

Synthesis of Yb³⁺ Doped TiO₂ Nano Particles Powder as IR Filter via Sol-Gel

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ABSTRACT

Ytterbium doped TiO₂ nano-particles were prepared via Sol-Gel Technique under varying conditions to investigate the effects of Ytterbium ion doping on the Titania optical properties in MID IR range. X-ray diffraction analyses show that prepared Yb³⁺ doped TiO₂ samples have polycrystalline structure in Anatas phase. FTIR spectrums for pure and doped samples after annealing process show a single transmission peak at wave number around 1145 cm⁻¹. Transmission rate of this peak depends on Yb³⁺ concentrations and its value rises from 1.82% (for pure TiO₂) to 58.1% (for doped with 1.13 wt% Yb³⁺). Slightly peak shift occurs at a lower doping rate with no further response to higher concentration rate. FTIR spectra gives a good indication in direction of preparation of optical band-pass filter at a wavelength around 8.733 μm (~1145 cm⁻¹).

Keywords: Sol-Gel; Nano Technology; TiO₂; MID IR Optical Filter

1. Introduction

Titanium dioxide as a semiconductor photocatalyst has attracted a great deal of attention due to its scientific and technological importance. Nano-particles TiO₂ has promising applications in many fields such as photocatalyst for environmental purification, solar energy conversion, sensors, self-cleaning, super-hydrophilic smart material and so on [1-2].

One of the most important aspects of TiO₂ is its photocatalytic activity of degrading various organic pollutants with its high catalytic efficiency, high chemical stability, non-toxicity and low cost. Despite its promising properties, however, there are several problems that limit the photocatalytic activity of TiO₂. For example, due to the large band gap, it can be photo-excited only in the ultra-violet region, which comprises less than 5% of the overall solar energy spectrum. Another problem is the recombination of electrons and holes that generally take place in the large TiO₂ particles. It substantially reduces the number of free charges on the surface and lowers the photocatalytic activity. Therefore, several methods have been employed to improve the photocatalytic efficiency of TiO₂.

Recently, doping lanthanide into TiO₂ attracted much attention [3-7]. Lanthanide ions are known for their abil-

ity to form complexes with various Lewis bases (e.g. acids, amines, aldehydes, alcohols, thiols, etc.) due to the interaction of these functional groups with the f-orbital of the lanthanides [6]. Thus, incorporation of lanthanide ions into a TiO₂ matrix could provide a way to concentrate the organic pollutant on the semiconductor surface and therefore enhance the photoactivity of Titania [6].

Sol-gel process is one of the most successful techniques for preparing nanocrystalline metallic oxide materials due to low cost, easy of fabrication (flexibility) and low processing temperatures. Generally, in a typical sol-gel process, a colloidal suspension or a sol is formed due to the hydrolysis and polymerization reactions of the precursors, which on complete polymerization and loss of solvent leads to the transition from the liquid sol into a solid gel phase. The wet gel can be converted into nanocrystals with further drying and hydrothermal treatment [8,9].

2. Experimental

2.1. Samples Preparation

The doped and un-doped titania nanoparticles were synthesized by sol-gel method from Titanium (IV)-isopropoxide (TTIP) (Aldrich 98%), Ethanol (EtOH 99.9%)

from GCC, hydrochloric acid (HCl, 34.5%) from BDH and Ytterbium(III) oxide (Aldrich). Deionized water was used for the hydrolysis of (TTIP) and preparation of pure and doped TiO_2 Sol. The final solution was left for 30 minutes under magnetic stirring. The reaction was performed at a cooler water jacket (*i.e.* the solution was cooled by water at temperature $10^\circ\text{C} - 15^\circ\text{C}$). The amount of each chemical in this procedure was TTIP:H₂O:EtOH:HCl = 1:1:10:0.1 in molar ratio. The gelation of monoliths was achieved by pouring the sol into covered glass tube at room temperature. After aging for 24 hour, first drying occurred for 1 hour at temperature 60°C . Then samples left in room temperature without covers in order to permit solvent evaporation through the drying process. The doping rate of samples with Yb^{3+} equal to: 0 wt%, 0.21 wt%, 0.68 wt% and 1.13 wt%.

2.2. Samples Characterization

All samples were calcinated for 2 hour in a box furnace operating at 500°C in an ambient atmosphere. Structural characterizations of the doped and un-doped TiO_2 samples were done by X-ray diffraction (XRD). $\theta - 2\theta$ scans were recorded using ITAL-STRUCTURE diffractometer equipped. While Mid-IR spectra were obtained for the prepared samples using FT-IR spectrometer, Shimadzu, on KBr pellets of the samples

3. Result and Discussion

The X-ray diffraction spectrums (XRD) were illustrated in **Figure 1** for the doped and undoped samples after annealing process, which show that prepared samples have polycrystalline structure in Anatas phase [10-12].

The FTIR spectrums were illustrated in **Figures 2** and **3** for the samples before and after annealing process respectively. The peaks at 667 cm^{-1} , 505 cm^{-1} and 447 cm^{-1} are attributed to Ti-O bond. The peak at 667 cm^{-1} refers to symmetric O-Ti-O stretch while peak at 447 cm^{-1} and 505 cm^{-1} are due to the vibration of Ti-O bond [13-14]. Another two bands were appeared at about 1600 cm^{-1} and 3400 cm^{-1} . These two absorption bands are attributed to the characteristics vibration of O-H bond in water molecules [15,16], and indicating that the drying process at 60°C does not completely trap the water molecules from the pores of Titania network. After annealing process at 500°C the last two absorption bands were disappeared from FTIR spectrums (**Figure 3**).

The important result that could be seen from the FTIR spectra is the existence of transmission peak centered at around 1145 cm^{-1} . Annealing process at 500°C improves the transmission rate of this peak. Furthermore, doping with Yb^{3+} ion makes this peak so much strong and becomes the only major peak could be observed from the FTIR spectrums in range of 4000 cm^{-1} to 400 cm^{-1}

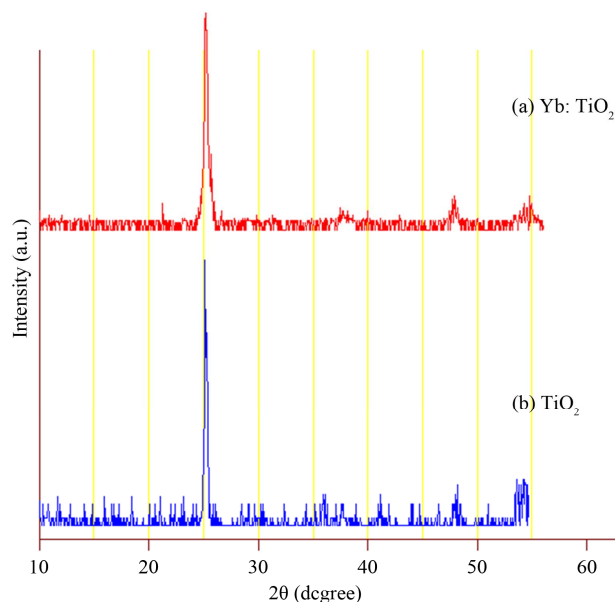


Figure 1. XRD spectrums for samples after annealing process. (a) Yb:TiO₂ doped; (b) Pure TiO₂.

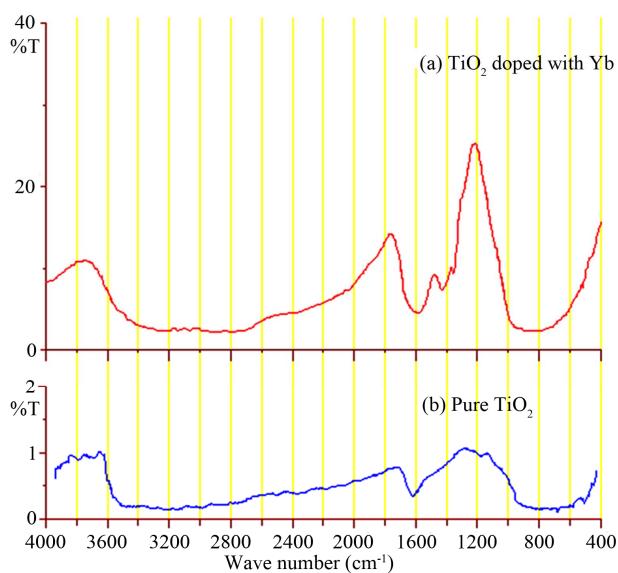


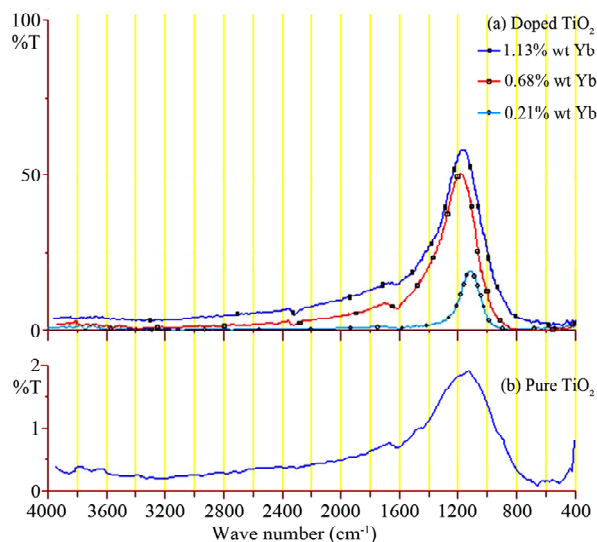
Figure 2. FTIR spectrums for samples before annealing process. (a) TiO₂ doped with Yb³⁺; (b) Pure TiO₂.

(**Figure 3**). This result gives a good indication in direction of production of optical band-pass filter around the wavelength $8.733\text{ }\mu\text{m}$ ($\sim 1145\text{ cm}^{-1}$) [3]. The effect of doping rate on amplitude, FWHM of the band width and maximum peak wavelength is summarized in **Table 1**.

From **Table 1**, it can see that the two parameter transmission rate and FWHM are increase with increasing of doping rate. That's mean the two parameters effected with the amount of doping rate. While the maximum of transmission peak shifted with increasing of doping at the lower doping rate then become stable at higher concentration [3].

Table 1. Transmission peak parameter for Yb:TiO₂ samples after annealing process.

Doping Rate wt% Yb	Transmission rate	FWHM of Band width cm ⁻¹	Wave Number cm ⁻¹	Wave Length μm
0%	1.82%	520	1145	8.733
0.21%	19%	145	1135	8.810
0.68%	50.1%	287	1195	8.368
1.13%	58.1%	354	1189	8.410

**Figure 3. FTIR spectra for samples after annealing process. (a) Yb:TiO₂ with different doped rate; (b) Pure TiO₂.**

4. Conclusion

Both of annealing process and doping with Yb³⁺ increase the photocatalytic efficiency of nanoparticles TiO₂ through enhancement of the transmission rate at the wave number around the 1145 cm⁻¹. The band width of this transmission peak is increase with increasing of doping rate.

The maximal of the transmission peak shifted with increasing of Yb³⁺ concentration at the lower doping rate then became stable at higher concentration.

FTIR spectra gives a good indication in direction of preparation of optical band-pass filter at a wavelength around 8.733 μm (~1145 cm⁻¹).

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