SYNTHESIS AND CHARACTERIZATION OF TITANIUM DIOXIDE NANOPARTICLES
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Abstract

Titanium dioxide nanoparticles were successfully synthesized. The as-synthesized TiO₂ nanoparticles were subjected to X-ray diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), UV-Vis spectroscopy and Scanning Electron Microscopy (SEM) studies. XRD confirms the formation of anatase phase. The crystallite size of TiO₂ nanoparticles calculated from the broadening of diffraction peaks using Scherer formula was approximately 19.72 nm. From the FTIR spectrum it was observed that the strong band in the range of 900 and 500cm⁻¹ was associated with the characteristic vibrational modes of Ti-O bond and O-Ti-O bridge. This confirms that TiO₂ phase was formed. The optical properties of the synthesized nanoparticles indicate the quantum confinement effect. The absence of any absorption peak in the spectra of as-synthesized TiO₂ nanoparticles were in good agreement with the wide bandgap nature of samples and inability to absorb in the visible range.

Keywords: TiO₂; Nanoparticles; Optical properties; bandgap; Scanning Electron Microscopy; X-ray diffraction.

1. Introduction

Today physical, biological scientists and engineers have an impressive array of powerful and elegant tools for obtaining qualitative and quantitative information about the composition and structure of matter. The development of these tools began over two centuries ago and the search still continues. The use of instrumentation is an exciting and fascinating part of any analysis that pervades all the areas of pure and applied sciences. In
recent years, metal oxide nanoparticles have attracted much attention by their potential application in diverse fields including catalysis, magnetic recording media, microelectronics, medicine. For example, titanium oxide nanoparticles are very important due to their various applications; removing the environmental pollution, sterilization and restraining virus, defending UV, keep rust away, and depigment [1].

TiO$_2$ is also a potent photocatalyst that can break down almost any organic compound when exposed to sunlight, and a number of companies are seeking to capitalize on titanium dioxide’s reactivity by developing a wide range of environmentally beneficial products, including self-cleaning fabrics, auto body finishes, and ceramic tiles. Titanium dioxide is a well-known photocatalyst for water and air treatment as well as for catalytic production of gases. Titanium dioxide’s photocatalytic characteristics are greatly enhanced due to the advent of nanotechnology. At nano-scale, not only the surface area of titanium dioxide particle increases dramatically but also it exhibits other effects on optical properties and size quantization. An increased rate in photocatalytic reaction is observed as the redox potential increases and the size decreases. In the present work, Titanium Di-Oxide (TiO$_2$) nanoparticles were synthesized which was characterized by X-Ray Diffraction (XRD), UV-Visible spectroscopy (UV) studies, Photoluminescence (PL), Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM).

2. Synthesis of TiO$_2$ nanoparticles

For the synthesis of TiO$_2$ nanoparticles, 1:0.05 volume ratio of isopropanol and titanium isopropoxide was added and stirred the contents using magnetic stirrer for 30 minutes. The metal oxide gel was produced by increasing the pH by drop wise addition of (1N) NH$_3$ solution. The resultant solution was stirred for 24 hours and kept for 1 day aging. The precipitate was washed several times with distilled water and dried in oven for 24 hours to remove the solvent. Removal of residual organics and the stabilization of the materials were carried out by calcination for 5 hrs at 500$^\circ$C.

The X- Ray Diffraction (XRD) analysis was conducted by PAN analytical X’pert – PRO diffractometer using monochromatic Cu K$_\alpha$ radiation ($\lambda = 1.5406$ Å) running at 40 kv and 30mA. The optical properties of the prepared nanoparticles were studied using UV-Vis absorption spectrometer (Double Beam Spectrometer- UV-1700) with samples in
quartz cuvette. FTIR measurements were obtained on a Nexus 670 FTIR instrument with the sample as KBr pellets.

3. Result and discussion

3.1. X-ray diffraction (XRD) analysis

The X-ray diffraction pattern of the TiO$_2$ nanoparticles calcinated at 500°C for 5 hours demonstrated the formation of anatase phase of TiO$_2$ as shown in Fig 1. It was observed that all the diffraction peaks are in good agreement with a reference pattern (JCPDS no. 21-1272). The peak position at 25.3°, 37.8°, 48.0°, 53.9°, 55.0°, 62.7°, 68.77° and 75.49° corresponds to [101], [004], [200], [105], [211], [204], [116] and [215] respectively can be indexed to the reflection planes of body centered tetragonal structure of anatase TiO$_2$. The broad peak indicates that the particles are of very small crystallite size.

![Fig. 1 X-ray diffraction pattern of TiO$_2$ nanoparticles](image)

The relative crystallite size of TiO$_2$ nanoparticles was determined from the line broadening using the Scherer equation [2] $D = \frac{0.9\lambda}{\beta\cos\theta}$, where $D$ – Crystallite size, $\lambda$ – Wavelength, $\beta$ – Full Width half maximum, $\theta$ – Angle of diffraction. The crystallite size of TiO$_2$ nanoparticle calculated from line broadening of diffraction peaks using Scherer formula was approximately 19.72 nm.
3.2 Fourier Transform Infrared Spectroscopy Analysis

The presence of functional groups in TiO$_2$ nanoparticles was quantitatively analyzed by infrared spectrum. The spectra were recorded in the range of 500-4000 cm$^{-1}$. The recorded spectra of as-synthesized TiO$_2$ were shown in the Fig.2. From the spectrum, it can be observed that the strong band in the range of 900 and 500 cm$^{-1}$ was associated with the characteristic vibrational modes of Ti-O bond and O-Ti-O Bridge [3]. This confirms that the TiO$_2$ phase was formed. The absorption peaks around 3743 cm$^{-1}$ to 2306 cm$^{-1}$ may be related to the presence of O-H stretching vibration. The absorption band at 1645 cm$^{-1}$ indicates the presence of O-H bending vibration of free water. The hydroxyl groups on the particles surface contributes to the improvement of the photocatalytic activity by the interaction with photo generated holes giving better charge transfer, which inhibits the recombination of electron-hole pair [4].

![Fig.2 FT-IR Spectrum of as-synthesized TiO$_2$ nanoparticles](image)

3.3 UV-vis Absorption Spectroscopy

The UV-Vis absorption study was carried out in order to characterize the optical absorbance of the as-synthesized samples. The absorption spectra of the as synthesized TiO$_2$ was shown in Fig.3. The absence of any absorption peak in the spectra of as-synthesized TiO$_2$ nanoparticles were in good agreement with the wide
band gap nature of samples and its inability to absorb in the visible range. TiO$_2$ nanoparticles were capable of absorbing light only in the ultra-violet region and transmit light. The absorption edge for pure TiO$_2$ observed at 380 nm. The band gap energies ($E_g$) of as-synthesized TiO$_2$ nanoparticles observed at $E_g$=3.25eV which are larger than the value of 3.2 eV for the bulk TiO$_2$.

![Fig. 3. UV-Vis Spectrum of TiO$_2$ nanoparticles](image)

**Fig. 3. UV-Vis Spectrum of TiO$_2$ nanoparticles**

*Inset shows the corresponding plot of $(\alpha h\nu)^2$ vs photon energy (hv)*

### 34 Photoluminescence

![Fig. 6.5 Photoluminescence spectrum (PL) of TiO$_2$ nanoparticles](image)

**Fig. 6.5 Photoluminescence spectrum (PL) of TiO$_2$ nanoparticles**
PL emission spectra were widely used to investigate the efficiency of charge carrier trapping, immigration, and transfer to understand the fate of photo induced electron-hole pair in semiconductors [5]. Fig.4 shows the photoluminescence spectra for as-synthesized TiO$_2$ at the excitation wavelength of 350 nm. The PL spectra shows two peaks at 420 nm and 448 nm for as-synthesized TiO$_2$.

**3.5 Scanning Electron Spectroscopy**

Scanning electron microscopy (SEM) has been used to characterize the size, shape and morphologies of formed TiO$_2$ nanoparticles. The SEM image of Fig 5 showing the presence of clustered tetrahedral TiO$_2$ nanoparticles with average size of 31.33 nm ranging from 27 to 33 nm size were observed at Fig.5.

![Scanning electron microscopy of TiO$_2$ nanoparticles](image.png)

**Fig. 5 Scanning electron microscopy of TiO$_2$ nanoparticles**

The presence of primary particles and small aggregates nanoparticles observed in Fig. 5. It is observed that the nanoparticles tend to grow in size and agglomerated with the energy received from the dopant atoms. It is quite reasonable to assume that the introduction of large neighbouring atoms into the matrix without altering the overall structure may cause redistribution and lead to agglomeration.

**4. Conclusion**

Titanium dioxide (TiO$_2$) nanoparticles were successfully synthesized using titanium isopropoxide and ammonium fluoride as precursor as sol-gel method. The as-synthesized TiO$_2$ nanoparticles were calcinated at 500°C for 5 hours. The as-
synthesized TiO$_2$ nanoparticles were subjected to X-Ray Diffraction spectroscopy (XRD), UV-Visible spectroscopy (UV) studies, Photoluminescence (PL), Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) studies. From XRD analysis it was observed that all diffraction peaks are in good agreement with a reference pattern (JCPDS no. 21-1272). The crystallite size of as-synthesized TiO$_2$ nanoparticles calculated from the broadening of diffraction peaks using Scherer formula were approximately 19.72 nm. From the FTIR spectrum it was observed that the strong band in the range of 900 and 500 cm$^{-1}$ was associated with the characteristic vibrational modes of Ti-O bond and O-Ti-O bridge. This confirms that TiO$_2$ phase was formed. The lower PL intensity indicates the lower recombination rate of electron-hole under light irradiation. This result confirms TiO$_2$ could reduce the electron-hole recombination rate and hence it enhances photocatalytic activity. SEM shows the formation of anatase crystallites and tetrahedral structures.

References
