**  $= w \times c^3 \times 10^3 \text{ mg/cm}$

**Over all Flexural rigidity**  $= (G_1 \times G_2)^{1/2}$

**Bending modulus**  $= (12G \times 10^{-6}) / g^3 \times 10^3 \text{ g kg/sq cm}$

#### 4.1.1 OVER ALL FABRIC TEST RESULTS:

Table 7 test result

FABRIC SAMPLES

--	--	--

S NO	PARTICULARS	GREY FABRIC	CHEMICAL TREATED	PLASMA TREATED
01.	ENDS PER INCH	58	58	58
02.	PICKS PER INCH	82	82	82
03.	WARP COVER FACTOR	10.96	10.96	10.96
04.	WEFT COVER FACTOR	14.97	14.97	14.97
05.	CLOTH COVER FACTOR	20.07	20.07	20.07
06.	WARP CRIMP %	5%	5%	5%
07.	WEFT CRIMP %	15%	15%	15%
08.	DRAPE	77.36%	88.98%	83.77%
09.	ABBRASSION RESISTANCE	97.91	97.9	97.2
10.	CREASE RECOVERY	Warp-75 <sup>0</sup> Weft-85 <sup>0</sup>	Warp-78.7 <sup>0</sup> Weft-86.5 <sup>0</sup>	Warp-74.7 <sup>0</sup> Weft-83.2 <sup>0</sup>
11.	FABRIC STIFFNESS (over all flexural rigidity)	63.40mg/cm	65.50mg/cm	62.5mg/cm
12.	BALLISTIC STRENGTH	Warp-75"/lbs Weft-77"/lbs	Warp-72"/lbs Weft-75"/lbs	Warp-73"/lbs Weft-76"/lbs
13.	TENSILE STRENGTH	15Nm	12Nm	9.5Nm

## 4.2 Untreated sample SEM results:

Figure 15 Untreated sample SEM results

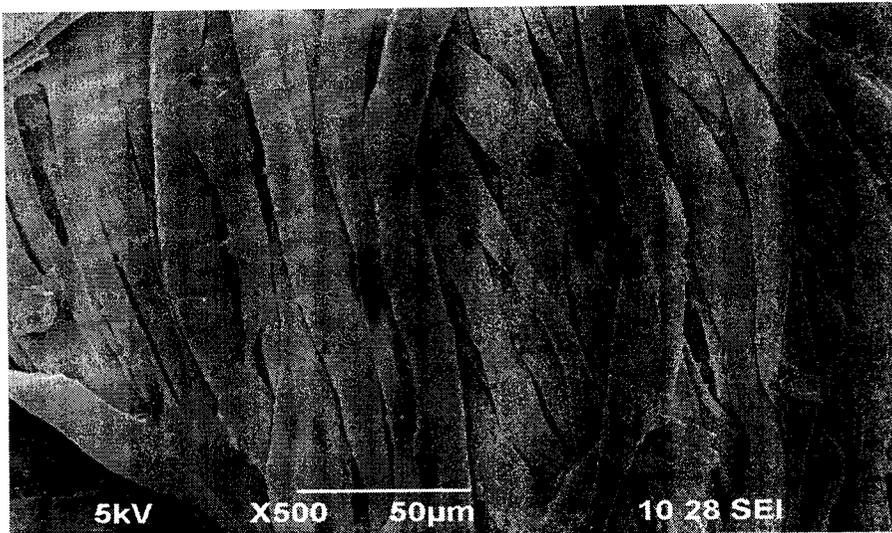
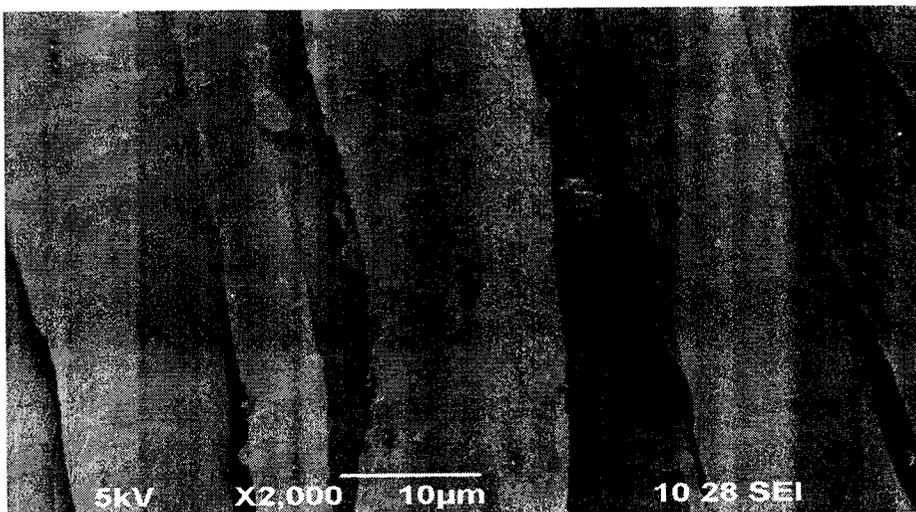


Figure 16 Untreated sample SEM results



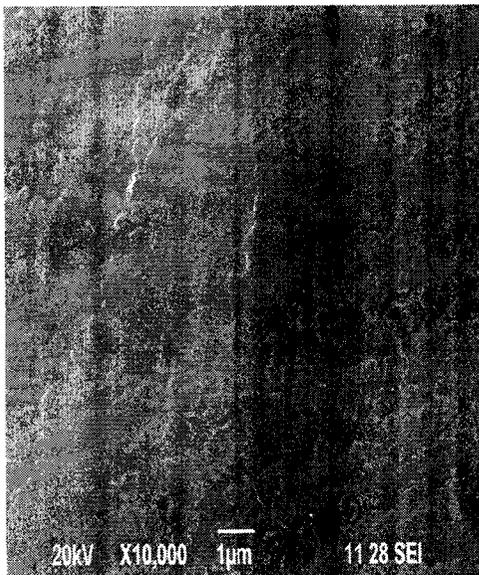
### 4.3. CHEMICAL PROCESS RESULT:

The work leads us to draw the following conclusion. The effect of modifying  $\text{TiCl}_4$  into nano form of  $\text{TiO}_2$  prepared by sol- gel process,  $\text{TiO}_2$  nano particles were successfully prepared with crystalline size of  $\sim 17\text{nm}$ . The product is calculated for 6 hours at room temperature showing an Anatase phase. The crystallinity of chemical is observed by XRD and SEM.

The chemical is coated to the fabric by padding at normal pressure, dried and cured at  $160^\circ$  celcius.

#### 4.3.1 Chemical processed fabric SEM

Figure 17 Chemical processed fabric SEM



#### 4.4. Plasma treatment:

Here the small untreated fabric which is used for the chemical process is also used for the plasma treatment (low pressure plasma). The vacuum is removed till it obtained 0.05. The Argon gas with pressure 0.2 bar is inerted. Thus the process is completed with the time period of 2minutes obtaining the water repellency finish.

Both the chemical treated and plasma treated samples are tested for the stain resistance as per AATCC standard and soil release – oily stain release method (AATCC standard).

##### 4.4.1. Plasma treated fabric SEM

Figure 18 Plasma treated fabric SEM

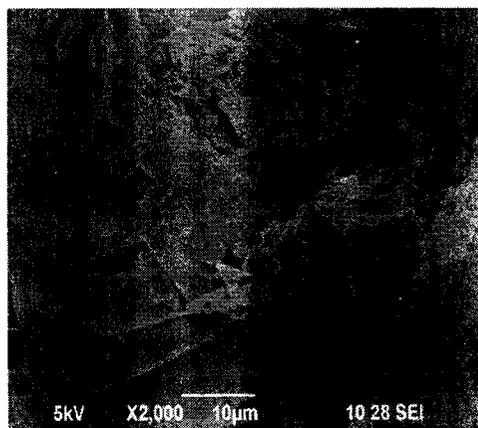
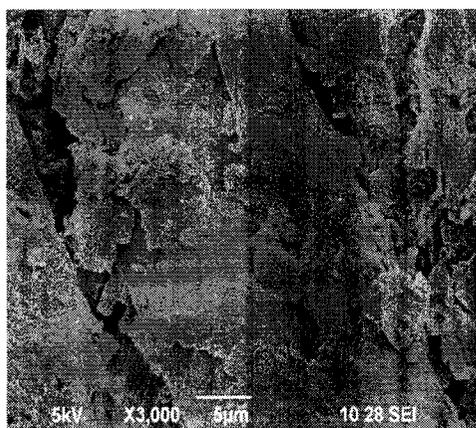


Table 8 test results

Sl no.	Type of test	Chemical treated sample	Plasma treated sample
1	Stain resistance (AATCC TM 175)	4	9
2	Soil release – oily stain release method (AATCC 130: 2010)	2.5	3.5

**Stain Resistance Test (AATCC TM 175)**

Grade 10 indicates – No staining and,

Grade 1 indicates – severe staining

**Soil release – oily stain release method (AATCC 130: 2010)**

Grade 5 indicates - no staining and,

Grade 1 indicates - severely staining

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XXIV/23090/11

31/03/2011

Mr. B. Madhan  
Kumaraguru College of Technology  
Saravanampatti  
Coimbatore - 641 006

Customer Reference : dt.29.03.2011  
Date of Receipt of Samples : 29/03/2011  
Test Report No. : ChTR.No. : 3180  
Sample Identification : CC7384, CC7385

## TEST RESULTS

Lab Code No.	CC7384	CC7385
Sample Particulars.:	Chemical Treated	Plasma Treated

Stain Resistance (AATCC TM 175)	4	9
---------------------------------	---	---

Note : The stain resistance of the test specimen by AATCC Red 40 stain scale,  
Where 10 is No staining and 1 is Severely stained.

  
HOD/Lab Incharge Textile Chemistry

Encl. : Bill

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**TEST REPORT**

**Report No. : TR:TX:2440027592**

**DATE : 31/03/2011**

**KUMARAGURU COLLEGE OF TECHNOLOGY  
CHINNAVEDAMPATTI POST, SARAVANAMPATTY  
COIMBATORE-641006  
INDIA  
CONTACT PERSON : MR.VIJAYGANESH**

**THE FOLLOWING SAMPLE(S) WAS/WERE SUBMITTED AND IDENTIFIED BY/ON BEHALF OF THE CUSTOMER AS :**

<b>SAMPLE DESCRIPTION</b>	WOVEN FABRIC 1/2 METER OF FABRIC WITH NANO TIO2 COATING SUBMITTED	
<b>COLOUR</b>	WHITE	
<b>FIBRE CONTENT</b>	100% COTTON	
<b>SAMPLE RECD ON</b>	29/03/2011	<b>TESTING PERIOD : 29/03/2011 - 31/03/2011</b>
<b>TEST(S) REQUESTED</b>	SOIL RELEASE-OILY STAIN RELEASE METHOD	

Per pro SGS India Private Ltd.

**P. M. Reddy**  
Lab Manager  
pm\_reddy@sgs.com

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**DATE : 31/03/2011**

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INDIA  
**CONTACT PERSON : MR.VIJAYGANESH**

**THE FOLLOWING SAMPLE(S) WAS/WERE SUBMITTED AND IDENTIFIED BY/ON BEHALF OF THE CUSTOMER AS :**

<b>SAMPLE DESCRIPTION</b>	WOVEN FABRIC 1/2 METER OF FABRIC WITH PLASMA COATING SUBMITTED
<b>COLOUR</b>	WHITE
<b>FIBRE CONTENT</b>	100% COTTON
<b>SAMPLE RECD ON</b>	29/03/2011
<b>TEST(S) REQUESTED</b>	SOIL RELEASE-OILY STAIN RELEASE METHOD

**TESTING PERIOD : 29/03/2011 - 31/03/2011**

Per pro SGS India Private Ltd.

**P. M. Reddy**  
Lab Manager  
pm\_reddy@sgs.com

**This is a computer generated report hence a signature is not available. An original signed hardcopy can be provided upon request at pm\_reddy@sgs.com**

JOE No. : 1144813250

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Control No.:24450

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TEST REPORT

Report No. : TR:TX:2440027584

DATE : 31/03/2011

**R E S U L T S**

SOIL RELEASE-OILY STAIN RELEASE METHOD

AATCC 130:2010  
Temperature 40°C

3.5

\*\*\*\*\* End of Report

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