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Synthesis, Structural and Optical Properties of Co Doped TiO₂ Nanocrystals by Sol-Gel Method

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ABSTRACT. A TiO₂ nanoparticle doped with cobalt was synthesized by sol-gel technique employed at room temperature with appropriate reactants. In the present case, we used titanium tetra isoprotoxide (TTIP) and 2-propanol as a common starting material and the obtained products were calcined at 450 °C. From the Powder XRD data the particle size was calculated by Scherrer method. The FE-SEM analysis shows the morphology of cobalt doped TiO₂ nanoparticles. The various functional groups of the samples were identified by Fourier transform spectroscopy (FT-IR). The UV-Vis-NIR spectra of cobalt doped TiO₂ material shows two absorption peaks in the visible region related to d-d transitions of Co²⁺ in TiO₂ lattice. Compared to un-doped TiO₂ nanoparticles, the cobalt doped material show a red shift in the band gap.

1. Introduction. Titanium dioxide or titania (TiO₂) is the potential material as semiconductor having high photochemical stability, moderate thermal stability and low cost. Well – disband TiO₂ nanoparticle with very tiny (nm) sizes are hopeful many applications such as pigments, catalytic and adsortments. The Cobalt doped TiO₂ nanocrystals have consumed great attention due to its enhanced photocatalytic activity [1]. Earlier researchers noticed the photocatalytic splitting of water on a TiO₂ electrode under ultraviolet light, many synthesis methods for preparing TiO₂ nanoparticles and their applications in the environmental and energy fields mainly hydrogen storage, water splitting and photovoltaics have been investigated [2]. In recent days, the fine nanoparticles of TiO₂ have attracted a great deal of attention, because of their unique properties as a superior semiconducting material, such as luminous material, solar cell and photocatalyst for photolysis of water and organic compounds and for bactericidal action [3]. In the present paper, the Co-doped TiO₂ nanoparticles have consumed great interest due to its superior photocatalytic activity. In this paper, we report the preparation of Co doped TiO₂ nanoparticles by a sol-gel mode.

2. Experimental procedure

2.1. Preparation of cobalt-doped TiO₂. All the starting materials were of analytical reagent, 90 ml of 2 – propanol was taken as primary precursor and 10 ml titanium (IV) isopropoxide (TTIP) was added into drop wise with vigorously stirred for an hr in order to form solutions. Aqueous solution of cobalt nitrate of desired concentration (5%) was poured slowly drop by drop to that mixture with continued stirring. After aging a day, the solvents were transformed into gel form. The collected gel were dried at 85°C for 24 hr to remove the water and other organic compounds. After that, the dried
A gel was sintered at 450°C for 8 hr in high temperature programmable furnace. Finally, the Co-doped TiO$_2$ nanoparticle were obtained.

3. RESULTS AND DISCUSSION

3.1. Powder X-Ray diffraction studies (PXRD). X-ray diffraction pattern of pure tio$_2$ and co-doped TiO$_2$ were carried out by powder x-ray diffractometer (bruker-d8 advance) using Cu-k$_{α1}$ radiation. Fig.1 shows pure and co-doped TiO$_2$ nanocrystals (ncys) calcined at 450°C for 8 hr. From the (powder x-ray) diffraction patterns shows pure and co-doped TiO$_2$ are in anatase phase. From the XRD patterns shows in the present study are indistinguishable with earlier report [4]. For pure and co doped TiO$_2$ Ncrys, the diffraction peaks occurring at 25.42°, 38.28°, 48.32°, 53.90°, 54.84°, 63.60°, 68.96° and 70.30° have been assigned to the lattice planes (101), (004), (200), (105), (211), (204), (116), (220) and (215) respectively. These lattice planes are attributed to the signals of pure tetragonal anatase phase of TiO$_2$ with a space group i4/ and (jcpds file no. 78-2486). From the XRD data, the avaerage Ncys size was calculated by debye- scherrer formula:

\[ d = \frac{k\lambda}{\beta \cos \theta}, \]

where d is the crystalline size, k is the shape factor, λ is the wavelength in nm, β is the full width at half maximum, θ is the reflection angle and the results were presented in table.1

![Fig. 1. PXRD pattern of Pure TiO$_2$ and Co-doped TiO$_2$.](image)

<table>
<thead>
<tr>
<th>Samples</th>
<th>Crystalline Size (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure TiO$_2$</td>
<td>16.21</td>
</tr>
<tr>
<td>5% Co-doped TiO$_2$</td>
<td>20.589</td>
</tr>
</tbody>
</table>

3.2. FT-IR studies. Fig.2 shows the FT-IR spectrum of pure and co-doped TiO$_2$ ncys calcined at 450°C for 8 hr. From the spectrum there are two prominent peaks present between 400 and 600 cm$^-1$ [5]. The presence of band at 2854 cm$^-1$ was due to the C-H bond of the organic compound [6]. There are two prominent absorption bands present at 3421 and 1645 cm$^-1$ in the materials can be recognized.
as the stretching and bending vibrations of H₂O molecules. From the Fig. 2 shows the intensity of two bands in Co is weakened compared with pure TiO₂. The peaks in between 2915 and 2869 cm⁻¹ are assigned to C-H stretching vibrations of alkenes groups. A broad absorption band between 500 and 1000 cm⁻¹ is attributed to the vibration of ti-o-ti association in tio₂ ncys [7].

![FT-IR spectrum of CO-doped TiO₂](image)

**Fig. 2.** FT-IR spectrum of CO-doped TiO₂

### 3.3. Optical absorption studies.

Fig. 3 shows the optical absorption spectrum of pure and Co-doped TiO₂ NCYS calcined at 450°C for 8 hr. The optical properties and calculated the energy band gap of pure and co doped tio₂ ncys by UV–VIS-NIR absorption spectroscopy as shown in Fig. 3. The absorbance can vary depending upon the particle size, oxygen deficiency, purity of the material, etc. The relation connecting the absorption coefficient (α) of semiconductors, the incident photon energy (hν) and optical band gap (Eg), from the TAC’S relation the calculated optical energy band gap of synthesized pure and co-doped TiO₂ NCYS are found to be 3.25 ev and 3.39 ev. In this results were coincides the earlier reported the value 3.23 ev for pure anatase phase tio₂ [8-9].

![UV-Vis-NIR spectrum of Co-doped TiO₂](image)

**Fig. 3.** UV-Vis-NIR spectrum of Co-doped TiO₂.

### 3.4. EDAX studies.

The elemental analysis of pure and Co-doped TiO₂ NCys were analyzed by using electron diffraction X-ray analysis (EDAX). The spectrum shows, the strong X-ray peaks associated with Ti Kα and O Kα were found in the EDAX spectrum Fig.4a. Fig.4b shows depicted the successful doping of Co into TiO₂ NCys.
3.5. FE-SEM studies. Scanning electron microscope (SEM) images of pure and Co-doped TiO$_2$ NCys synthesized by sol gel method and calcined at 450°C for 8 hr are shown in Fig. 5a and Fig. 5b. It is clearly shows very closely packed spherical and bubbles shaped nanocrystals (NCys).

Summary. The Pure TiO$_2$ and Co-doped TiO$_2$ nanocrystals have been synthesized by sol gel method at room temperature. The synthesized materials were calcined at 450°C for getting anatase phase. From the PXRD data the synthesized material shows a space group I4$_1$. The calcined samples were characterized by the techniques like PXRD, FT-IR, Optical absorption, FESEM and EDAX. From the results of PXRD patterns, it is confirmed that the TiO$_2$ was in anatase phase with crystalline size in the range of 16.21 to 20.58 nm. The presents of functional groups were identified by FT-IR analysis. The UV cut of wavelength and optical band energy gap of the undoped and doped materials are have the values between 3.25 and 3.39 eV. The SEM with EDAX images confirmed the spherical and bubbles morphology of the products.

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References


Cite the paper