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Synthesis and characterization of Pure Titanium dioxide nanoparticles by Sol- gel method

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Abstract : Titanium dioxide (TiO2) finds large area of applications ranging from CMOS to photo catalyst.Titanium dioxide is an important Bio and semiconductor material with a wide range of applications such as optical coating, dye sensitized solar cell, gas sensors and cosmetics etc has been synthesized by novel sol-gel technique[1,2].It is also high band-gap semiconductor, which is transparent to visible light and so excellent optical transmittance. The Sol-Gel technique is most attractive technique due to its many advantages such as easy preparation method, less complicating instruments and less time consuming. Here the Sol-Gel technique was successfully used for synthesising pure TiO2 nano-particles followed by characterization process. TiO2 nano-particles were synthesised using sol-gel technique followed by annealing. These synthesised nano-particles were characterized by various methods such as X-ray Diffraction (XRD), Fourier Transmission Infrared spectroscopy (FTIR), Ultra-Violet visible spectroscopy (UV) and Scanning Electron Microscope (SEM). The XRD can be used to show the presence of anatase TiO2 nano-particles. FTIR can be used to calculate the transmission range of TiO2 nano-particles. The UV spectroscopy can be used to lookout the shifting of absorption edges of TiO2 towards visible light region. The SEM range reveals the structure of nano-particles. The mechanism of synthesising TiO2 nano-particles using sol-gel technique is discussed in this paper.
Key Words: Titanium dioxide (TiO2), Sol-gel, XRD, Grain size, Band gap, SEM, UV, FTIR.

Introduction:

Titanium dioxide (TiO2) with an energy band of about 3.2 eV has various applications, such as optical coating, dye sensitized solar cell, and gas sensors. The performance of TiO2 based devices is largely influenced by the sizes of the TiO2 building units, apparently at the nanometer scale. As the most promising photocatalyst[4-6]. Reports of TiO2 with different shape such as nanoparticles thin films, nanorods, nanowires and nanotubes have spurred a great interest in studies on TiO2 nanostructure synthesis and their application[7]. Shape control has been significant concern in nanotechnology. Properties also vary as the shapes of the shrinking nano materials change. Many excellent reviews and reports on the preparation and properties of nano materials have been published recently[8-10].TiO2 has three polymorphic forms of crystal structure namely brookite, anatase and rutile.

The photo catalytic activity of TiO2 is dependent on its crystal structure, crystal size distribution, surface roughness, surface hydroxyl group density, etc[11].The photo catalytic activity of TiO2 can be significantly improved by doping with noble metals such as platinum (Pt), which acts as the photo generated electron acceptor. Thus, under UV illumination, the photo generated electrons effectively transfer from the TiO2 surface to the doped Pt particles to suppress the recombination of electrons and holes and promote the transfer of holes on the TiO2 surface. This produces a longer electron–hole pair separation lifetime, resulting in improved quantum yield and photo catalytic efficiency[11]. It constitutes the main supply of sulfur in the diet, preventing disorders in hair, skin or nails. More-over, it helps to reduce cholesterol levels by increasing the lectin production in liver, being also a natural chelating agent for heavy metals.

Titanium dioxide is a semiconductor material with a wide variety of applications; ranging from catalysis and dye sensitized solar cells to cosmetics. Among other factors, this variety of applications of titanium dioxide is possible because of properties like high stability, low cost and non-toxicity. Catalytic applications of titanium dioxide have been studied during decades for the elimination of environmental pollutants. Particularly, in the last years TiO2 has been studied for photo catalytic processes regarding degradation of pollutants in air, water and soil[12-14]. Currently, synthesis of TiO2 by sol-gel methods has proven to be a very useful tool for photo induced molecular reactions to take place on titanium dioxide surface. There are special variables that affect the photo induced reactions such as particle size, phase composition, incident light and preparation method, for instance, anatase TiO2 nanoparticles has shown higher photocatalytic activity than rutile TiO2[15-17]. There are several factors in determining important properties in the performance of TiO2 for applications such as particle size, crystalline and the morphology.

Experimental

Titanium dioxide (Tio2) nanoparticles have been prepared by sol-gel method. Analytical grade of purity titanium isopropoxide (TIP), isopropyl alcohol ((CH3)2CHOH), methanol (CH3OH) and acetic acid were used as starting materials. In a typical experiment 3.17ml titaniumisopropoxide was added drop wise to 9.50ml isopropyl alcohol. These solutions were continuously stirred for one hour. Then 10.3ml acetic acid was introduced to the mixture as a chelating agent and stirred. After adding the above, 24ml methanol was mixed and the solutionwas transformed to a moonstone colored solution. For preparation of the final catalyst samples, the corresponding precursors were heated for 2 h at 200 ºC using hot air oven.

Result and Discussion:

The obtained XRD spectrum was used to find the crystallite size of the prepared sample using Debye Scherrer’s formula. FTIR spectrum was used to calculate the various functional groups present in the nanoparticles. UV - visible spectroscopy was used to determine the band gap energy based on the numerical derivative of the optical absorption coefficient. The fundamental absorption method refers to band to band transitions by using energy relation. The SEM study was taken to determine the surface morphology of the sample.

X-Ray Diffraction Analysis:

The XRD (SHIMIDZU make, Model: XRD 6000) pattern of theSynthesized Titanium dioxide (Tio2) nanoparticles obtained from sol gel method attemperature 200℃ is shown in the Figure 1. Here three strongest peaks are taken for consideration. And based on that the grain size for those three peaks alone calculated, using the Debye- Scherer formula.The diffraction peaks existed at 2Θ =36.122, 35.948 and 34.662 reveals that all these peaks are indicative that the prepared sample is in hexagonal structure. The calculations of the smallest peaks 10, 23 and 31 of the XRD graph reveals the crystalline size, which the pattern match well with the standard JCPDS files # 21-1272[9].

Table 1: Strongest three peaks in XRD

No	2Theta(deg)	d(A)	FWHM
1	36.122	2.4846	0.76710
2	35.948	2.4962	0.80480
3	34.662	3.5858	0.87790

Table 1.1: Smallest three peaks in XRD

No	2Theta(deg)	d(A)	FWHM
1	25.122	3.5147	0.235
2	37.948	2.424	0.184
3	48.662	1.8742	0.40

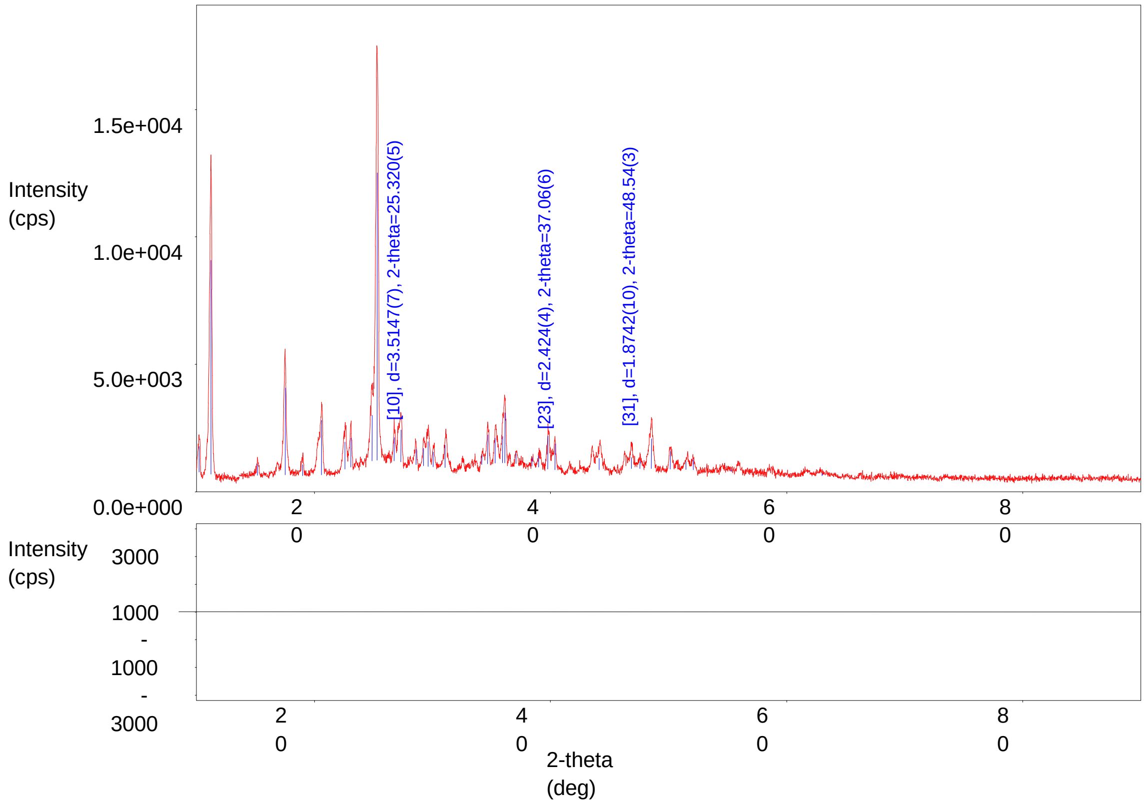

Figure 1: Graph of XRD

The crystallite size of Titanium dioxide nanoparticles evaluated using the Debye-Scherer formula.

$$D = \frac{K\lambda}{\beta COS\theta}$$

Where k is the constant (0.9), λ is the x wave length of X-ray (1.54 $\times 10^{-10}$), β is the full width half maximum (FWHM) of the peak and Θ is the reflection angle. The crystallite size of the Titanium dioxide nanoparticles is 5nm under optimized conditions.

Fourier Transform Infrared Spectroscopy (FTIR):

FTIR (Make: SHIMIDZU, Model: IR-AFFINITY-1) spectrum was used to calculate the various functional groups present in Titanium dioxide nanoparticles. Figure2 represents the FT-IR spectra of sol-gel derived TiO2 in the range of 400–4000 cm−1. The peaks at 3438.70 and 1640.26cm−1 in the spectra are due to the stretching and bending vibration of the -OH group. In the spectrum of pure TiO2, the peaks at 523.88 cm-1 show stretching vibration of Ti-O and peaks at 1416.38 cm-1shows stretching vibrations of Ti-O-Ti. Peaks at 3438.70 cm−1 indicates the presence of amines, Peaks at3287.11 cm−1 indicates the presence of Alkynes, Peaks at 3011.35 cm−1 indicates the presence of Aromatic rings ,Peaks at 1712.28 cm−1 indicates the presence of pyridines, Peaks at 1243.18cm−1 indicates the presence of Thiophenes.

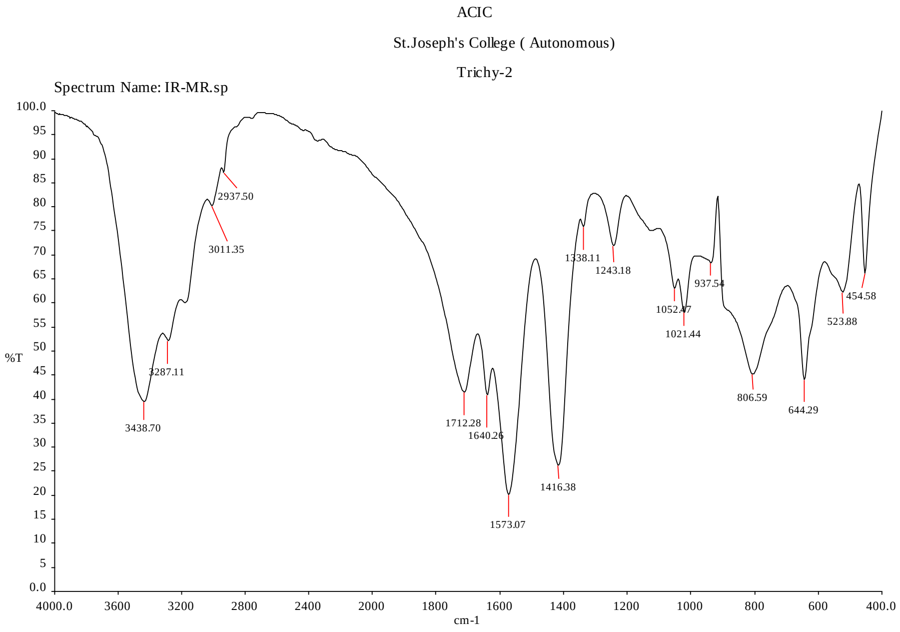

Figure 2:FTIR -Spectrum Peak Value

UV - Visible Spectroscopy

The band gap energy was determined based on the numerical derivative of the optical absorption coefficient. The fundamental absorption method refers to band to band transitions by using energy relation.

$$E = h\nu$$

Where h is the planks constantν=c/λ, where c is the speed of light in vacuum and λ is the wave length of the spectrum

Table2:Wavelength of Absorption & Transmittance.

No	Wavelength	%A	%T
1	218.76	1.7277	1.8714
2	203.04	1.0057	9.8649
3	200.01	0.90187	12.535

Band gap energy is calculated as 3.196 eV. The figure 3 and figure 4 shows the UV spectrum Peak value for absorption and transmittance respectively. The UV spectroscopy utilized was SHIMIDZU make and model: LAMDA-35.

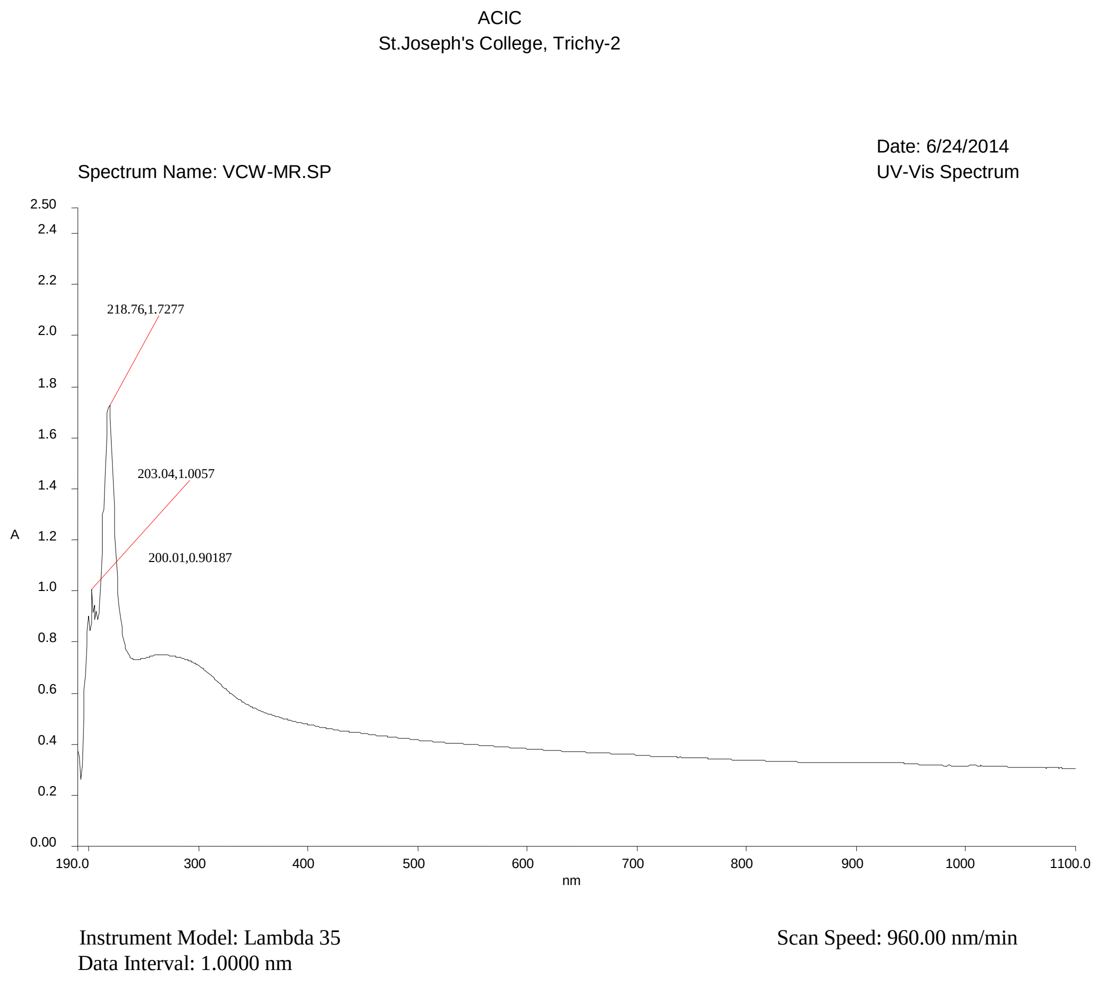

Figure 3: UV-Spectrum Peak Value (Absorption).

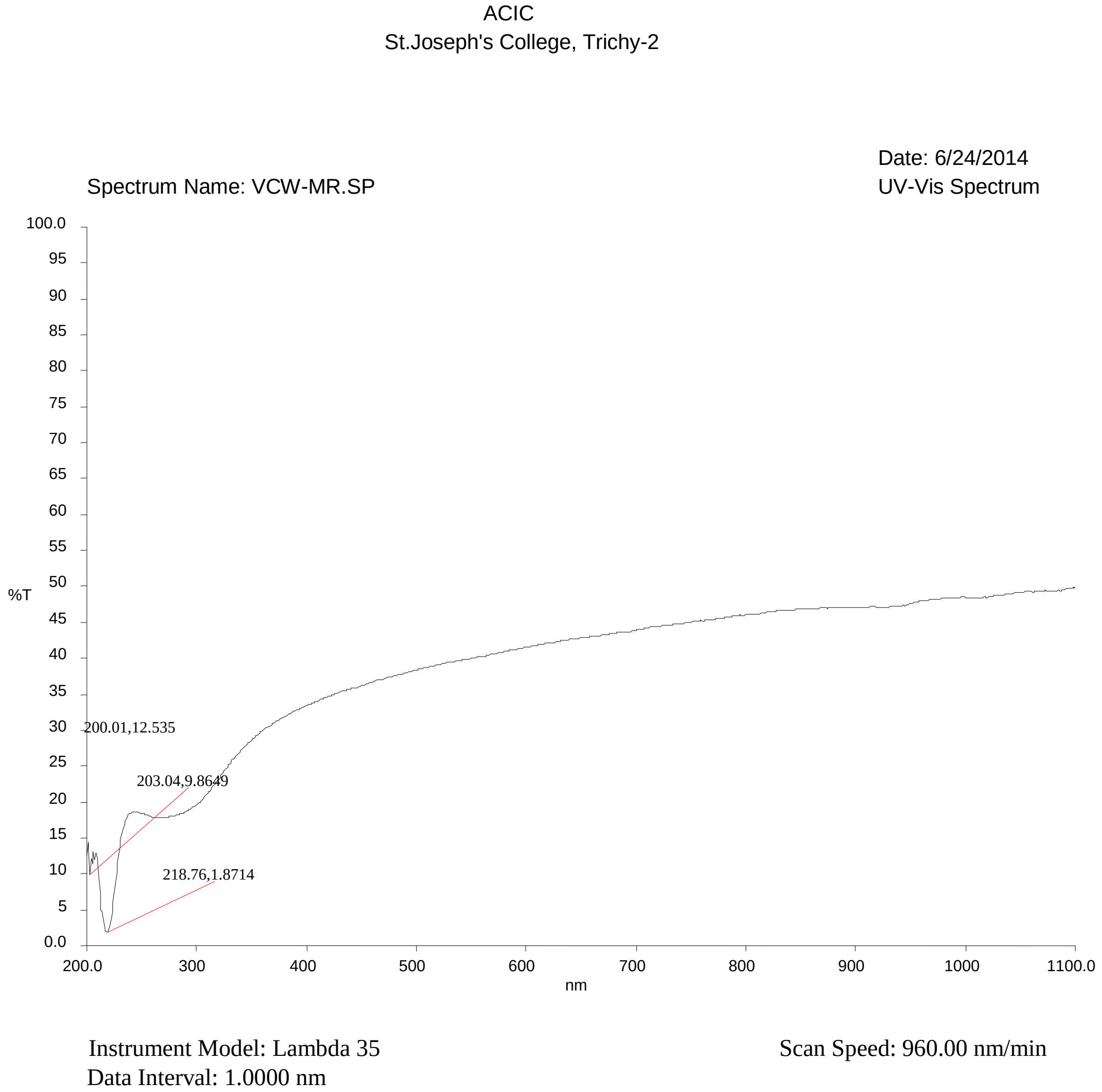

Figure 4: UV-Spectrum Peak Value (Transmittance)

Scanning Electron Microscope (SEM):

The surface morphology of the prepared sample was characterized by SEM (Make: TESCAN VEGA3, Model : JSM 6390). The figure5 to figure8 shows that the SEM image of Titanium dioxide (Tio2) nanoparticles. The nano particles in the SEM figures reveal hexagonal shapein the structure.

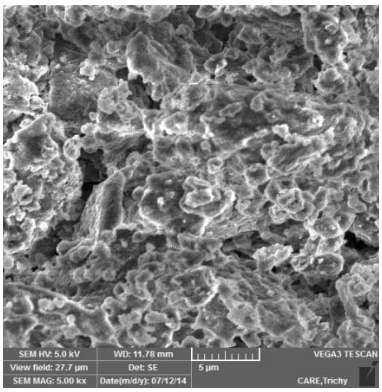

Figure 5: SEM image1

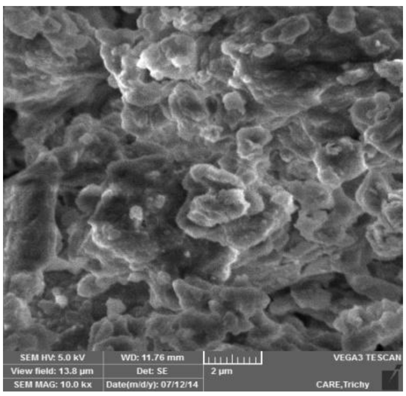

Figure 6: SEM image2

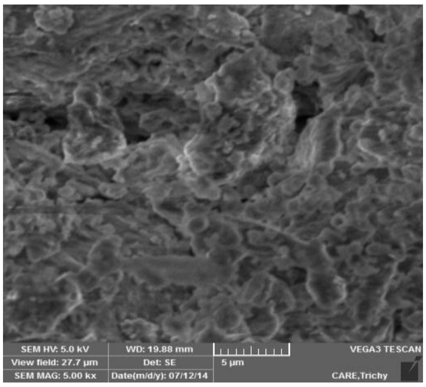

Figure 7: SEM image3

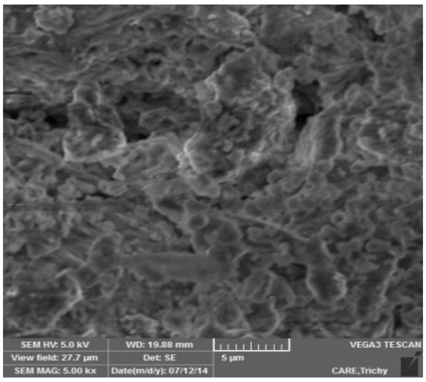

Figure 8: SEM image4

Conclusion

Titanium dioxide (Tio2) nanoparticles were synthesized through the sol gel method at 200°C. From X-ray diffraction analysis the crystallite size of the Titanium dioxide (Tio2) nanoparticles found to be 10 nm at 200°C. FTIR showed a various functional groups in Titanium dioxide (Tio2) nanoparticles and also determined by the transmission and absorptions range. The optical transmittance of the UV – VIS measurements indicates that the Titanium dioxide (Tio2) nano particles have a direct band gab energy is 3.196 eV. The SEM results showed that the formation of Titanium dioxide (Tio2) nanoparticles in cluster like structure.

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