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RESEARCH ARTICLE

Titania Nanoparticles Synthesized by Sol-Gel Technique

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Abstract: Titania (TiO_2) nanoparticles were prepared by combining bulk titania with trisodium citrate solution at room temperature without calcination. The formation of titania nanoparticles was confirmed from XRD and by the dominant FTIR peaks at 621 cm^{-1}, 412 cm^{-1}. UV-Visible analysis shows the occurrence of strong red shift, confirming the presence of nanoparticles which is essential for higher photo catalytic activities. TGA analysis reveals that the synthesized titania nanoparticles were thermally stable up to 700 °C. SEM image shows that the particles of the synthesized sample are in nanometer range which is in accordance with UV-Visible studies.

Keywords: Titania, Nanoparticle, Synthsis, Sol-gel technique

Introduction

Titania / Titanium dioxide (TiO_2) nanoparticles is a promising material, widely used in many applications due to its high photo catalytic activity[1] , excellent gas-sensitive properties[2] , dielectric properties[3] , high stability, low cost and non-toxicity[4]. The unique optical property and chemical stability of titania makes it well suited in the splitting of water[5] and in the photo-oxidation[6] processes. As nanosized particles, these materials exhibit broad band UV absorption, a benefit that currently has been exploited in sunscreen applications. Also, the addition of nanoparticles would likely enhance the stiffness, toughness and service life of polymeric materials[7].

The sol-gel method is an attractive method for the synthesis of titania[8]. Since this method is carried out in solution, tailoring of certain desired structural characteristics such as compositional homogeneity, grain size, particle morphology and porosity is possible. A uniform distribution of the particles is important for optimal control of grain size and micro structure to maintain high reliability[9,10].

The focus of the present work is to synthesize high efficient nanosized titania particles having large surface area and to characterize the prepared sample using techniques like XRD, SEM, FTIR, UV- Vis, Fluorescence and TGA.

Experimental

Titanium dioxide (TiO_2) [Loba Chemi, India] and Trisodium citrate ($Na_3C_6H_5O_7$) [Reachem, India] have been used in the synthesis of titania nano particles.

Experimental procedure

A quantity of 1% TiO_2 was dissolved in 50 mL of distilled water and subjected to stirring at a temperature of 45 °C. Then 4% trisodium citrate was dissolved in 50 mL of distilled water which was then added drop wise into the reaction media with a feed rate of 0.42 mL/min to maintain the ratio of titania and trisodium citrate as 1:4. The stirring was continued for 2 h. The temperature of 45 °C was maintained till the end of the reaction. Drying was carried out using a conventional oven at 110 °C for 24 h to obtain titania nanoparticles.

Results and Discussion

X-ray diffraction (XRD) analysis

Figure 1 shows the X-ray diffraction (XRD) patterns of the synthesized titania sample. Peaks at 25^o, 37^o, 38^o, 48^o, 54^o, 55^o, 62^o, 69^o, 75^o (JCPDS-781510) were observed which confirms the formation of anatase phase of titania[11,1]. The appearance of sharp diffraction patterns indicates the small size, high purity and crystallinity of the synthesized sample[12]. The particle size (t) was calculated from the XRD spectra using the well-known Debye-Scherrer formula.

$$t = K\lambda / \beta \mathrm{Cos}\theta \qquad (1)$$

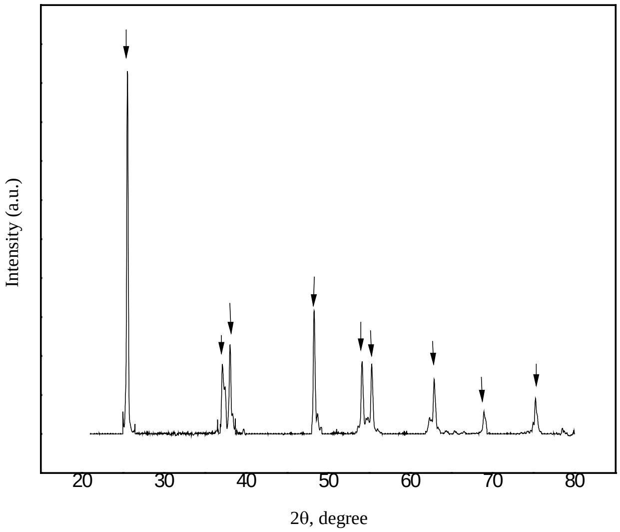

Figure 1. XRD spectra of synthesized titania

Where t is the particle size, λ the x-ray wavelength, β the full width at half maximum (FWHM) of the diffraction peak (radian), K is a coefficient (0.89) and θ is the diffraction angle at the peak maximum. In the present study, the particle size of the titania prepared by sol gel technique was found to be 37 nm.

Scanning electron microscopy (SEM) analysis

Scanning electron microscopy (SEM) of the sample was carried out to estimate the surface morphology of the sample. XRD and SEM together provide exact knowledge regarding the particles size and characteristics of the synthesized sample. Figure 2 shows the SEM images of the synthesized titania sample. The particles are spherical in shape with uniform size. It is clear from the image that titania particles are evenly distributed on the surfaces without any aggregation. The average particle size of titania particle was found to be 86 nm which was in accordance with the XRD result.

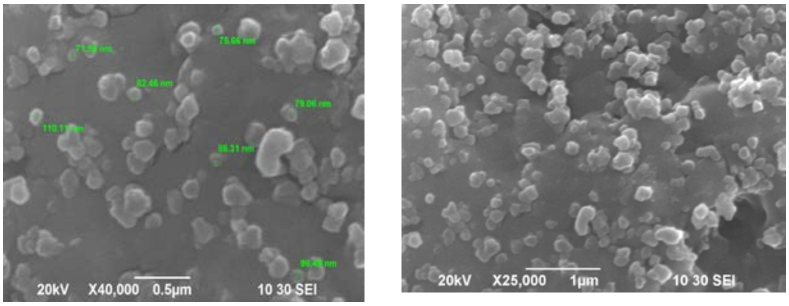

Figure 2. SEM images of synthesized titania with different magnification

Fourier transform infrared (FTIR) spectra analysis

FTIR spectra of synthesized titania sample is as shown in Figure 3. Peaks at 3450 cm^{-1}, 3377 cm^{-1} and 3267 cm^{-1} correspond to stretching vibration of O-H bond. Peaks observed at 2962 cm^{-1}, 2922 cm^{-1} and 2854 cm^{-1} corresponds to the C-H stretching vibrations. The sharp peak at 1589 cm^{-1}, 1020 cm^{-1} corresponds to bending vibrations of water. Peak observed at 1658 cm^{-1} corresponds to the C=O vibrations[13]. The peak at 1394 cm^{-1} and 1155 cm^{-1} corresponds to C-O vibrations. Peaks observed at 621 cm^{-1}[11, 12], 594 cm^{-1}, 549 cm^{-1} and 412 cm^{-1} corresponds to Ti-O vibrations[13-16].

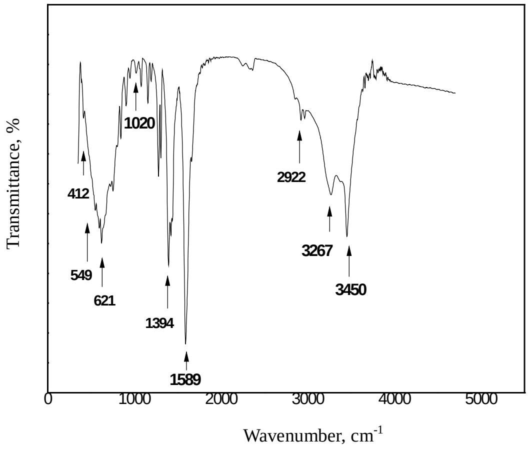

Figure 3. FTIR spectra of titania nanoparticles

UV-Visible spectra analysis

To investigate the optical absorption properties of titania sample under present investigation, UV-Vis study was carried out (Figure 4). The band gap energy (E_g) was calculated according to the equation:

$$E_g = h\,c/\,\lambda \tag{2}$$

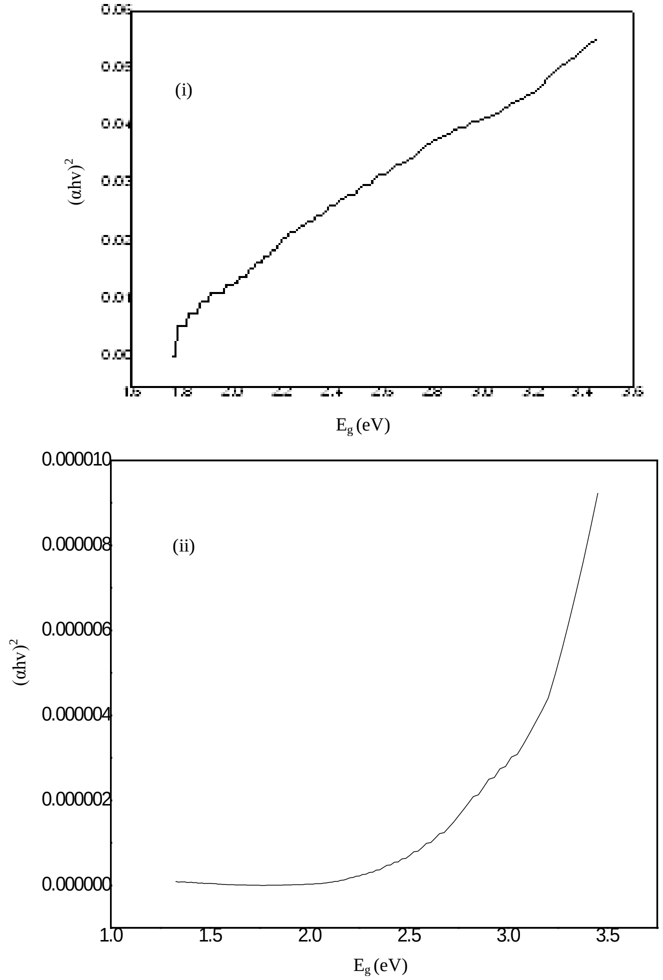

Figure 4. (i) UV-Vis spectra and (ii) Band gap plot of synthesized titania

Where E_g is the band gap energy (eV), h is Planck's constant, c is the velocity of light (m/s) and λ is the wavelength in nm[1]. The absorption at the wavelength of 408 nm can be attributed to the absorption of the titanium species and the quantum size effect of the nanoparticles as reported by Xu *et al.*[17]. The band gap value calculated from the above formula has been 3.0 eV. Earlier reports shows the average value for absorption to be 385 nm and the corresponding band gap energy[18-20] for bulk titania as 3.2 eV. The resulting red shift of the absorption is due to the change of particle size. The quantity of photons reaching the core of a spherical particle depends on the size of the particle and the optical properties of the titania.

The smaller crystals are generally poorer light scattered than larger crystals. Also, the penetration of light into the particle is influenced by the superficial morphology of the particles[21]. Particles formed from large titania crystals have smoother surface than the particles made from small crystals. On the smooth surface, the incident photons are scattered and small crystals allows a greater number of scattered photons to penetrate into the particle.

Fluorescence analysis

Fluorescence emission spectrum of the synthesized titania sample is shown in the Figure 5. The emission peak is found at 512 nm with respect to the excitation peak at 507.5 nm. The increasing wavelength suggests the strong red shift in the optical spectra with respect to the bulk titania. This is due to the presence of more oxygen vacancies on the surface of titania nanoparticles and the size of the particle was fine and hence the average distance of the electrons could make the oxygen vacancies very easily bind electrons to form excitons. Thus, the exciton energy level near the bottom of the conduction band could make the strong excitonic spectrum[22,23]. This narrower band-gap will facilitate easy excitation of electrons from the valence band to the conduction band which will result in higher photo catalytic activities[24].

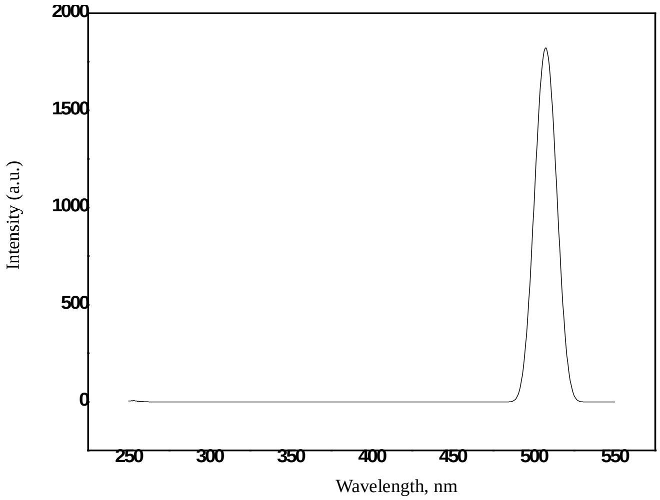

Figure 5. Fluorescence spectra of synthesized titania

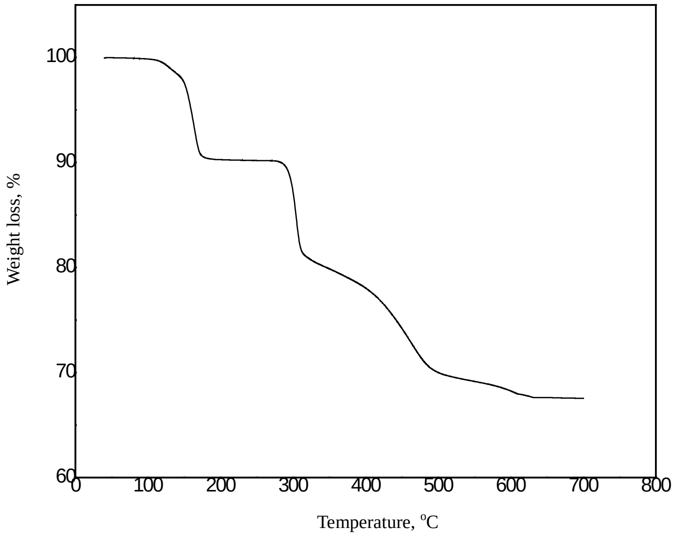

Figure 6. Thermogram of synthesized titania

Thermo gravimetric (TGA) analysis

TGA curve of titania nanoparticles is shown in Figure 6. The degradation has occurred in three steps. The first degradation from 100 °C to 180 °C is attributed to the removal of water from the surface (mass loss of about 10%). The second degradation is from 180 °C to 300 °C. 13% mass reduction has been observed in this region. This is due to the dehydration and removal of organic residues[1]. The third degradation from 500 °C to 600 °C results in an additional mass loss of about 10%. Above 700 °C, an essentially constant mass (67% sample) has been found indicating the thermal stability of the sample.

Conclusion

The titania nanoparticles were successfully synthesized using the simple and cost effective sol gel technique. XRD shows the formation of high purity titania nanoparticles. Calculated Band gap energy from the UV Visible spectra analysis has been found to be 3.0 eV. This wide band gap makes it well suited for solar cell applications. A strong red shift observed in fluorescence analysis reveals higher photocatalytic activity of the synthesized titania. TGA analysis confirms the thermal stablity of titania particle (nearly 67% remained after 700 ^{o}C). SEM analysis reveals the average particle size of the synthesized titania to be 86 nm.

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