

# Study of Photocatalytic Degradation Of Congo Red By Using $\text{TiO}_2$ nanoparticles

Dissertation submitted to

Lovely Professional University, India



For the partial fulfillment of the award

Of

**Masters of Science in Chemistry (Hons.)**

By

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Under the guidance of

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## **CERTIFICATE**

This is to certify that the capstone project entitled “**Study of Photocatalytic Degradation Of Congo Red By Using TiO<sub>2</sub> nanoparticles**” submitted by Ruby Gill to the Lovely Professional University, Punjab, India is documentation of research work approved under my guidance and is commendable of consideration for the degree of Masters of Science in Chemistry(Hons.) of the University.

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**Supervisor**

**Dr. Balwinder Saini**

**Assistant Professor**

## DECLARATION

I certify that

- The work enclosed on this these is innovative and has been carried out by me under the guidance of my supervisor, Dr. Balwinder Saini.
- The present work has not been submitted earlier to any other university for any degree.
- I have been following the guiding principle provided by the university in the preparation of the report.
- Whenever I have used resources (such as data, theoretical representations, any figure, and text) from other sources, I have given due recognition to them by citing them in the report and providing their details in the bibliography.

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Ruby Gill

Date:

## ACKNOWLEDGEMENT

It is my great pleasure to present report on “**Study of Photocatalytic Degradation Of Congo Red By Using TiO<sub>2</sub> nanoparticles**”. Every work skilled is pleasure wisdom. However a number of people always inspire and welcome a work with their objectives ideas and opinions.

I would like to thank all, who have helped me to do this work.

I wish to express my sincere gratitude to **Dr. Balwinder Saini** for their guidance and encouragement in carrying out this work. I would also like to thank my university for providing us with the necessary assets. I am also thankful to the Head of my Department **Dr. Ramesh Chand Thakur** for guiding and encouraging me.

**Ruby Gill**

Lovely Professional University, Punjab.

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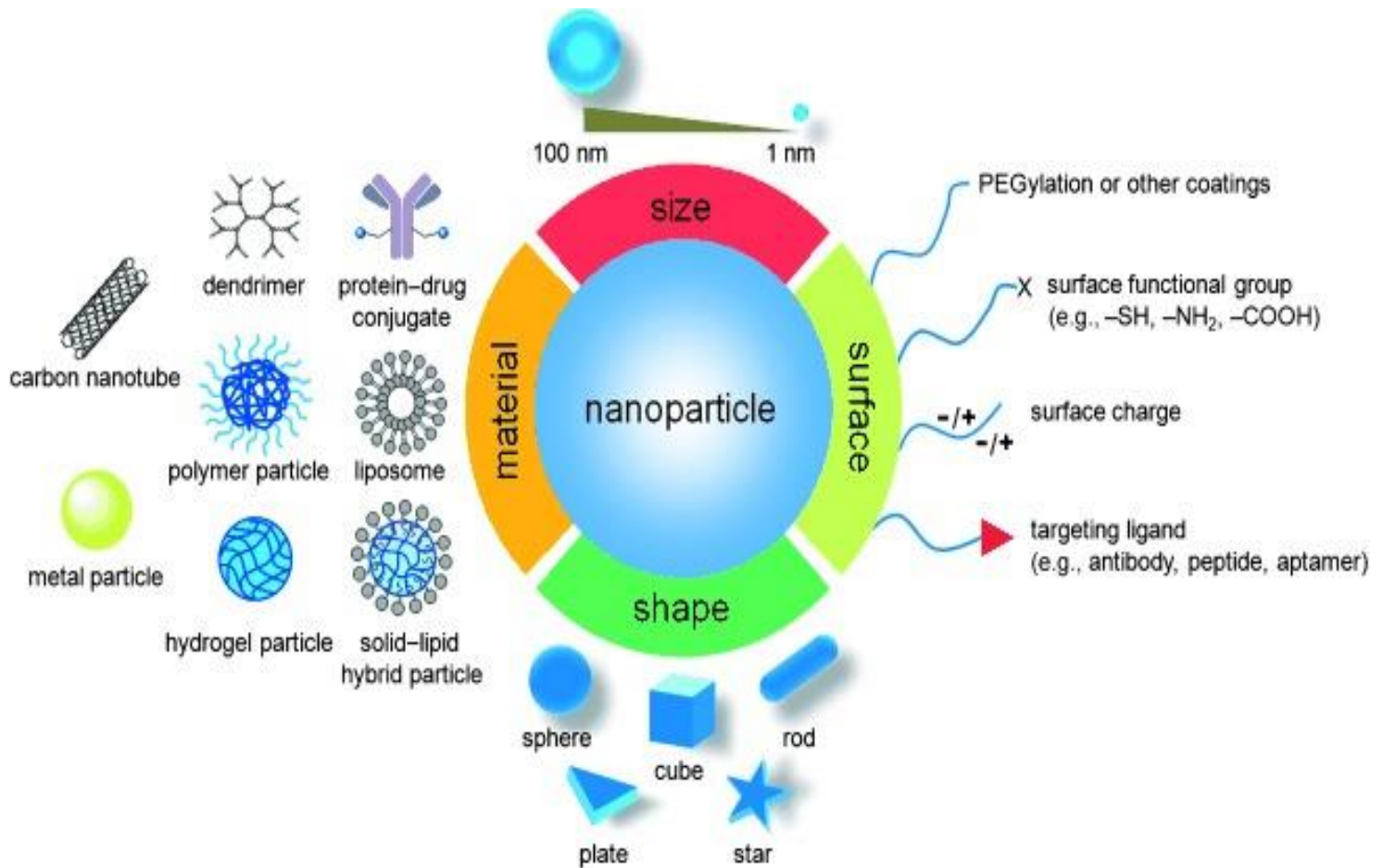
# INTRODUCTION

## DEFINITION

The metal oxide particles falling in range 1nm-100nm are considered as nanoparticles and acts as precursors for the synthesis of new material <sup>[1]</sup>

## HISTORY OF NANO-PARTICLES:

Father of nanotechnology is Richard Feynman. Although nanotechnology is recent development but the growth of its central concepts happened over a larger period of time. The concept of the nanotechnology got a boost in the early 1980s. It was led by the two chief developments that are the beginning of cluster science and the innovation of the scanning tunneling microscope. Also these developments led to the breakthrough of fullerenes in 1985 and structure of the carbon nanotubes. The first size measurements and observations of nanoparticles have been done by Richard Adolf Zsigmondy in 20th century and got a Nobel Prize. He made a study about gold sols and nanoparticles with sizes less than 10nm using an ultra-microscope. He gave the term “nanometer” for characterizing the particle size. Later on the development of Nano crystals were studied. This led to the enlargement in the number of semiconductor nanoparticles <sup>[2]</sup>.



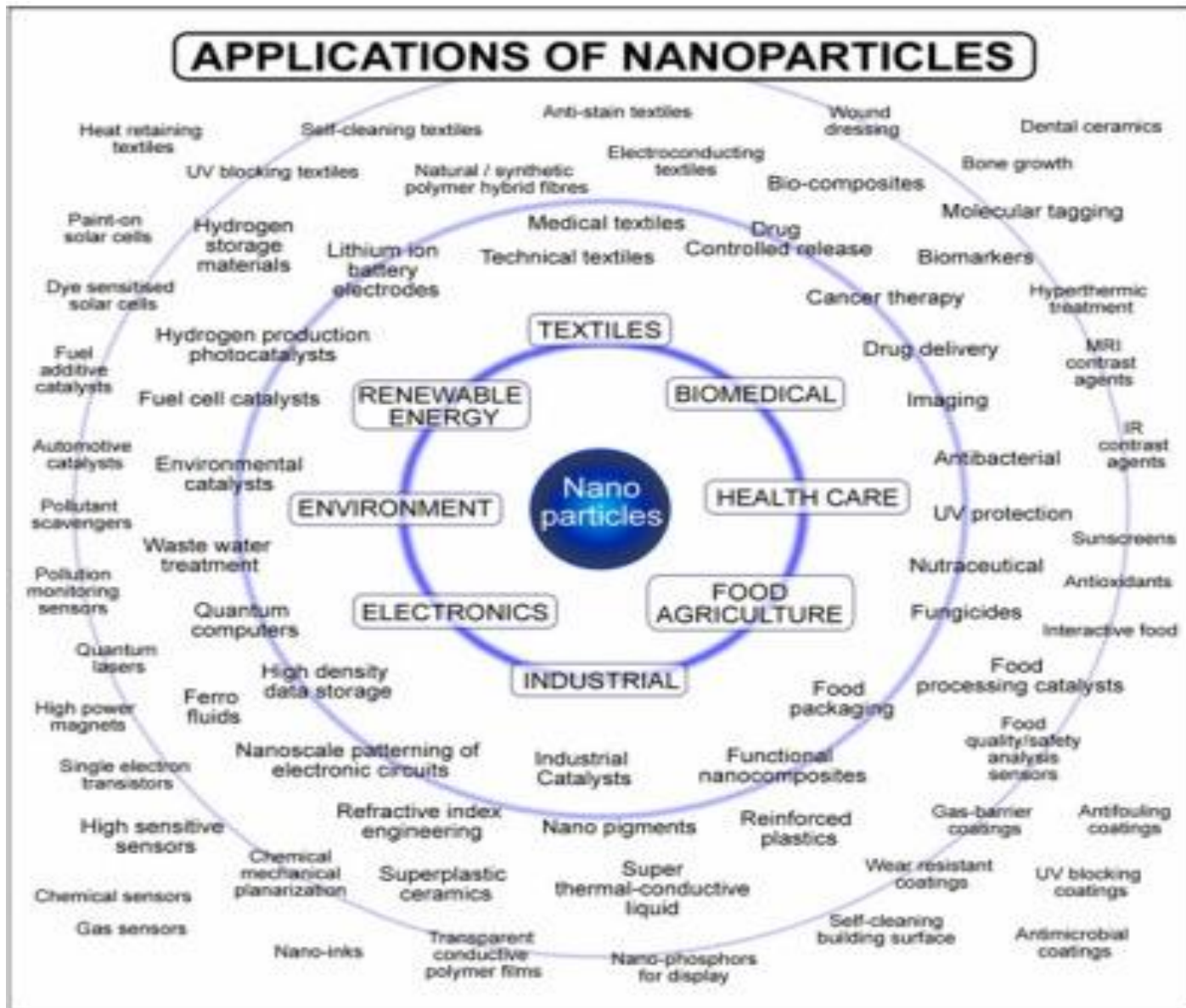
## TYPES OF NANO-FIBRES:

Nanofabrication is of two types:

**Top-down nanofabrication** – start with large material and bring it down to the nanoscale.

**Bottom-up nanofabrication** – start with individual atoms and build upwards to make a nanostructure

There is speedy development of nanotechnology which result an increasing number of nanomaterials based consumer products and industries as they have unique physical properties Nanoparticles have various application of commercial product including wound, dressing cosmetics, detergents, food packing, drug delivery, biosensors & antimicrobial coating<sup>[3]</sup>



### INTRODUCTION TO TiO<sub>2</sub> NANOPARTICLES AND ITS CRYSTALLINE PHASES:-

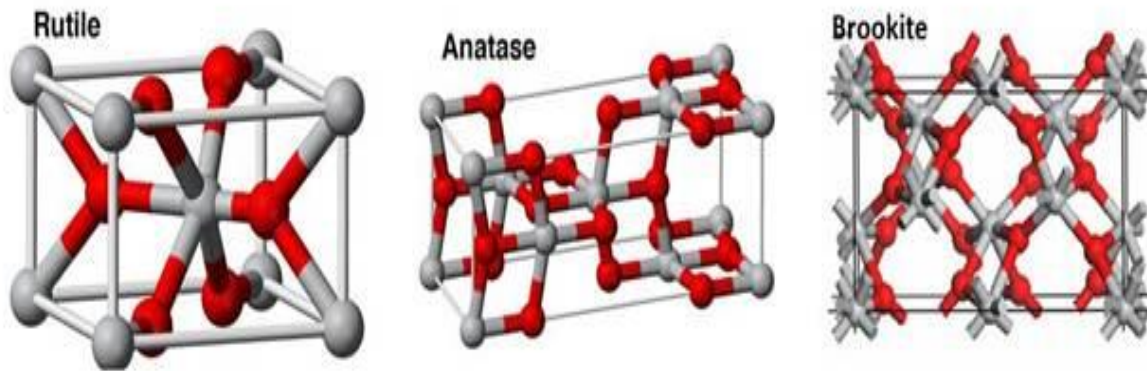
Titanium dioxide i.e. TiO<sub>2</sub> also known as Titania, has three crystalline phases. The three phases are as follows :

1. Rutile
2. Anatase



### 3. Brookite

Anatase is having photo catalytic effect, on the other hand, Rutile is used as a pigment. Most important phase is anatase and is stable at temperature lower than 600 degree Celsius. It gets converted into Rutile phase when temperature exceeds 600 degree Celsius.



Anatase, one of the crystalline form of titania, absorbs in UV region only. Rutile absorbs visible light to some extent. But rutile doesn't show properties of a good photo catalyst.

Anatase, Brookite and Rutile structure will be thermodynamically stable if their total free energies are minimized. For photo catalysis, Anatase is used, due to its highly photo reactive nature. P-25 is commercial form of titania powder containing 80% anatase and 20% rutile phase.

## ABOUT TITANIUM DIOXIDE

Many metal oxides attracting scientific research. Many scientists have concentrated on the preparation and characteristics of various metal oxide nanoparticles and also their applications in previous years.

The Titanium dioxide ( $\text{TiO}_2$ ) is a naturally occurring mineral used as a brilliant white pigment for paint, in the food industry as a coloring, in sunscreens and cosmetics, and in other industrial uses. Titanium dioxide has admirable ultraviolet (UV) resistant qualities and behaves as a UV absorbent.

The FDA has approved the safety of titanium dioxide for applying as a colorant in food, drugs and cosmetics, as well as sunscreens. However, disagreement exists as to the safety of titanium dioxide nanoparticles used in the cosmetics industry, for example in sunscreens. Titanium and zinc oxides may be made into nanoparticle size (0.2-100 nanometers) to reduce the white appearance when applied topically, but retain the UV blocking properties. Recent studies propose titanium dioxide nanoparticles may be contaminated, although further research is needed.

Many researchers have focused on the preparation and characteristics of titanium dioxide nanoparticles as well as their application. Recently widely used as inorganic physical sunscreen as they can able to reflect and scatter UV-A and UV-B radiation while preventing skin irritation & disruption of the endocrine system typically induced by chemical filters. These nanoparticles are transparent as well as pleasant to touch.<sup>[3-4]</sup> These particles can also be used in toner and coating material. It is also usually used as pigment for pharmaceutical products such as gelatin capsules, tablet coatings and syrups. In the cosmetics industry, it is used in toothpaste, lipsticks, creams, ointments and powders. It can be used as a pacifier to construct pigments opaque.

$\text{TiO}_2$  thin films have various application such as memory card capacitors, multiplayer mirrors and filters, antireflection coating, and solar energy converters<sup>[3-4]</sup>  $\text{TiO}_2$  is having properties, Chemical stability, Cheap in production. On decreasing the size of nano particles, photo catalytic activity can be increased vividly. Performance of  $\text{TiO}_2$  can be affected by changing pH of the solution, temperature conditions and time ageing. Particle nucleation and growth can be inhibited by addition of surfactant. In order to determine the properties of final titania powder obtained, the most important factors to be considered are control of size of nano particles, shape and structure of precursor used.

## METHODS OF SYNTHESIS OF TiO<sub>2</sub> NANOPARTICLES:-

We can synthesize TiO<sub>2</sub> nano particles by several methods like micro emulsion method, sol gel method, precipitation method.<sup>3</sup> Titania powder can be obtained by various methods but most common method is hydrolysis of acidic solutions of Ti (IV) salts. With sol gel technique, we can synthesize titanium dioxide nano particles by controlled nucleation at low reaction temperature.<sup>11</sup>

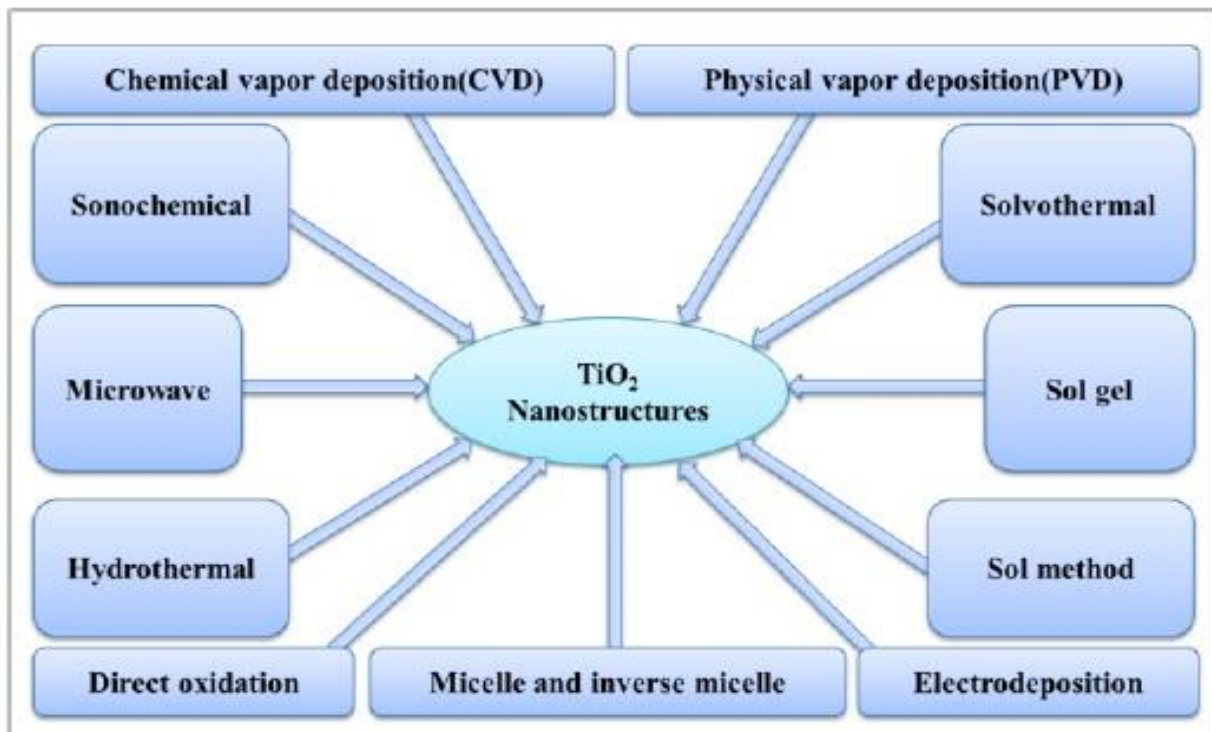


Figure 2: Common synthetic methods of TiO<sub>2</sub> nanostructures.

- **SOL – GEL METHOD :-**

Sol are the particles having size less than 50 nm which remain suspended in solution. These sol particles aggregate to form long chains in which solvent is incarcerated to give a homogeneous mixture called gel. Sol gel occurs in three steps:-

1. Hydrolysis.

2. Condensation.

3. Polymerization and particle growth.<sup>3</sup>

Cheaper and easier method for preparation of titania nano particles is sol-gel method.<sup>6</sup> In this Technique, Titania is obtained by drying of titanium hydroxide in gel form. If purity and homogeneity is taken under consideration, this method is always preferred. Particle size of TiO<sub>2</sub> can be varied by Calcination Temperature, composition, impurities.<sup>12</sup>

- **PRECIPITATION METHOD:-**

TiO<sub>2</sub> powder can be synthesized using this method in which titanium tetrachloride or TTB are used as reactants which are dissolved in solvent like water. On addition of a basic solution, precipitation of product takes place. In this particular method, the surface tension of solution was affected by surfactant and particle size was examined.<sup>3</sup>

#### **APPLICATIONS/USES OF TITANIUM DIOXIDE NANOPARTICLES:-**

1.) Titania nano particles are used in cosmetics, toner, etc.

2.) Thin films of TiO<sub>2</sub> are used in memory cell capacitors, multiplayer mirror and solar energy converters.<sup>7</sup>

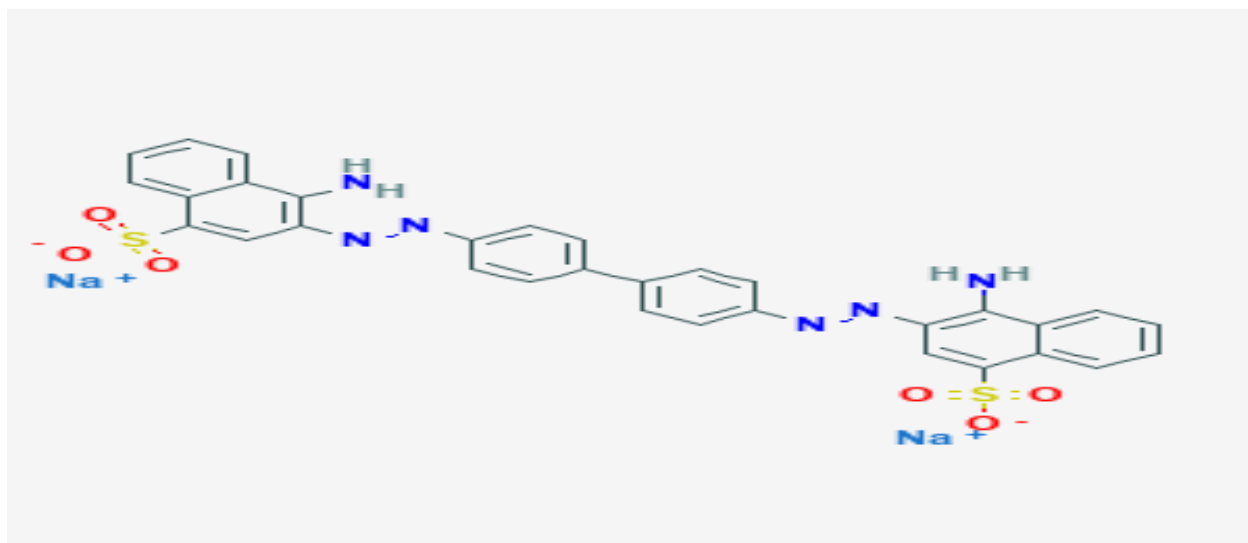
3.) titanium dioxide being non toxic in nature can be useful in photo catalytic processes in environment like preventing of strain, removal of pollutants from air and water and also in biological catalysts.<sup>6</sup>

4.) In order to degrade organic pollutants photo catalytically, titania nano particles act as a very good photocatalyst.<sup>10</sup>

- 5.) Titania can be useful in study of photoelectrochemical activity, photocatalysis<sup>8</sup> and also in solar energy conversion.<sup>13</sup>
- 6.) Growth of bacteria can be inhibited by using titanium dioxide as photocatalyst.<sup>9</sup>
- 7.) Titania nano particles doped with Ag<sup>+</sup> are used in manufacture of antibacterial plastics, coatings.<sup>14</sup>
- 8.) A nano porous film which is prepared from titanium dioxide powder is used to adsorb dye on it and an electrolyte which is constituent of solar cells.<sup>15</sup>
- 9.) As a thin film, TiO<sub>2</sub> can degrade organic pollutants.<sup>16</sup>
- 10.) Titania also possess specific properties due to which it can act as a luminescent material, used in photolysis of water as photo catalyst and for bacteriocidal action.<sup>8</sup>

## Congo Red

Congo Red is defined as the sodium salt of benzidinediazo-bis-1-naphthylamine-4-sulfonic acid which is a diazo dye which is in red color in alkaline solution and blue in color in acid solution and used as an indicator and as well as biological stain.



When the light hits the TiO<sub>2</sub> molecule, an electron gets excited, comes off and creates a free radical with the oxygen in the water which then destroys the Congo red.

# Literature review

In the 2014, Detonation method was used to prepare the mixed phase of TiO<sub>2</sub> nanoparticles consisting of anatase (34.8%~9.2 nm) and ructile (65.8 wt % ~18.2nm). To control the phases and size of nanoparticles the microstructures, crystal sizes, phase contents, thermal stability and kinetics of grain growth of mixed phase were studied. As the smaller TiO<sub>2</sub> particles at 720°C gain more kinetic energy & combine rapidly with increase in temperature and form large particles. So in this thermal treatment are used to control the particle size and phase content of mixed phase of TiO<sub>2</sub> particles <sup>[6]</sup>

In the 2014, Sol- gel method was used to synthesized the nanoparticles of nickel oxide (NiO). In this method the pH is maintain at 11 and calcinations temperature as 450°C. The morphology, structure, and particles size is studied. The cubic structure of NiO a was formed without any impurities that is confirmed by Structural analysis <sup>[7]</sup>

In 2014, In the article the nanoparticles of ZnO synthesized by simple aqueous chemical route without any capping agent. The size of nanoparticles at different calcination temperature was investigated. The decrease with increased in calcination temperature from 200°C to 500°C, the strain associated with sample gradual increase in crystallite size <sup>[8]</sup>

In 2012, the nanoparticles are synthesized by the sol-gel method. In this magnesium tetrahydrate & tartaric acid in ethanol used as precursor and then calcinated at 600°C for 6h to produce MgO nanoparticles. In this to reduce agglomeration of nanoparticles by using a cationic surfactant CTAB (cetyltrimethyl ammonium bromide). Two samples MgO and MgO-CTAB were characterized using instantaneous thermogravimetric analyzer (STA), X-ray diffraction (XRD), Field emission scanning electron microscope (FESEM). and nitrogen desorption measurement. So it was conclude that both samples have minute crystallite size that is less than 100nm & different morphology. But CTAB provide a spherical shape of MgO with less agglomerate <sup>[9]</sup>

In 2014, TiO<sub>2</sub> nanoparticles do faster photodegradation of paracetamol and an effective mineralization occurred. It was observed that more than 90% of 2.65 10<sup>-4</sup>M paracetamol was degraded under U-V

irradiation. By changing the pH values also affect the adsorption and photodegradation of paracetamol. It was found that pH 9 is optimum for photodegradation of paracetamol. <sup>[10]</sup>

In 2002, the titanium dioxide sol was synthesized using  $\text{TiCl}_4$  as precursor. In this HCL was added in the gel of hydrated titanium oxide at  $5^\circ\text{C}$ . The pH is maintained at the range of 8-12 by the aqueous sodium hydroxide. It was observed that at pH 8  $\text{TiO}_2$  particles had largest surface area of  $141\text{cm}^3/\text{g}$  and it was microporous. The concentration of acid, heating time, heating temperature can affect the morphology of  $\text{TiO}_2$ . By increases the concentration of acid the time for condensation reaction is reduce <sup>[11]</sup>

In 2012, ZnO and ZnO:  $\text{TiO}_2$  thin films with diverse thicknesses were prepared on glass substrate by by means of a sol-gel spray-spin coating method. The structural, optical, and electrical properties of ZnO thin films have been investigated and it is found to be prejudiced by the thickness of the film. The structural analysis showed that the surface morphology ZnO thin films are porous and also formed aggregates of ZnO nanoparticles measure up to composite ZnO:  $\text{TiO}_2$  thin films are homogenous, well disperse particles and have high surface area. As enlarged the thickness, the size particle does not increase. The transmittance spectra discovered that all films had low average transmittance and transparency of film decreases with the enhancement of thickness and roughness <sup>[12]</sup>

In 2014, Strontium doped  $\text{TiO}_2$  nanoparticles have been effectively synthesized by an ultrasonic hydrothermal method and post calcination treatments. Brilliant degradation efficiency of 96% was achieved within 60 minutes of photocatalytic reaction using the synthesized nanoparticles. It was found that Sr doping tremendously enhances the photocatalytic efficiency of  $\text{TiO}_2$  nanoparticles, This suggests that solar light can be utilize for the photocatalytic degradation of harmful organic contaminants existing in wastewater using alkaline earth metal doped  $\text{TiO}_2$  nanoparticles <sup>[13]</sup>

In 2014, Ni-doped  $\text{TiO}_2$  nanoparticles were prepared using hydrothermal method Photocatalytic capability of Ni-doped  $\text{TiO}_2$  nanoparticles was estimated by means of ofloxacin degradation under solar light. it was also observed that the antibacterial activity of ofloxacin was inhibited drastically against E. coli when exposed to ofloxacin solutions treated by Ni-doped  $\text{TiO}_2$  photocatalyst over short period of irradiation time. <sup>[14]</sup>

## DYE DEGRADATION:-

Degradation of various dyes by different scientists using titania nanoparticles as photocatalyst was done. Compiled data is given below in the table:-

Serial number	References	Catalyst used	Dye to be degraded	Source of light used	Observed degradation
1.	Puzenat et.al. (2003) <sup>17</sup>	P 25 TiO <sub>2</sub>	Congo red	UV - Philips HPK - 125W	Complete degradation in 6 hours.
2.	Ling et.al. (2004) <sup>18</sup>	Sol gel method was used to synthesize anatase TiO <sub>2</sub>	Methylene blue	UVL-28 lamp	50% degradation of dye was observed.
3.	Wahi et.al. (2005) <sup>19</sup>	Anatase TiO <sub>2</sub>	Congo red	14UVA lamp	Spherical shaped anatase was showing more photocatalytic activity than rod and flower like TiO <sub>2</sub> .
4.	Yang et.al. (2006) <sup>20</sup>	TiO <sub>2</sub> by sol gel technique	Methyl orange	Hg lamp, 125W	Half of the dye was degraded in 2 hours.
5.	Subramani et.al. (2007) <sup>21</sup>	TiO <sub>2</sub> by hydrothermal reaction	Indiogo caramine	UV source of light	Pure TiO <sub>2</sub> degrades 60% of dye in 6 hours.
Serial number	References	Catalyst used	Dye to be degraded	Source of light used	Observed degradation
6.	Rashed et.al. (2007) <sup>22</sup>	P 25 TiO <sub>2</sub>	Methyl orange	Flourescent light source	Light was irradiated for 3 hours and 34.36%



				was used	degradation of ,ethyl orange was observed.
<b>7.</b>	Tayade et.al. (2007) <sup>23</sup>	TiO <sub>2</sub> by hydrothermal process	Methylene blue,malachite green	Hg vapour lamp	Complete degradation of both the dyes in 6 hours was observed.
<b>8.</b>	Hussein et.al. (2008) <sup>24</sup>	TiO <sub>2</sub> from BDH limited was used	Thymol blue	Solar light	2 hours irradiation of solar light decolorized the dye about 86%.
<b>9.</b>	Hosseinnia et.al. (2010) <sup>25</sup>	TiO <sub>2</sub> by precipitation method	Methyl violet, Methylene blue, Methyl red, Rhodamine B, Sudan blue, Methyl orange	Halogen lamp	Methyl violet was completely degraded in 2 hours. Rate of degradation :- Methyl violet > Methylene blue > Rhodamine B.
<b>10.</b>	Yao et.al. (2010) <sup>26</sup>	TiO <sub>2</sub> was synthesized by tetrabutyl titanate	Methylene blue	UV light, Hg lamp	92% degradation of methylene blue was observed.
<b>11.</b>	A.R. Khataee et.al. (2009) <sup>27</sup>	TiO <sub>2</sub>	C.I. Acid Orange 10 (AO10), C.I. Acid Orange 12 (AO12) and C.I. Acid Orange 8 (AO8)	UV lamp	AO10 >AO12 >AO8 i.e. rate of decolorization of dyes used.

12.	Ammar Houas et.al. (2001) <sup>28</sup>	Titania Degussa P-25	Methylene blue	UV light source	Methylene blue is degraded at room temperature.
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### ANTI MICROBIAL/ANTI BACTERIAL ACTIVITIES BY TiO<sub>2</sub> :-

Guifen et.al. studied the antibacterial activities shown by TiO<sub>2</sub> on two bacteria that were Escherichia coli and Bacillus Megaterium. The bacteria were inhibited to an appreciable extent. It was concluded that small size, large surface area and large band gap of synthesized titania nanocomposites was responsible for good antibacterial activity and killing effectiveness of about 60 – 100 %.<sup>9</sup> Shao Feng Chen et.al. synthesized Ag doped Titania nanocomposites and observed antibacterial activity shown by them. Activity was studied with bacteria named E.coli . Inhibition of E.coli's growth can be done by Ag-doped TiO<sub>2</sub> nanoparticles even at a very small concentration of Ag in those nano particles i.e. 10µg/mL .Tremendous antibacterial properties of Ag-doped TiO<sub>2</sub> was due to electric attraction between E.coli (negative) and positively charged Ag nanoparticles.<sup>29</sup> Qilin Cheng et.al. adapted the TiO<sub>2</sub>/Ag<sup>+</sup> nanoparticles by γ-aminopropyltriethoxysilane (APS) using a method i.e. grafting. By different methods of characterization like AES, FT-IR, TEM and TGA, chemical bonding of APS to silver doped titania nanoparticles. Further PVC was added to the APS – Ag<sup>+</sup>/TiO<sub>2</sub> nanoparticles and antibacterial activity was studied. It was seen that changing the surface didn't affect the antibacterial property of nanocomposites . PVC – Ag<sup>+</sup>/TiO<sub>2</sub> also showed good antibacterial activity.<sup>14</sup> Yali Yual et.al. using hydrolysis method, synthesized titania nanoparticles. Doping of silver to resulted into broad diffraction peak. Doping with N yielded increased size of nanocomposites while Ag doping led to decreased size. In order to study the antibacterial activities agar diffusion method was worn and the bacteria were E.coli and Bacillus subtilis. Enhancement in antibacterial properties were noted in both the cases i.e. N- and Ag- doped titania. After cultivation of one complete day, 1% Ag-N-TiO<sub>2</sub> gave highest antibacterial activity with E.coli (circle 33.0 mm) and Bacillus subtilis circle = 22.8 mm).<sup>30</sup> A. A. Ashkarran et.al.

prepared silver doped titania nanoparticles and characterized them by instruments like XRD, BET, XPS, UV-Vis. Growth of E.coli was inhibited by nanoparticles showing antibacterial activity under visible light. Results showed that 0.15 g of nanocomposites prepared showed highest activity towards the bacteria. Ag is trapped in TiO<sub>2</sub> band gap and hence under visible light irradiation the antibacterial activity was improved in case of silver doped titania nanoparticles.<sup>31</sup> Saeed Rezaei-Zarchi et.al. studied the antibacterial activity shown by 0.01, 0.5 and 1% TiO<sub>2</sub> and CdO towards E.coli after culturing it in agar medium. Cell of E.coli were found dead completely after 13 hours in presence of 1% TiO<sub>2</sub> nanoparticles while in presence of 1% CdO, it took 15 hours for its death. 0.01% solutions of both nanocomposites gave unconsiderable results. It was concluded that titania nanoparticles were more competent than CdO nanoparticles as far as antibacterial activity is concerned.<sup>32</sup>

# Objectives

- The aim of our project is to synthesize  $\text{TiO}_2$  nanoparticles using Titanium tetrachloride as starting material at different CTAB concentrations i.e. 0.01 M ,0.002M 0.005 M .
- To see the effect of CTAB concentration on synthesized nanoparticles.
- To study the degradation of Congo Red using  $\text{TiO}_2$  and comparing the results at different concentrations of CTAB.
- To study the application of  $\text{TiO}_2$  showing antimicrobial activities against microbes and bacteria.

# MATERIAL AND METHODS

**Chemicals-** Titanium tetrachloride ( $\text{TiCl}_4$ , purity > 99.9 %)

Sodium hydroxide ( $\text{NaOH}$ , > 96%)

Hydrochloric acid ( $\text{HCl}$ , 35 %)

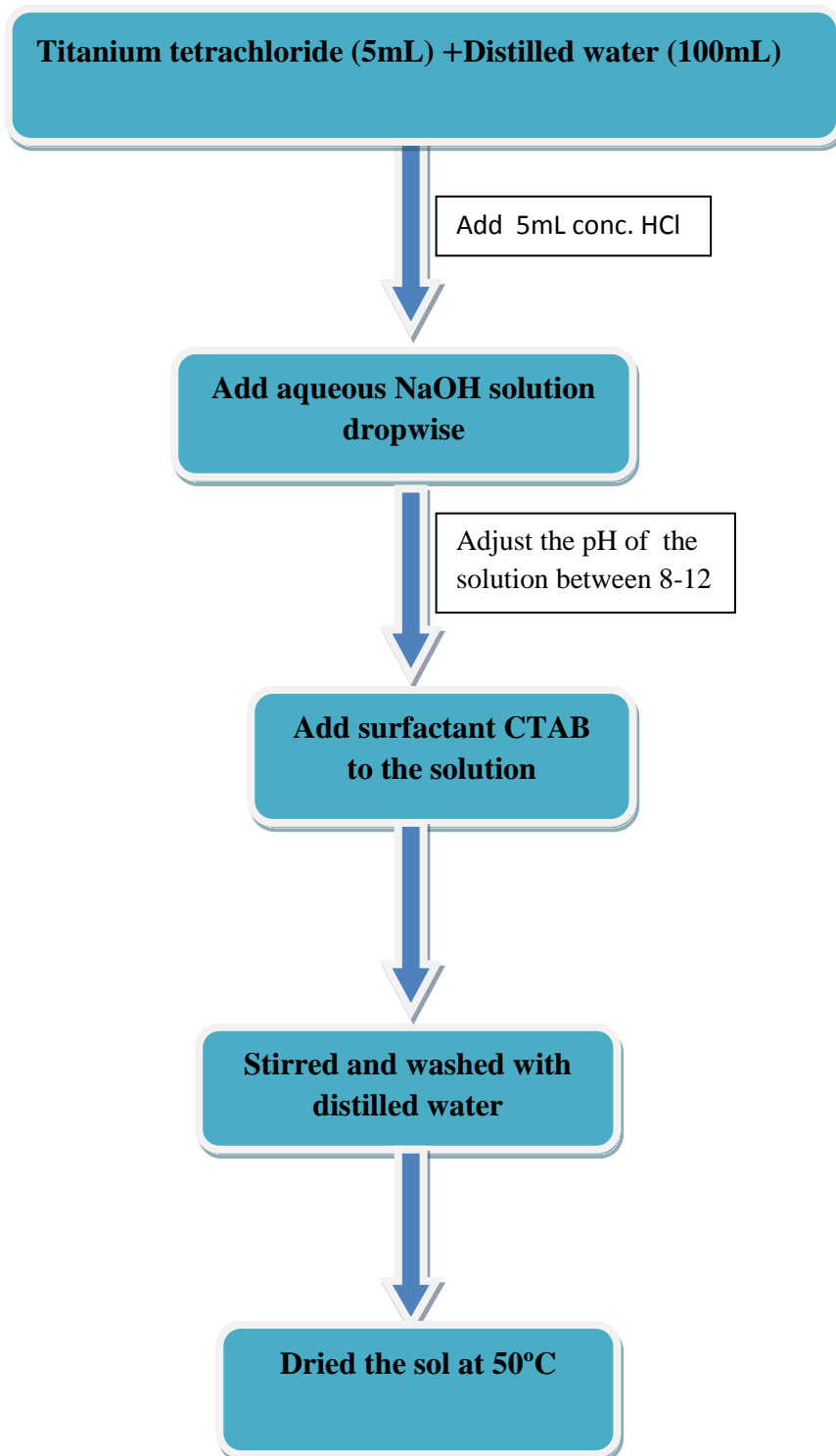
Cetyl trimethylammonium bromide (CTAB, purity>99.9) were purchased from S.D Fine Chemicals.

## **EXPERIMENTAL WORK**

**Preparation -:** Starting material for the synthesis was Titanium tetrachloride. In the very first step 100 ml of distilled water was taken and to it 5 ml conc.  $\text{HCl}$  was added. After which addition of 5 ml  $\text{TiCl}_4$  to the solution of water and acid is done. During the reaction, orthotitanic acid is formed i.e.  $\text{Ti}(\text{OH})_4$  due to exothermic reaction taking place. Purpose of addition of conc.  $\text{HCl}$  is to avoid the formation of orthotitanic acid. The aqueous  $\text{NaOH}$  solution was then added drop wise to adjust the pH of the solution in between 10-12. Now 0.01 M of surfactant CTAB was added and solution was stirred magnetically and washed with distilled water again and again. The precipitates were dried at  $50^\circ\text{C}$  until it is completely moisture free. After it another samples were also prepared different concentration of CTAB (0.002M, 0.005M).



Flowchart of the procedure that we have followed for the synthesis is shown :



## **PROCEDURE FOR ANTIMICROBIAL ACTIVITY:-**

The bactericidal activity of TiO<sub>2</sub> nanoparticles was observed by following method:-

- Neutral Agar(NA) was poured into the sterile petri plates and allowed to solidify.
- Two samples i.e. Escherichia Coli (E.Coli) and Staphylococcus aureus (S.aureus) were swabbed onto the plates.
- Wells were made into the plates with the help of borer.
- Different concentration of nanoparticles (0.01M, 0.002M and 0.005M) were loaded onto the wells.
- After 24 hours of incubation at 37°C, observed the zone formed and measured with the scale.

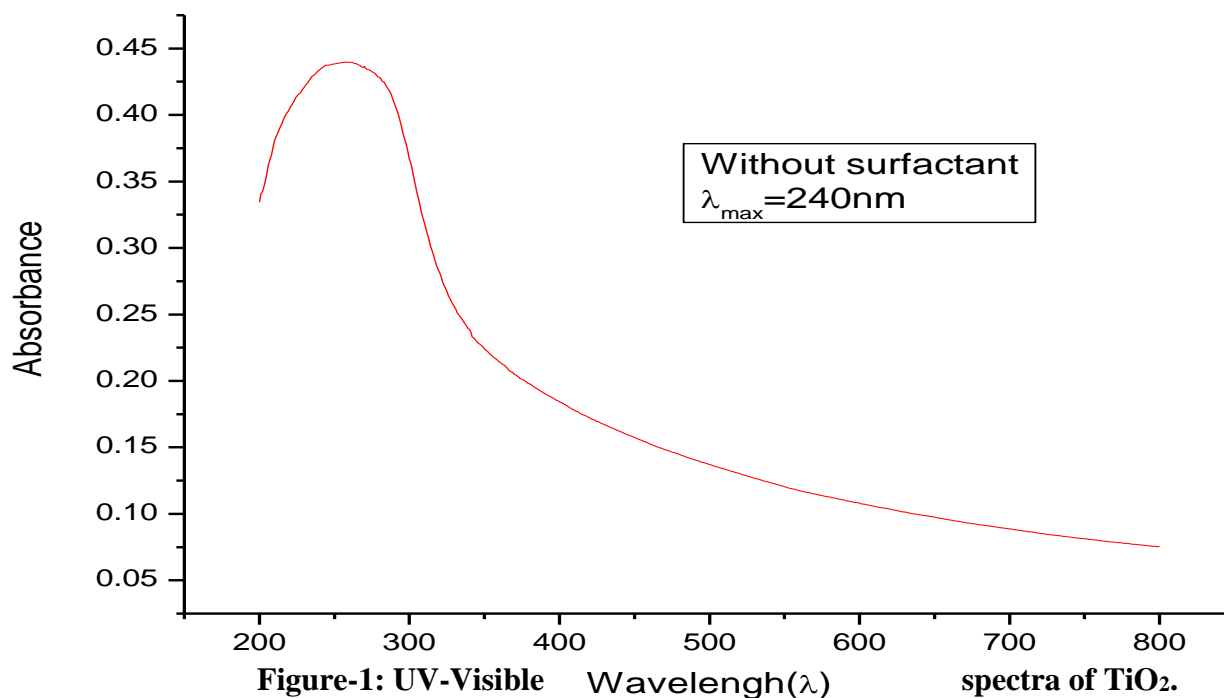
# Characterization

**Ultraviolet/visible absorption: Ultraviolet-visible spectroscopy or ultraviolet-visible spectrophotometry (UV-Vis or UV/Vis)** refers to absorption spectroscopy or reflectance spectroscopy in the ultraviolet-visible spectral region. Spectra was recorded in the range from 200nm to 800nm.

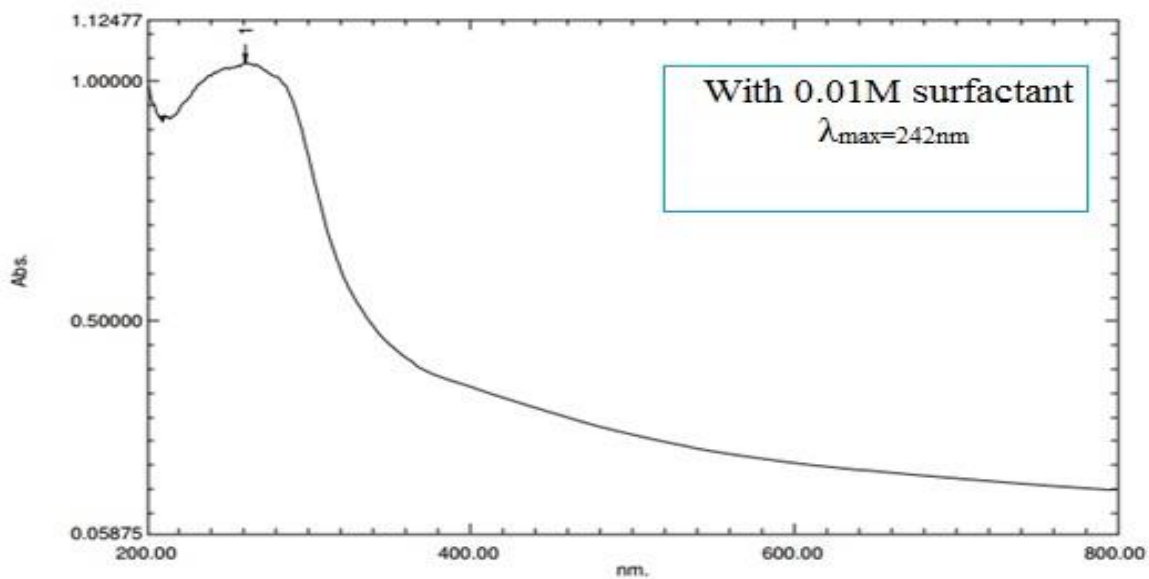
## **Instrumentation:**

### **Ultra violet spectroscopy**

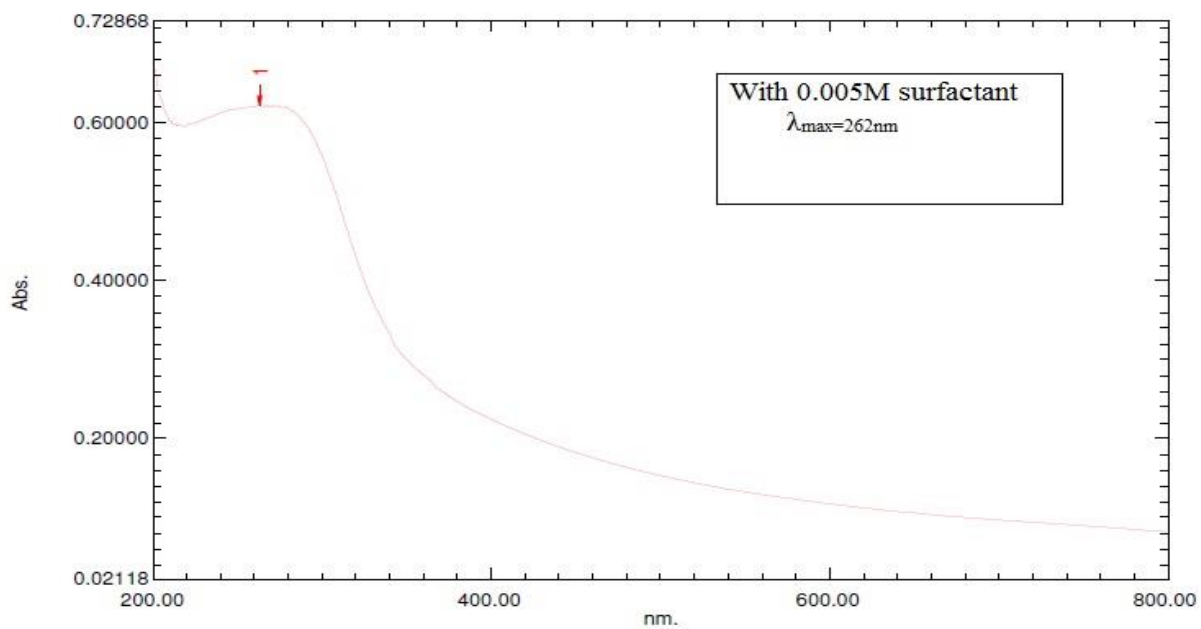
Ultra violet spectra were recorded from SHIMADZU UV-1800 spectrophotometer (Department of chemistry, Lovely professional university).



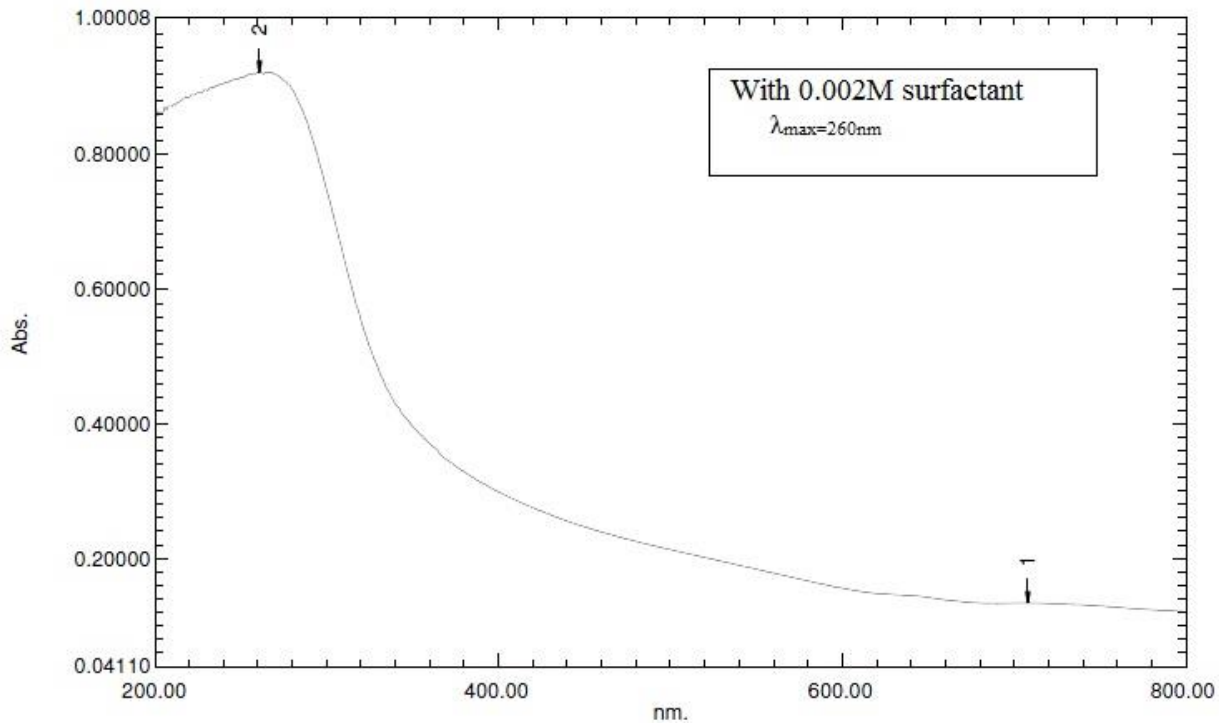




**Figure 2:- UV-Visible spectra of TiO<sub>2</sub> using 0.01M CTAB**

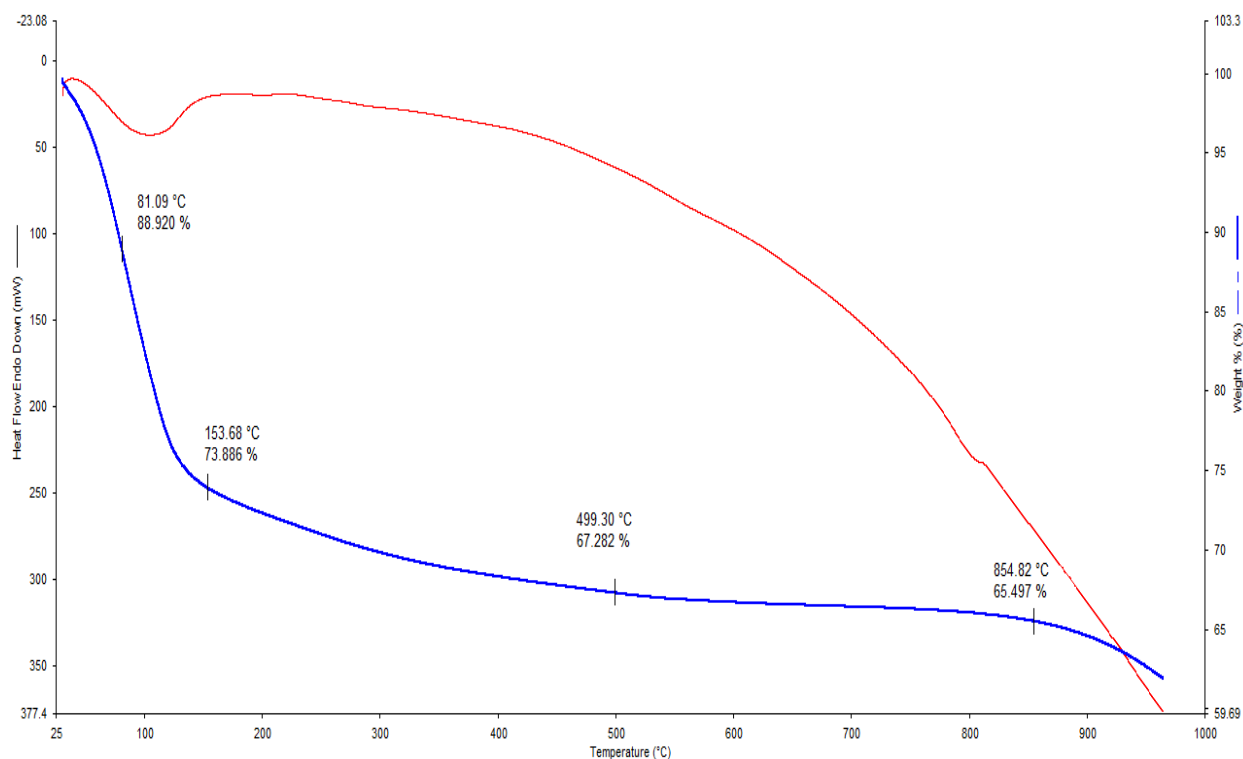


**Figure 3:- UV-Visible spectra of TiO<sub>2</sub> with CTAB having concentration 0.005M**



**Figure 4-: UV-Visible spectra of TiO<sub>2</sub> with CTAB having concentration 0.002M**

**Thermogravimetric analysis** or **thermal gravimetric analysis (TGA)** is a method of thermal analysis wherein changes in physical and chemical properties of materials are calculated as a function of increasing temperature (with constant heating rate), or as a function of time (with constant temperature and/or constant mass loss).



**Figure 5:- TGA of TiO<sub>2</sub> with CTAB conc. Of 0.01M**

In case of 0.01M CTAB the thermo gravimetric loss of 15.034% up to 153.68°C is because of evaporation of water present in the catalyst. The remaining loss of weight till 854.82°C is because of the loss of organic solvents and their residues that might be present in the sample.

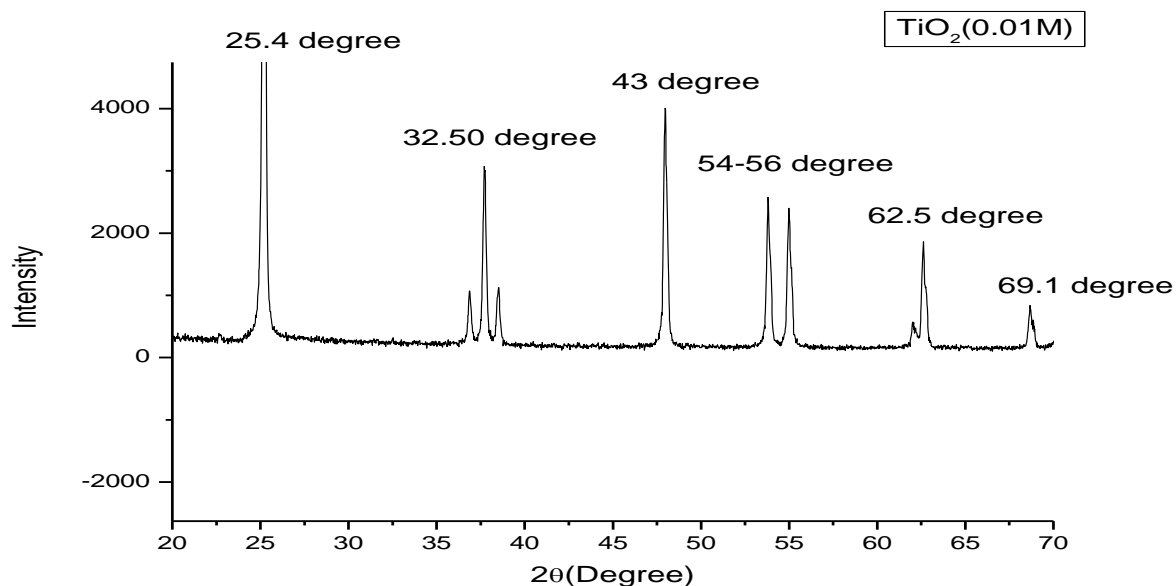
**XRD:** X-ray powder diffraction (XRD) is a rapid analytical technique mainly used for phase detection of a crystalline material and can give information on unit cell dimensions. The analyzed material is finely ground, homogenized, and average bulk composition is determined.

The crystalline structures of the products were analyzed by X-ray diffraction. In this Nickel-filtered Cu K $\alpha$  radiation having wavelength 0.15418nm was used with a generator voltage of 40 KV and a current of 29mA. Scherer's equation was used to analyzed the particles size of titanium dioxide

$$\tau = \frac{K\lambda}{\beta \cos \theta}$$

Where k is constant of 0.90,  $\theta$  is Bragg angle where  $\beta$  is half maximum line breath.

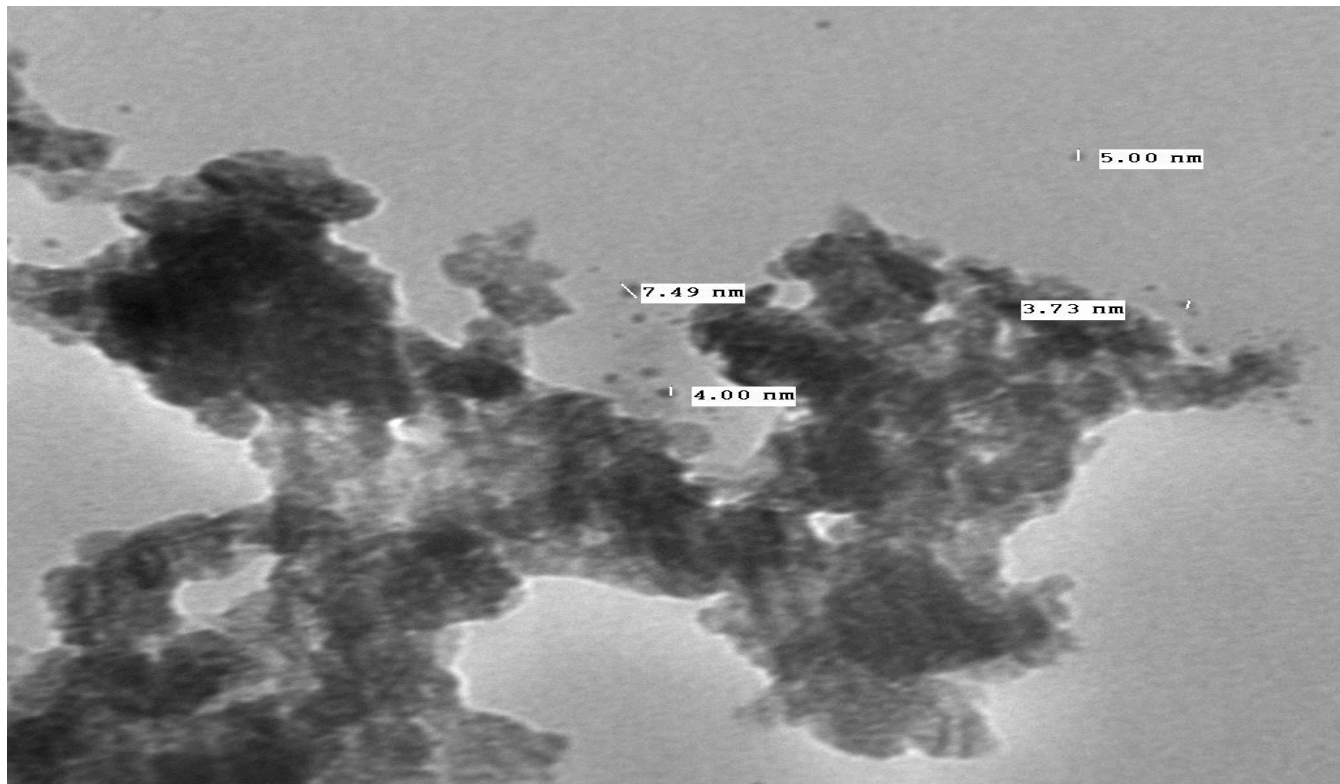
The peak at 25.2 degree was used to calculate the mean crystallite size Scherer's equation was used to calculate the mean diameter of the crystal particle. The mean diameter of crystallite for 25.4 degree is 7.8nm



**Figure 6-:** Shows the crystallinity and crystal phases of TiO<sub>2</sub> nano- particles by X-Ray diffraction several well defined peaks are observed in 25.4, 32.50, 43, 54 56 62.5, and 69.1 degree.

**Transmission Electron Microscope (TEM)**:- The transmission electron microscope is a very influential device for material science. A high energy beam of electrons is radiated through a very thin sample and the interactions between the electrons and the atoms can be used to examine features such as the crystal structure and features in the structure like dislocations and grain boundaries. High resolution can be used to investigate the quality, shape, size and density of quantum wells, wires and dots.

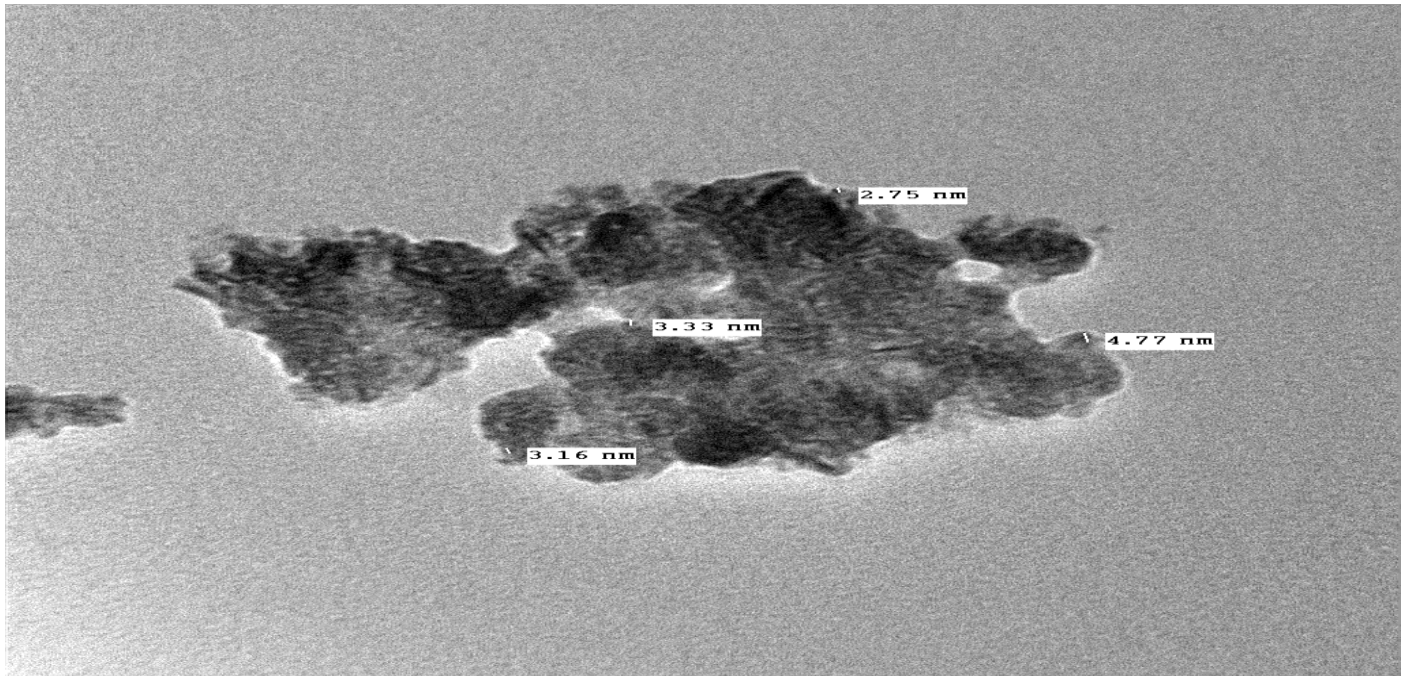
The characterization was based on TEM pictures since the material to study was a powder of nanosized particles.



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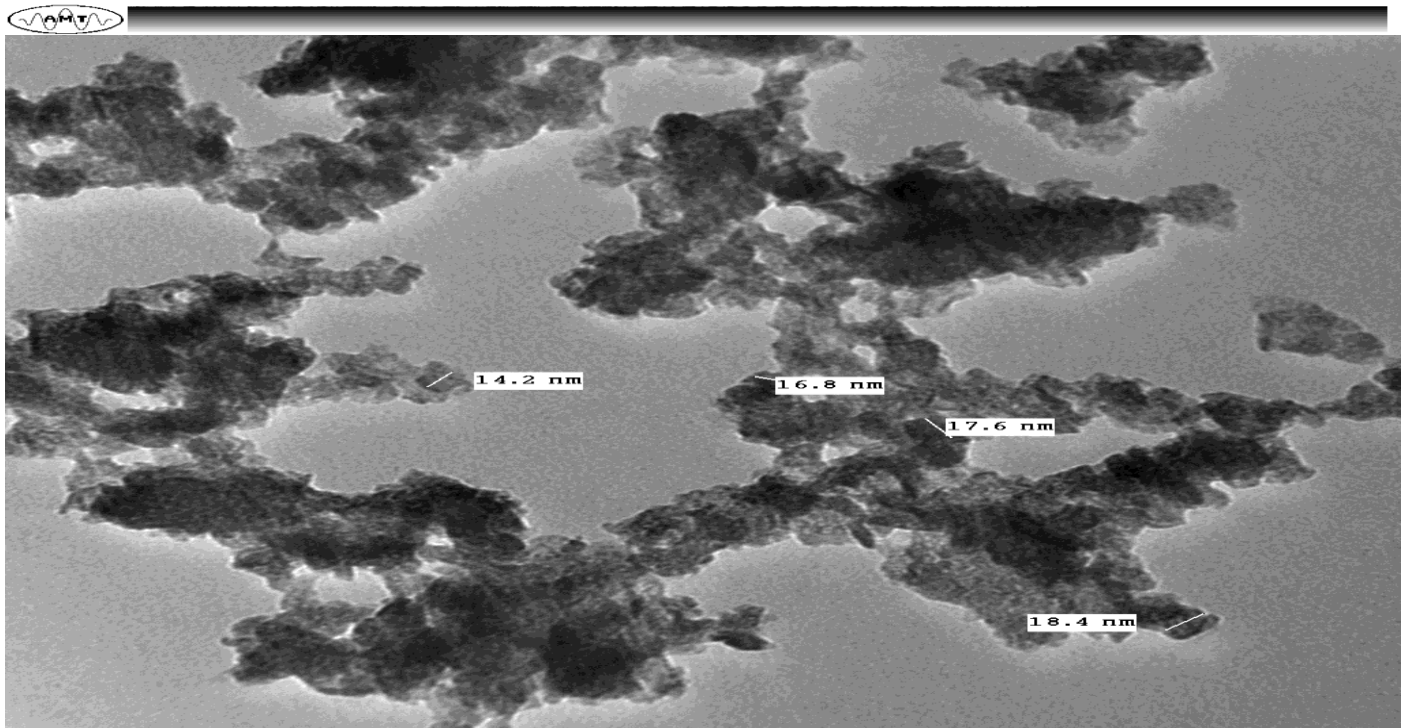
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AMT Camera System





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20 nm  
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AMT Camera System



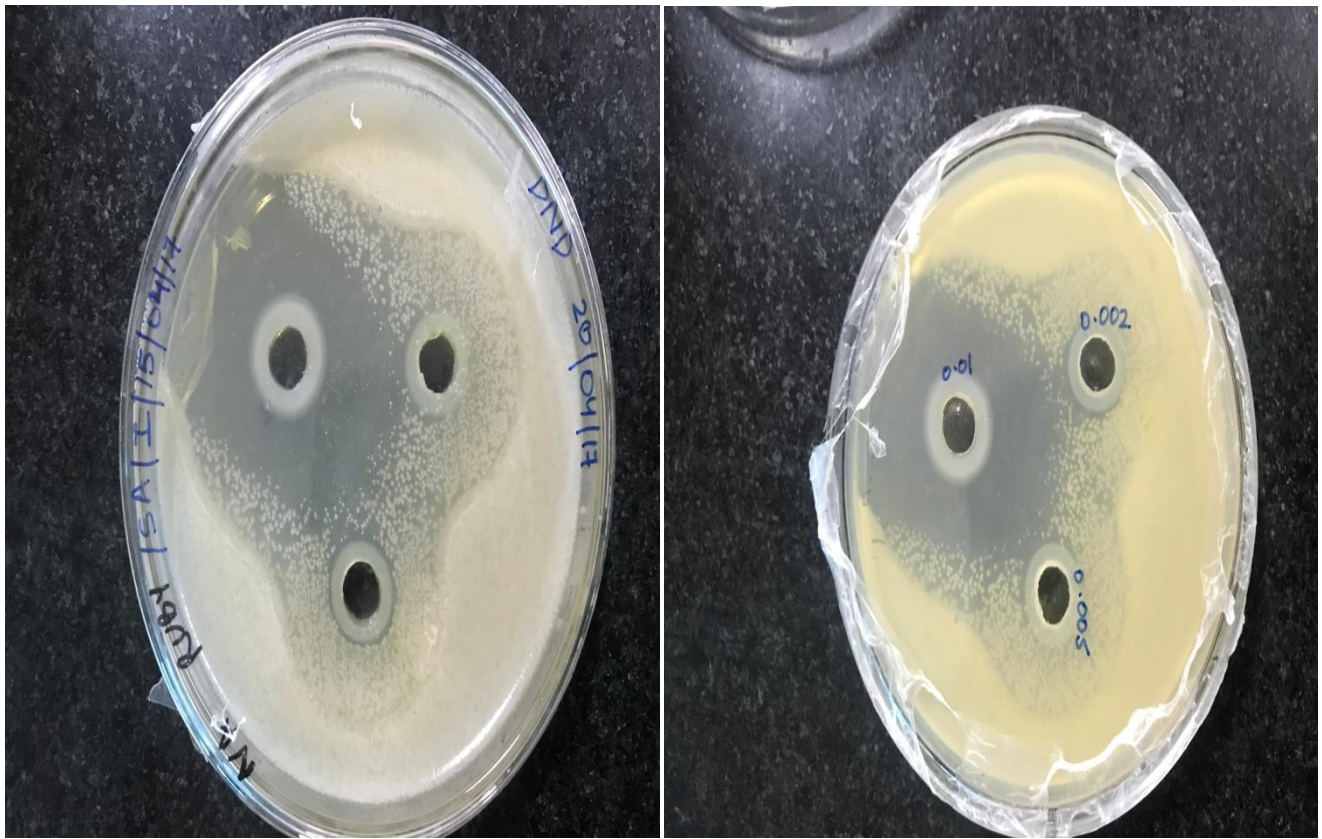
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10:48:58 a 04/26/17

100 nm  
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Direct Mag: 200000x  
AMT Camera System

**Bacterial Activity:-**

**Zone of Inhibition in case of S.aureus**

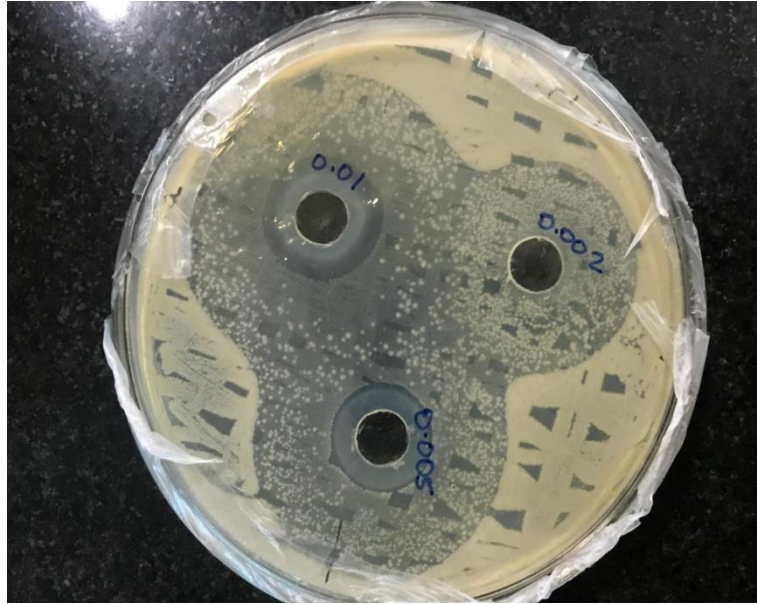
Molar Conc.	Diameter
0.01M	50mm
0.005M	35mm
0.002M	34mm



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Molar Conc.	Diameter
0.01M	46mm
0.005M	41mm
0.002M	32mm

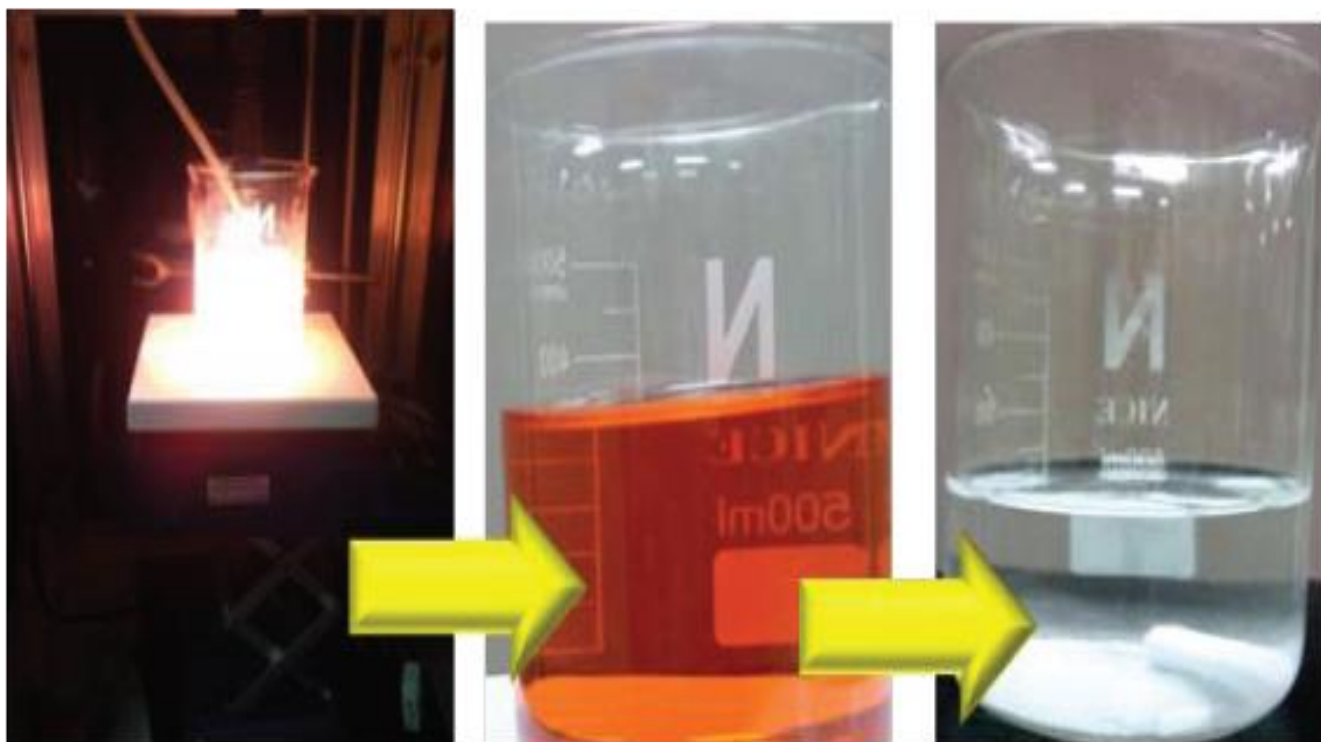
**Zone of Inhibition in case of E.Coli**



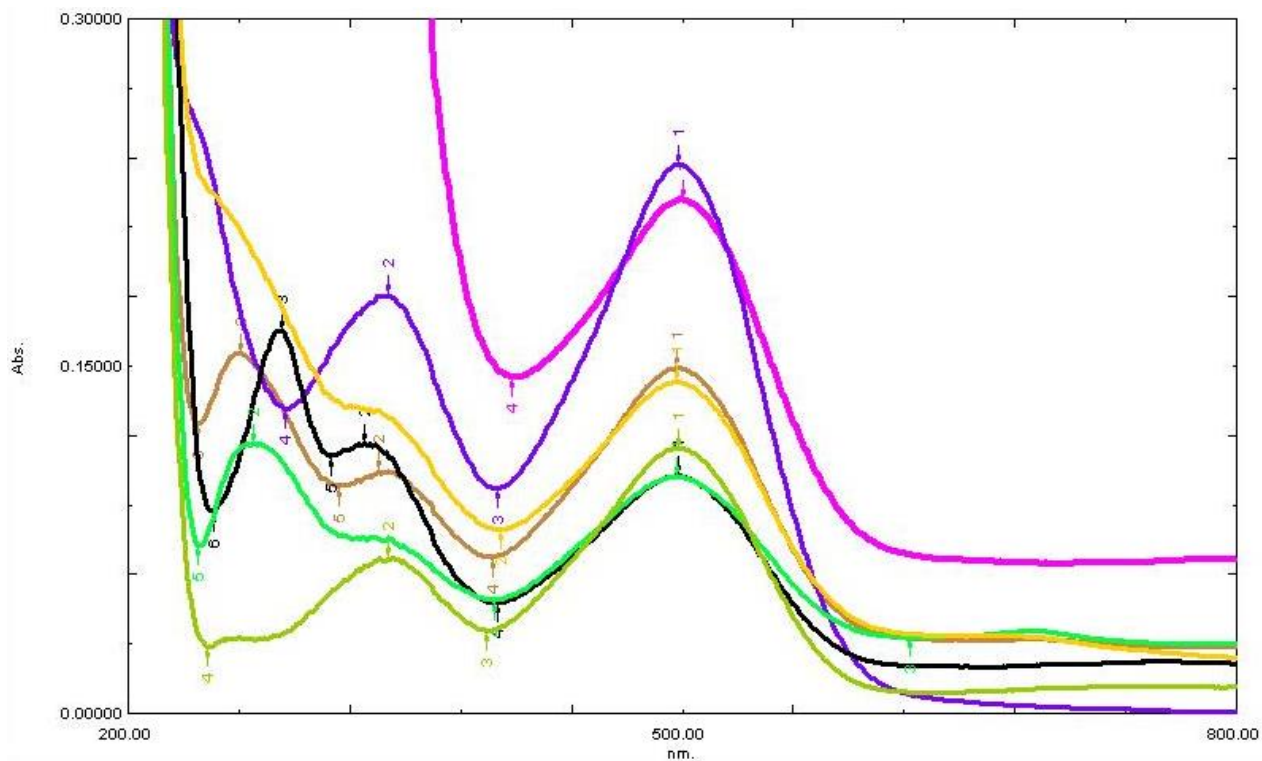


# PHOTOCATALYTIC EXPERIMENT

The photocatalytic activity of synthesized  $\text{TiO}_2$  catalyst was evaluated by degradation of an aqueous solution of Congo Red (0.001g) in 1000ml water. Take 100ml out of that solution and add 0.01g of  $\text{TiO}_2$  (0.01M) nanoparticles to it. The reaction mixture was kept in dark for 30 minutes so as to keep up adsorption – desorption equilibrium. The photocatalytic experiments were performed under Halogen Lamp with continuous stirring. At every 15 minutes interval, a UV Visible spectra was taken.



Gradually the bright color of Congo Red faded away with time when light was irradiated on it with continuous stirring.



**Figure 7:- UV-Vis spectrum of photodegradation of aqueous solution of Congo Red with TiO<sub>2</sub> catalyst having CTAB concentration 0.01 M.**

**The time interval interval between successive degradation is 10 minutes.**

**[Dye] =0.001g per 1000ml**

The progress of degradation of Congo red was monitored by the decrease in absorbance at 481 nm.

### **PERCENTAGE DEGRADATION OF CONGO RED**

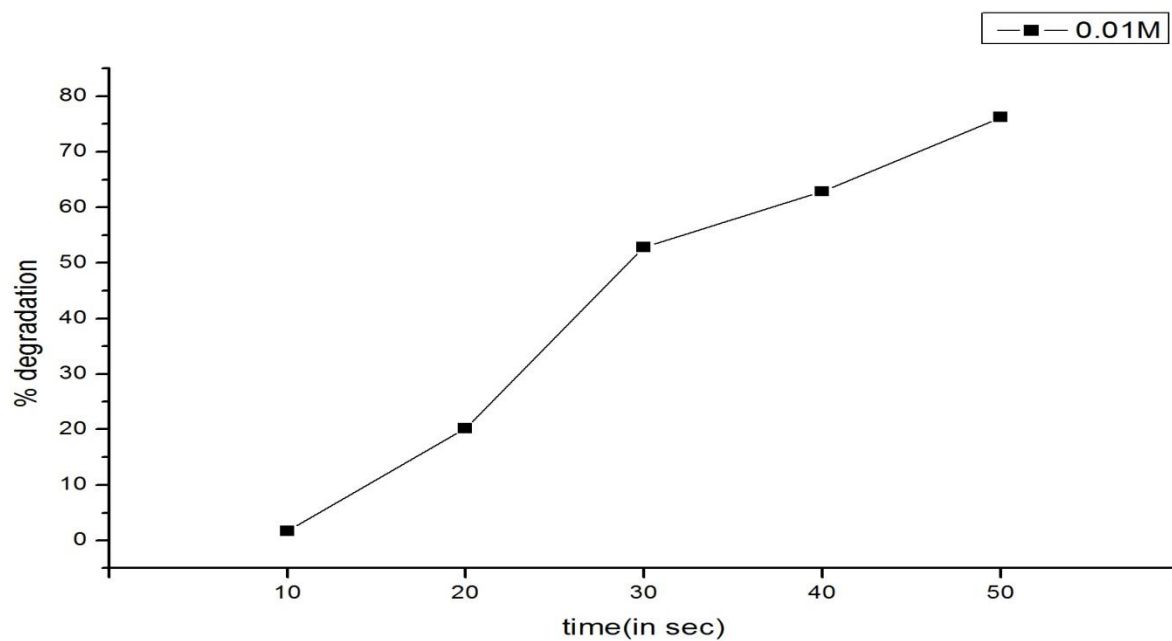
Formula used to calculate percentage degradation :-

$$\% \text{ age Degradation} = [ 1 - A_t/A_0 ]$$

A<sub>0</sub> is Absorbance of dye solution before degradation

$A_t$  is absorbance of dye solution after time “t”

<u>pH</u>	<u>CTAB concentration</u>	<u>Time(in sec)</u>	<u>%age Degradation</u>
10-11	0.01M	10	1.669%
10-11	0.01 M	20	20.18%
10-11	0.01 M	30	52.80%
10-11	0.01M	40	62.83%
10-11	0.01M	50	76.217%



**Figure Plot of Degradation Time Versus Percentage Degradation for Congo Red by TiO<sub>2</sub> having CTAB conc. 0.01 M**

# CONCLUSION

Anatase TiO<sub>2</sub> nanoparticles have been synthesized by SOL-GEL Technique. After Characterization results shows that these nanoparticles possessed good crystallinity, have a crystallite size of ~13nm and exhibit photocatalytic activity in the presence of halogen lamp. The TiO<sub>2</sub> nanoparticles synthesized by simple sol-gel method exhibits bacterial activity when irradiated with UV light against the common human pathogens tested. This opens up newer routes for the growth of solar assisted alternatives technologies for disinfection of water bodies contaminated with pathogenic organisms. TiO<sub>2</sub> is cost effective and very promising reusable photocatalyst.

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