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Preparation and Characterization of Titanium Dioxide Nano-particles by Novel Sol-Gel Method

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Abstract

Titanium dioxide (TiO_2) finds large area of applications ranging from CMOS to photo catalyst. Titanium dioxide is an important Bio and semiconductor material applied in advanced fields like biomedical engineering, DSSC, gas sensing, [8] cosmetics etc. such biomaterial is synthesized by novel sol-gel method [1], [2]. It has excellent optical transmission as because it is a high band-gap semiconductor, which is transparent to visible light. The Sol-Gel technique is the 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 TiO_2 nano-particles followed by characterization process. TiO_2 nano-particles were synthesised using sol-gel technique followed by annealing [10]. 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). XRD can be used to show the presence of anatase TiO_2 nano-particles. FTIR can be used to calculate the transmission range of TiO_2 nano-particles. The UV spectroscopy can be used to lookout the shifting of absorption edges of TiO_2 towards visible light region. The SEM range reveals the structure of nano-particles. The mechanism of synthesising TiO_2 nano-particles using sol-gel technique is discussed in this paper.

Keywords: Band gap, FTIR, Grain Size, SEM, Sol-Gel, Titanium Dioxide (TiO2), UV, XRD

1. Introduction

Titanium dioxide (TiO_2) is a biomaterial and semiconductor material with band gap energy of 3.2 eV has advanced applications, like biomedical engineering, DSSC, gas sensing and cosmetics etc [2]. The size of TiO_2 in nanometer scale largely influence the performance of TiO_2 based devices. TiO_2 nanoparticles exists in various shapes like Thin films, Nanorods, Nanowires and Nanotubes, hence created a wide interest in studies on TiO_2 nanostructure synthesis and their application [7]. Size and surface morphology are the main concern since Size control has been significant concern in nanotechnology. Properties vary with change in size of the nano materials [2]. We have many excellent reviews and reports on the preparation and properties of nano materials which have been published recently [8] – [10]. TiO_2 exists in three polymorphic forms of crystal structure namely brookite, anatase and rutile [2].

The photo catalytic activity of TiO_2 depends on its structure, size distribution, surface morphology, presence of hydroxyl groups, etc. It acts as photo generated electron acceptor when doped with latest metals like platinum (Pt), and improves is photo catality. It improves the transfer of holes on TiO_2 surface under UV illumination. This produces improvement in efficiency of Photo catalysis. In cosmetic field, it prevents main supply of sulfur in the diet, preventing disorders in hair, skin or nails, it helps to reduce cholesterol levels by increasing the lectin production in liver and acts as a natural chelating agent for heavy metals.

Titanium dioxide is a semiconductor material with advanced applications; ranging from catalysis, dye sensitized solar cells and cosmetics [3]. All this happens due to is properties like high stability, low cost and non-toxicity. Researchers show multiple interest in Catalytic applications of titanium dioxide, since it helps in the elimination of environmental pollutants. Mainly it is applied in deg-

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radation of pollutants in air, water and soil. Recently synthesis of TiO_2 by sol-gel methods has proven to be a very simple method for photo induced molecular reactions that carry out on titanium dioxide surface. Particle size, phase composition, incident light and preparation method affect the photo induced reactions. For example, anatase TiO_2 nanoparticles has higher photocatalytic activity than rutile TiO_2. There are several factors in determining important properties in the performance of TiO_2 for applications such as particle size, crystalline and the morphology.

2. Experiment

Here we undergo Synthesis of Titanium dioxide (TiO_2) nanoparticles by sol-gel method. Aldrich grade of Pure Titanium isopropoxide (TIP), isopropyl alcohol ((CH3)2CHOH), methanol (CH3OH) and acetic acid were used as starting materials. In a typical experiment 3.17ml titanium isopropoxide 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 solution was transformed to a moonstone colored solution. For preparation of the final catalyst samples, the corresponding precursors were heated for 2 h at 200 °C using hot air oven.

3. Results and Discussion

The obtained XRD spectrum was used to calculate the crystallite size of the TiO_2 nanoparticles using Debye Scherrer's formula. FTIR spectrum is used to calculate the various functional groups present in the nanoparticles. UV-visible spectroscopy was used to determine the band gap energy. The SEM analysis reveals the surface morphology of TiO_2 nanoparticles.

3.1 X-Ray Diffraction Analysis

The XRD pattern of the Synthesized Titanium dioxide (TiO_2) nanoparticles obtained from sol gel method at temperature 200 °C is shown in the Figure 1. The diffraction peaks existed at 2Θ =36.122, 35.948 and 34.662 in Table 1 reveals that all these peaks are indicative that the prepared sample is in hexagonal structure.

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

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

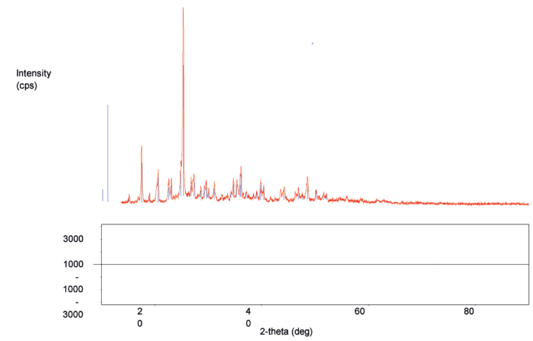

Figure 1. Graph of XRD.

Table 1. Strongest three peaks in XRD

No	2θ (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

where k is the constant (0.9), λ is the x wave length of X-ray (1.54×10^{-10}), β is the Full Width Half Maximum (FWHM) of the peak and θ is the reflection angle. The crystallite size of Titanium dioxide nanoparticles is 10nm under optimized conditions. The pattern match well with the standard JCPDS files # 21-1272 [9].

3.2 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectrum is used to calculate the various functional groups present in Titanium dioxide nanoparticles. Figure 2 represents the FT-IR spectra of sol-gel derived TiO_2 in the range of 400–4000 cm^{-1}. The peaks at 3438.70 and 1640.26 cm^{-1} [5] in the spectra are due to the stretching and bending vibration of the -OH group. In the spectrum of pure TiO_2, the peaks at 523.88 cm^{-1} show stretching vibration of Ti-O and peaks at 1416.38 cm^{-1} shows stretching vibrations of Ti-O-Ti. Peaks at 3438.70 cm^{-1} [6] indicates the presence of amines, Peaks at 3287.11 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.

3.3 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 [4].

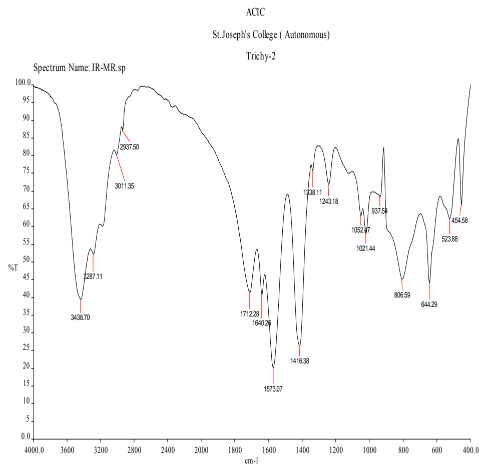

Figure 2. FTIR-Spectrum Peak Value.

$$E = h\nu$$

Where h is the planks constant $\nu = c/\lambda$, where c represents the speed of light in vacuum and λ, the wave length of the spectrum.

The graph is (αhγ) Vs (hγ) is shown in Figure 3 & 4.

Band gap energy is calculated as 3.196 eV.

Table 2. Wavelength of Absorption & Transmittance

No	2θ (deg)	d(A)	FWHM
1	218.76	1.7277	1.8714
2	203.04	1.0057	9.8649
3	200.01	0.90187	12.535

3.4 Scanning Electron Microscope (SEM)

The surface morphology of the prepared sample was characterized by SEM. Figure 5 to Figure 8 shows that the SEM image

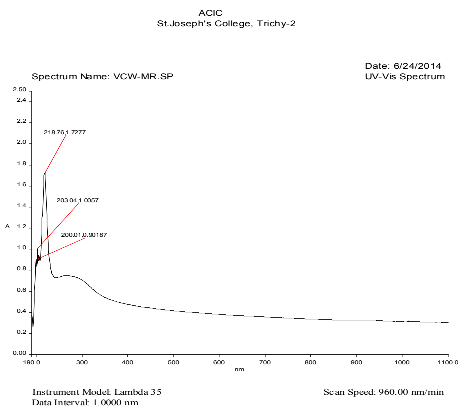

Figure 3. UV-Spectrum Peak Value (Absorption).

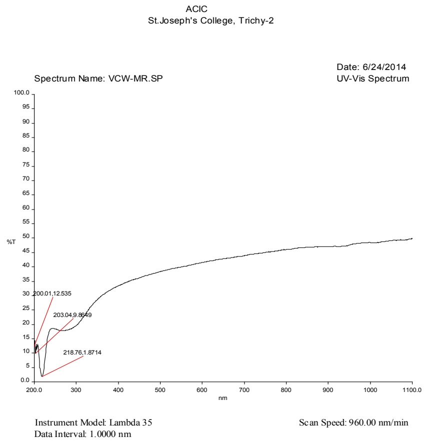

Figure 4. UV-Spectrum Peak Value (Transmittance).

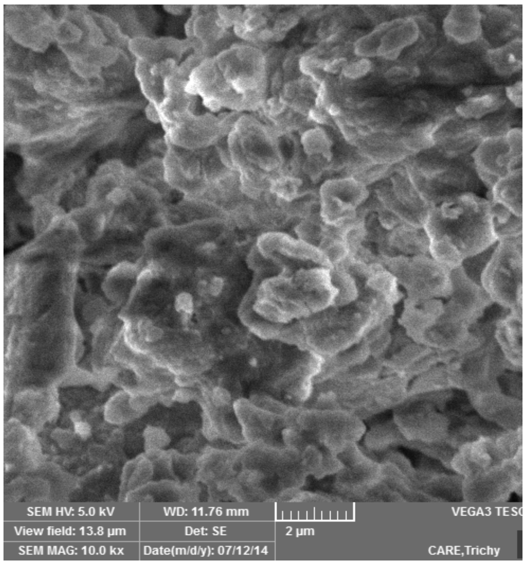

Figure 5. SEM image 1.

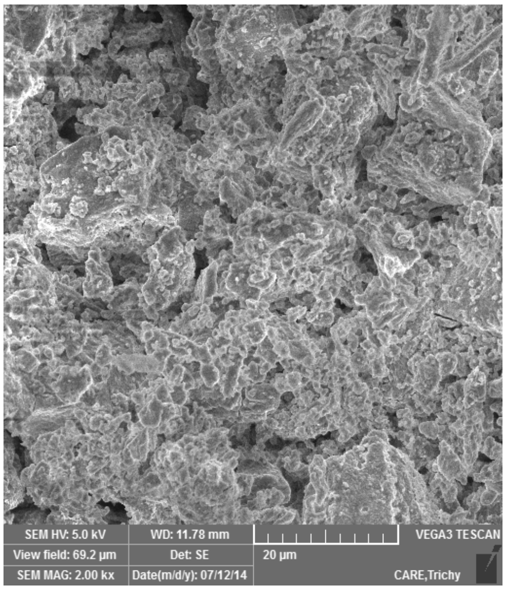

Figure 6. SEM image 2.

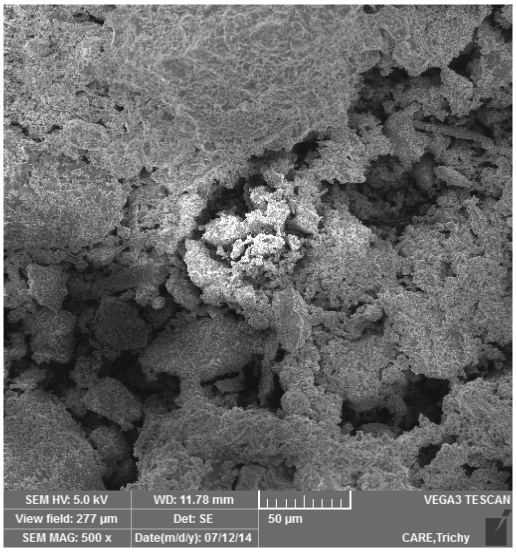

Figure 7. SEM image 3.

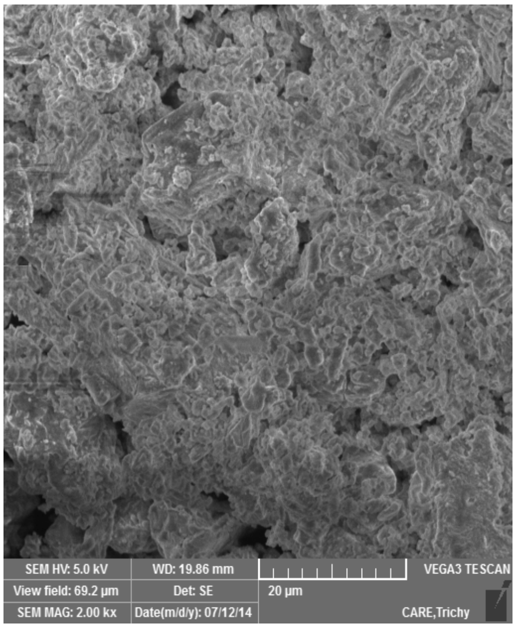

Figure 8. SEM image 4.

of Titanium dioxide (TiO_2) nanoparticles in the agglomerated structure due to vander waals force of attraction.

4. Conclusion

Titanium dioxide (TiO_2) nanoparticles were synthesized via the sol gel method at 200 °C. From X-ray diffraction analysis the crystallite size of the Titanium dioxide (TiO_2) nanoparticles found to be 10 nm at 200 °C. FTIR showed a various functional groups in Titanium dioxide (TiO_2) nanoparticles and also determined by the transmission and absorptions range. The optical transmittance of the UV – VIS measurements indicates that the Titanium dioxide (TiO_2) nano particles have a direct band gab energy is 3.196 eV. The SEM results reveals the formation of Titanium dioxide (TiO_2) nanoparticles in agglomerated structure.

5. References

1. Li W., Kang J., Li X., Fang S., Lin Y., Wang G., Xiao X., "A novel polymer quaternary ammonium iodide and application in quasi-solid-state dye-sensitized solar cells", *J. Photochem. Photobiol. Chem.*, vol. 170, p. 1–6, 2005.
2. Patil U.M., Gurav K.V., Joo O-S., Lokhande C.D., "Synthesis of photosensitive nanograined TiO_2 thin films by SILAR method", *J. Alloy. Comp*., vol. 478. p. 711–715, 2009.
3. Mane R.S., Hwang Y., Lokhande C.D., Sartale S.D., Han S-H., "Room temperature synthesis of compact TiO_2 thin films for 3-D solar cells by chemical arrested route", *Applied Surface Science*, vol. 246(1), p. 271–278, 2005.
4. Mosaddeq-ur-Rahman Md., Krishna K.M., Miki T., Soga T., Igarashi K., Tanemura S., Umeno M., "Investigation of solid state Pb doped TiO_2 solar cell", *Sol. Energ. Mater. Sol. Cell.*, vol. 48. p. 123–130, 1997.
5. O'Regan B., Gratzel M., "A low-cost, high-efficiency solar cell based on dye-sensitized colloidal TiO_2 films", *Nature*, vol. 353. p. 737–740, 1991.
6. Roberson L.B., Poggi M.A., Kowalik J., Smested G.P., Bottomley L. A., Tolbert L. M., "Correlation of morphology and device performance in inorganic–organic TiO_2-polythiophene hybrid solid-state solar cells", *Coordination Chemistry Reviews*, vol. 248(13), p. 1491–1499, 2004.
7. Liu Z., Pan K., Liu M., Zhang Q., Li J., Liu Y., Lü Q., Li J., Wang D., Bai Y., Li T., "Influence of the binder on the electron transport in the dye-sensitized TiO_2 electrode". *Thin Solid Films*, vol. 484. p. 346–351, 2005.
8. More A.M., Kunjkar J.I., Lokhande C.D., "Liquefied petroleum gas (LPG) sensor properties of interconnected web-like structured sprayed TiO_2 films", *Sensor Actuator B. Chem.*, vol. 129. p. 671–677, 2008.
9. Cao Y., Yang W., Chen Y., Du H., Yue P., "Effect of chemisorbed surface species on the photocatalytic activity of TiO_2 nanoparticulate films", *Appl. Surf. Sci.,* vol. 236. p. 223–230, 2004.
10. Gokilamani N., Muthukumarasamy N., Thambidurai M., "Synthesis and characterization of nanocrystalline TiO_2 thin films by solgel dip coating method", A*dvanced Materials Research*, vol. 678. p. 108–112. 2013 Apr.