ANNALS OF FACULTY ENGINEERING HUNEDOARA
– International Journal of Engineering
Tome XI (Year 2013) – FASCICULE 3 (ISSN 1584 – 2673)

[1.] M.U. SHAHAB, [2.] T.A.TABISH, [3.] B. ZAMAN, [4.] ZAHRA TARIQ, [5.] M. KAMRAN

CHARACTERIZATION AND SYNTHESIS OF NANOSIZED TiO_2 PARTICLES

[1,3-5.] CEET, UNIVERSITY OF THE PUNJAB, LAHORE 6400, PAKISTAN
[2.] INSTITUTE OF ADVANCED MATERIALS, BAHAUDDIN ZAKARIYA UNIVERSITY, MULTAN 54800, PAKISTAN

ABSTRACT: *Semiconductor and Inorganic metal oxide TiO_2 of Nano-sized has extraordinary enhanced morphology, grain size, high transparency, biological stability, chemical and physical properties.TiO_2 Nanoparticles are especially suitable material for optical coatings and protective layers for very large scale integrated circuits due to excellent transmittance for visible range and high refractive index.TiO_2 Nanoparticles were prepared by wet chemical method which is known as Sol-Gel method that is achieved by drop wise addition of titanium tetrachloride in Ethyl alcohol followed by calcination at 500°C.After this calcination, Anatase phase TiO_2 Nanoparticles were achieved in the Nanoscale particle size range.TiO_2 Nanoparticles are characterized by FTIR, UV-vis and SEM techniques which give the evidence of formation TiO_2 Nanoparticles.*
KEYWORDS: *Nanoparticles, TiO_2, sol-gel method, Anatase*

INTRODUCTION

Semiconductor and Inorganic metal oxide TiO_2 of Nano-sized has extraordinary enhanced morphology, grain size, high transparency, biological stability, chemical and physical properties.TiO_2 Nanoparticles are especially suitable material for optical coatings and protective layers for very large scale integrated circuits [1] due to excellent transmittance for visible range and high refractive index. The nano-crystalline TiO_2 particles achieve increased redox potential and high surface area due to its polymorphic forms. Nanoparticles of TiO_2 have occurred in main three forms: Anatase, Rutile and Brookite. Rutile phase is highly stable phase at high temperature whereas Anatase has a wider optical band gap (3.2 eV), a smaller electron effective mass, a higher Fermi level and high mobility of charge carriers [2].These phases make nano-phased TiO_2 as promising material and attracted attention for its significant applications.TiO_2 Nanoparticles are widely used as photo catalyst material for degradation of organic contaminants[3] , sensors and photo-electric chemical conversions in solar cells[4], surface coatings and polymer industry as a pigment and a filler[5].

Furthermore the phase and degree of crystallinity of TiO_2 Nanoparticles play general role in various applications due to crystallinity and phase formation of TiO_2 Nanoparticles. As TiO_2 Nanoparticles has much importance in industrial field, on the other hand TiO_2 Nanoparticles has various synthesizing methods such as Flame aerosol synthesis[6], Hydrothermal synthesis[6-7] and hydrolysis of inorganic salt[8]. Sol-Gel synthesis [9-11] is one of the easiest methods to fabricate Nano-sized TiO_2 Nanoparticles because of its lower calcination conditions and proper allowable cost. Chan Baek et al. [12] reported in 2009 that Sol-Gel method is convenient and superior method among all other processes because it consists of hydrolysis, water condensation and Alcohol condensation which form high area of Nanoparticles. Sang Yoo et al. [13] in 2005 reported the Sol-Gel synthesis as wet chemical method in which alkoxide oxide is used as a precursor which starts chemical reaction at relatively lower temperature.

In Present Research the synthesis of TiO_2 Nanoparticles has been reported by Sol-Gel Method with ethyl alcohol and Titanium Tetrachloride $TiCl_4$ as precursor respectively. It is then characterized by Fourier Transform infrared spectroscopy (FTIR), Uv-Vis spectroscopy (UV-Vis) and Scanning Electron Microscopy (SEM).This characterization investigates the evidence of TiO_2 Nano-sized particles (≥45 nm) in this particle size range.

EXPERIMENTAL WORK

Titanium Tetrachloride ($TiCl_4$, 98%, Fluka) was taken as a precursor which is added drop wise by micropipette in ethyl alcohol (C_2H_5OH). The solution is operated at room temperature in a beaker which is placed on magnetic stirrer. The Agitator in a magnetic stirrer is made of Teflon which is used for mixing the solution.

This process is conducted under fume hood in order to evolve the gases HCl and Cl_2 generated during synthesizing. In starting the colour of Ethyl alcohol is White but after sometime the colour of the solution is changed in yellowish by titanium tetrachloride drop wise addition. The viscosity of the solution is changed and it transformed into Gel. This Gel contains the colloidal suspension of Titanium particles which stays here due to Brownian motion. This yellowish suspension is formed after 2 hours and then magnetic stirrer is stopped. After this suspension is given time to cool at room temperature for about 24 hour. This cooling evolved the moisture to some extent from the suspension.

After all this dried suspension is heated in oven at 120^0C for about 3 hours. After drying, the suspension is changed into amorphous powder of Yellowish colour. For further phase Transformation it is calcined at 500^0C for about 2 hours. This calcination gives phase transformation and also the yellowish colour of particles changed into White powder. The characterization of TiO_2 Nanoparticles gives the evidence of these particles.TiO_2 Nanoparticles are characterized by Fourier Transform infrared spectroscopy in which (SHIMADZU, IR SPECTAGEL-21), was used to study stretching and bending of bond which absorbs selected frequencies or energies of infrared radiation. UV-Vis spectroscopy was done with (SPECTRO- UV) to study the absorption bands corresponding to various structural groups by exposing UV having spectral bandwidth of 2.00 nm. Scanning Electron Microscopy (SEM) of prepared TiO_2 Nanoparticles was done which operated at 15 KV (12.0 mm x 30.0 K, SE) to visually characterize TiO_2 Nanoparticles.

RESULTS AND DISCUSSION

Figure 1 shows the FTIR spectra of as prepared TiO_2 sample. The graph is plotted between % transmittance and wave number (cm^{-1}). In this graph different peaks formed at different wave number. It is observed in the graph that TiO_2 Nanoparticles have various frequency vibrations which are shown by different peaks formed. The peak in range 1600-1650 cm^{-1} were characteristic of O—Ti—O bond and narrow adsorption bond is observed due to Ti==O bending region. The broad adsorption band is observed at 3000-3600 cm^{-1} is corresponding to O—H stretch region.

Figure 2 shows the UV spectra of TiO_2 Nanoparticles. In this spectroscopy graph is plotted between Absorbance and wave number. UV spectroscopy observed the peak of TiO_2 Nanoparticles formed below the 325 nm. It is observed that peak of Anatase TiO_2 Nanoparticles absorbed most of the UV light with a lower wavelength (<325 nm).This shows the formation of Anatase TiO_2 Nanoparticles.

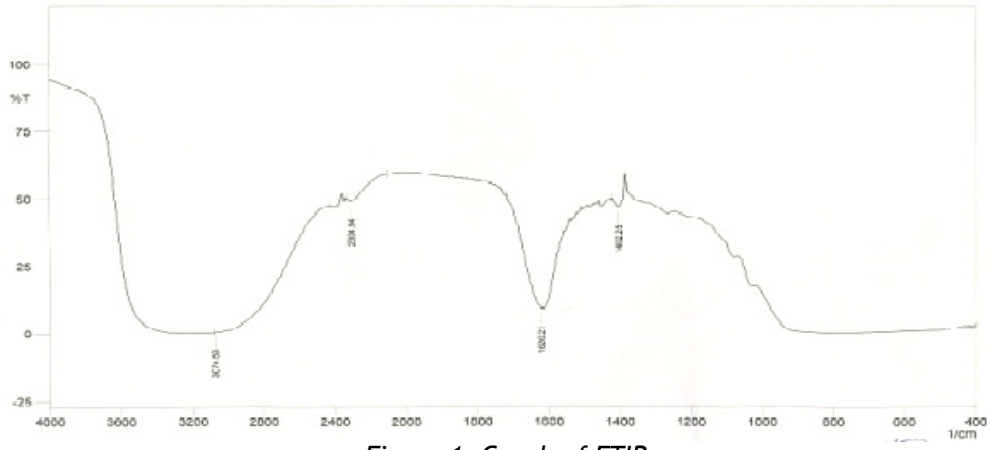

Figure 1. Graph of FTIR

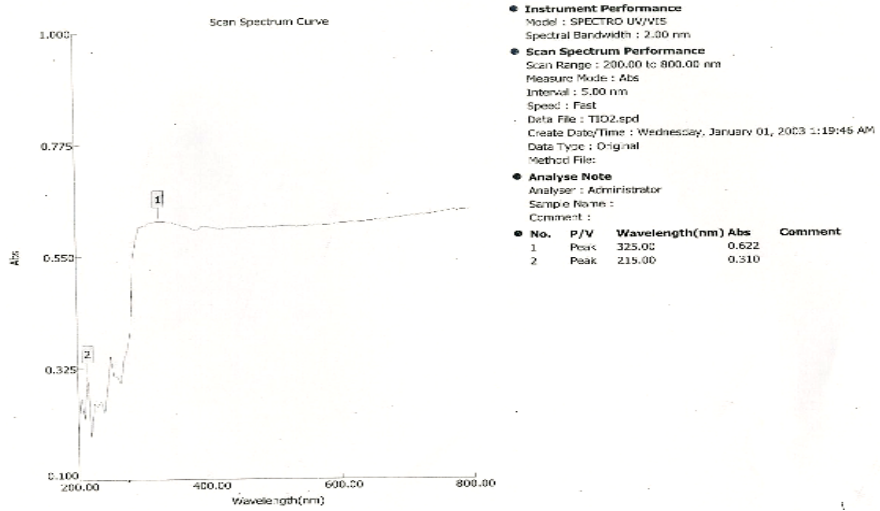

Figure 2. Graph of UV

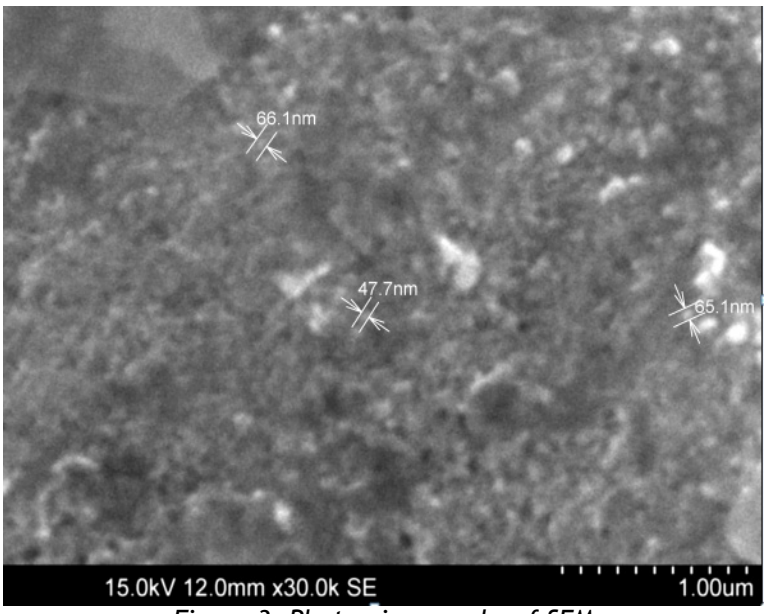

Figure 3. Photomicrographs of SEM

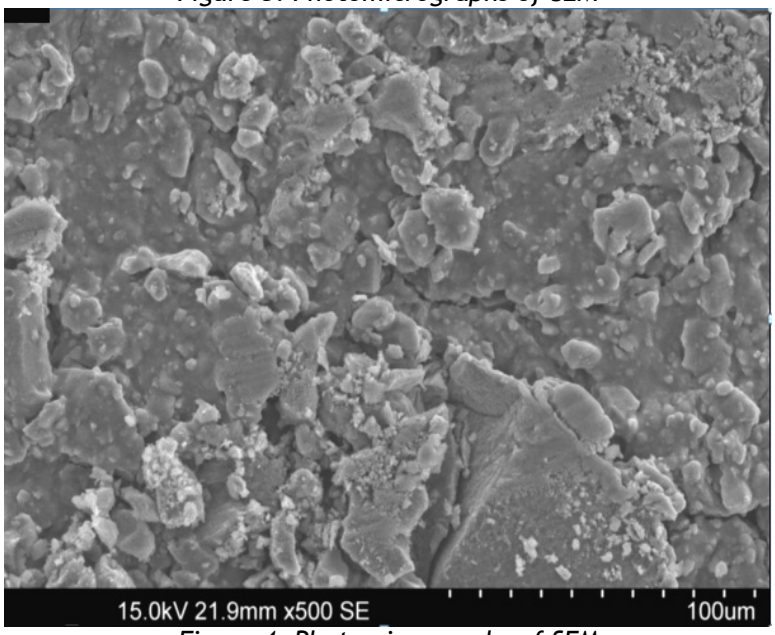

Figure 4. Photomicrographs of SEM

Figure 3 shows the cross sectional image of SEM of as prepared TiO_2 Nanoparticles calcined at 500°C.SEM images depicted TiO_2 particles at 1μm are Nano-sized in the range of ≥ 45 nm.

Figure 4 shows the image of SEM which gives the difference of TiO_2 Nanoparticles by varying scale at 100 μm.

CONCLUSIONS

Nanophased TiO_2 particles are synthesized by sol-gel method at room temperature with Titanium Tetrachloride and Ethyl alcohol as feed material.Titania particles are amorphous in nature which are then transformed into Anatase TiO2 Nanoparticles by calcining at 500°C and characterized by FTIR, UV-vis and SEM.Results are found that as prepared TiO_2 particles are Nano-sized and possess Anatase phase.

REFERENCES

[1] *Cheol Ho Heo, Soon-Bo Lee, Jin-Hyo Boo, Deposition of TiO_2 thin films using magnetron Sputtering method and study of their surface characteristics, Thin Solid Films 475 (2005) 183.*

[2] *K. Mogyorosi, I. Dekany, J. H. Fendler, Preparation and characterization of clay mineral, Intercalated Titanium Dioxide Nanoparticles, Langmuir 19, 2938 (2003).*

[3] *Desong Wang, Libin Xiao, Qingzhi Luo, Xueyan Li, Jing An, Yandong Duan, Highly efficient visible light TiO_2 photocatalyst prepared by sol-gel method at temperatures lower than 300 ◦C, Journal of Hazardous Materials 192, 150 (2011).*

[4] *M. Alam Khan, M. Shaheer Akhtar, O-Bong Yang, Synthesis, characterization and application of sol-gel derived mesoporous TiO_2 nanoparticles for dye-sensitized solar cells, Solar energy 84, 2195 (2010).*

[5] Krishnamurthy Prasad, D.V. Pinjari, A.B. Pandit, S.T. Mhaske, Phase transformation of nanostructured titanium dioxide from anatase-to-rutile via combined ultrasound assisted sol-gel technique, Ultrasonics Sonochemistry 17, 409 (2010).
[6] M. Hussain, R. Ceccarelli, D. L. Marchisio, D. Fino, N. Russo, F. Geobeldo, Synthesis, characterization, and photocatalytic application of novel TiO2 Nanoparticles, Chemical Engineering Journal 157, 45 (2010).
[7] A. Ahmad. Dr., Gul Hameed Awan, Salman Aziz, Synthesis and applications OF TiO_2 NanoParticles, Pakistan Engineering Congress, 70th Annual Session Proceedings, 70, 407 (2007).
[8] Yassine Bessekhouad, Didier Robert, Jean Victor Weber, Synthesis of photocatalytic TiO_2 nanoparticles: optimization of the preparation conditions, Journal of Photochemistry and Photobiology A: Chemistry 157,47 (2003).
[9] Liqiang Jing, Shudan Li, Shu Song, Lianpeng Xue, Honggang Fu, Investigation on the electron transfer between anatase and rutile in nano-sized TiO_2 by means of surface photovoltage technique and its effects on the photocatalytic activity, Solar Energy Materials & Solar Cells 92, 1031 (2008).
[10] Liqun Mao, Qinglin Li, Hongxin Dang, Zhijun Zhang, Synthesis of nanocrystalline TiO_2 with high photoactivity and large specific surface area by sol-gel method, Materials Research Bulletin 40, 202 (2005).
[11] Y. Q. Wang, X. J. Yu, D. Z. Sun, Synthesis, characterization, and photocatalytic activity of $TiO_{2-x}N_x$ nanocatalyst, Journal of Hazardous Materials 144, 329 (2007).
[12] In Chan Baek, Muga Vithal, Jeong Ah Chang, Jun-Ho Yum, Md. K Nazeeruddin, Michael Gratzel, Yong-Chae Chung, Sang Il Seok, Facile preparation of large aspect ratio ellipsoidal anatase TiO_2 nanoparticles and their application to dye-sensitized solar cell, Electrochemistry Communications 11, 909 (2009).
[13] Kye Sang Yoo, Tai Gyu Lee, Jinsoo Kim, Preparation and characterization of mesoporous TiO_2 articles by modified sol-gel method using ionic liquids, Microporous and Mesoporous Materials 84, 211 (2005).

ANNALS of Faculty Engineering Hunedoara

- International Journal of Engineering