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SYNTHESIS OF SM3+ DOPED WITH TIO2 NANOPARTICLES POWDER AS MID-IR OPTICAL FILTER

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Abstract

Samarium ions doped TiO2 nanoparticles were prepared successfully via Sol-Gel Technique with varying conditions. Effects of Sm3+ doping concentrations on the optical properties in the mid-infrared region was studied. FTIR spectra for pure and doped samples after annealing process show a single transition peak at wave number around 1109 cm-1 and 1116 cm-1 respectively, the wave number of the single transition peak depends on the Sm+3 doping ratio. By Comparing with wavelength of the high transition ratio of the pure TiO2 sample, slightly decries shift on the peak wavelength occur with an increment of doping concentration ratio. The FTIR spectrum gives a good indication the direction of synthesis of optical band-pass filter at a wavelength around 8.964µm (~1116 Cm-1).
Keywords: Sol-Gel; Nano Technology; Tio2; Mid Ir Optical Filter.

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Introduction

Recently, Titanium dioxide has attracted a great interest and attention due to its scientific and technological importance. Nano- particles Titanium dioxide has promising applications in many fields such as photocatalyst for solar energy conversion, environmental purification, self-cleaning, sensors, 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, nontoxicity and low cost. There are several problems that limit the photocatalytic activity of TiO2. For example, due to the large band gap, it can be photo-exited only the ultraviolet region, which comprises less than 5% of the overall solar energy spectrum.
TiO2 doped with lanthanide is target for much research [3-4]. Lanthanide ions are known for their ability 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 [5]. Thus, incorporation of lanthanide ions into a TiO2 matrix could provide a means to concentrate the organic pollutant on the semiconductor surface and therefore enhance the photoactivity of Titania. Hamza investigates the effects of doping with neodymium ions or Ytterbium ion on the Titania optical properties in MID IR range [3,4], he suggests it could use Nd:TiO2 and Yb:TiO2 prepared via sol gel to synthesis of optical band-pass filter at a wavelength around 8.34µm and 8.733µm respectively.

The Sol - gel process is one of the most successful techniques for preparing nanoparticle metal 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 formed due to the hydrolysis and condensation reactions of the precursors. The hydrolysis is the reaction between metal alkoxide M(OR)z and the water to form a metal hydroxide M(OH), the reaction can be written as follows [6,7]:

$$\equiv M-(O\text{-}R)+H_2O \underset{\text{Re-estrification}}{\overset{\text{Hydrolysis}}{\rightleftharpoons}} \equiv M-OH+ROH \text{----------} (1)$$

The condensation reaction will start when the M (OH) is present in the solution. There are two types of condensation, first one is water condensation; whereas the second is alcohol condensation. Both reactions are given by [6,7]:

$$\equiv \mathrm{M-OH} + \equiv \mathrm{M-OH} \underset{\text{Hydrolysis}}{\overset{\text{Water Condensation}}{\rightleftharpoons}} \equiv \mathrm{M-O-M} \equiv +\mathrm{H_2O} \ldots\ldots\ldots(2)$$

$$\equiv \mathrm{M-(O-R)} + \equiv \mathrm{M-OH} \underset{\text{Hydrolysis}}{\overset{\text{Alcohol Condensation}}{\rightleftharpoons}} \equiv \mathrm{M-O-M} \equiv +\mathrm{ROH} \quad \ldots(3)$$

The wet gel could be converted into nanoparticle with further drying and hydrothermal treatment. In the present work, Sol Gel method is employed to prepare of Titanium dioxide doped with Samarium ions. The prepared samples will be analyses with FTIR technique to investigate the capability of the sample to be as optical band-pass filter at Mid-IR optical filter

Experimental

Samples Preparation

The pure TiO2 and TiO2 doped Sm3+ samples were synthesized by sol–gel method from Titanium iso–propoxide (TTIP) (Aldrich 98%), hydrochloric acid HCl 34.5% of BDH company, deionized water, Ethanol (EtOH 99.9%) from GCC company and Samarium (III) chloride (Aldrich), Figure (1) summarized the whole process. The doping ratio of samples with Sm3+ equal to1.18% ppm, 2.24% ppm,3.3% ppm and 4.6% ppm. The Deionized water was used for the hydrolysis of (TTIP) and preparation of pure and doped TiO2 sol. The performed of reaction process was carried out at room temperature. The ratio of each chemical in this procedure was TEIP:H2O:EtOH:HCl= 1:1:10:0.1 in molar ratio. All solution was prepared as follows: 1 mole of Titanium (IV) - ISO–propoxide (TTIP) and ethanol (EtOH) were mixed and stirred for 10 min. At the stirring time, 0.1M catalysts in water were added drop wise to the solution till the ratio of water to TTIP equal to 2. Samarium (III) chloride was solved in ethanol and used to mixing with TTIP. All solutions were then leaved to stir for about 2 hours further at room temperature and aged for 24 hours before using it.

All samples were annealed for 2 hours in a box furnace operating at 650oC in an ambient atmosphere. Mid-IR spectra were obtained from the prepared samples using Fourier transform infrared spectroscopy FTIR type Shimadzu FTIR-8400S, on KBr pellets of the samples.

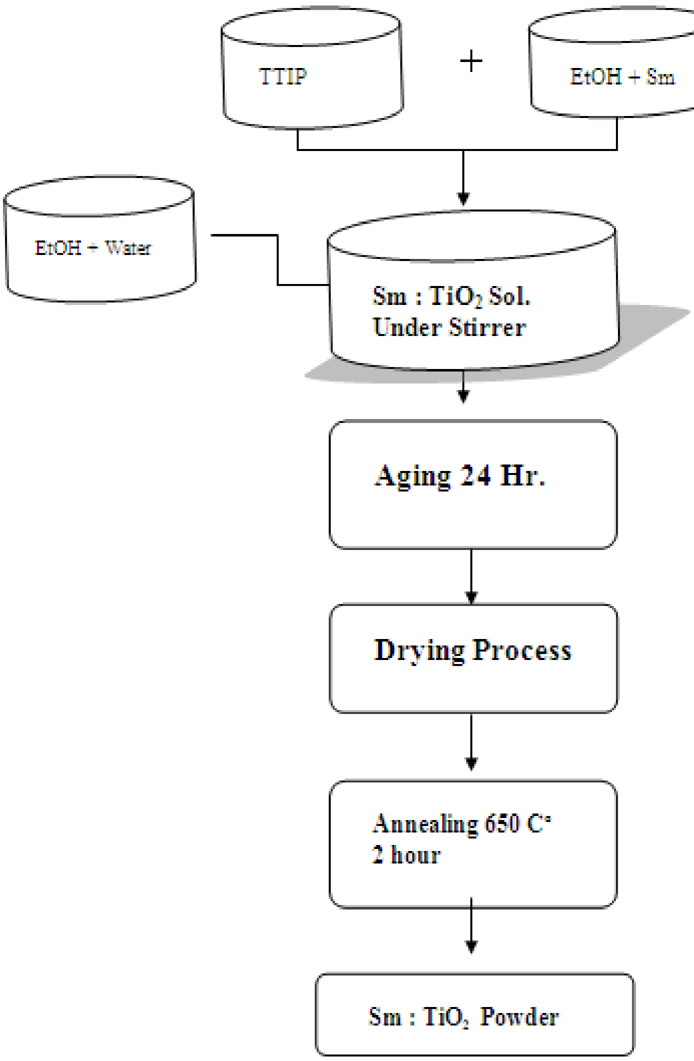

Figure (1): Scheme of the prepared Sm:TiO2 Samples by sol-gel method.

Result and discussion

The FTIR spectra of the samples before and after annealing process are illustratiod in Figures (2) and 3) 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 [8-9]. 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 [10,11], and indicating that the drying process does not completely trap the water molecules from the pores of Titania network. Due the annealing process at 650oC, the water molecules are almost completely evaporatiod from the samples which lead to disappear of the last two absorption bands from FTIR spectra, (Figure 3).

The FTIR spectra for samples after annealing process, in range of 4000 cm-1 to 400 cm-1, gives an important result which concern on observing a single transition peak wavelength of high transition ratio centered close to 1109cm-1 (~9.017µm) and 1116cm-1(~8.964µm) for pure and doped samples respectively. The first observation is related to the effect of Sm3+doping ratio on the bandwidth of this peak. This result is in good agreed with the other researches results [3, 4]. By Comparing with wavelength the high transition ratio of pure TiO2 sample, it could noted that a slightly blue shift occurs with increment of Sm+3 doping concentration ratio. Thus, it could clearly indicate that the wavelength of high ratio transition peak depends on doping ratio of Samarium ions.

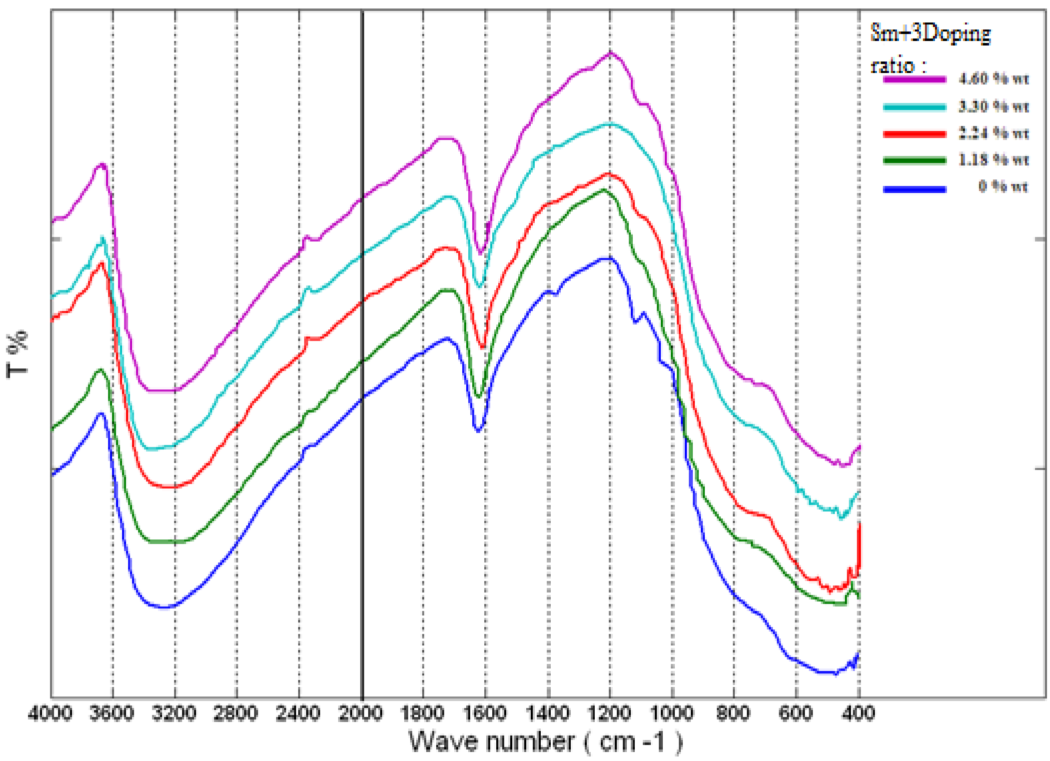

Figure 2: FTIR spectra for Sm:TiO2 samples with different doping ratio before annealing process.

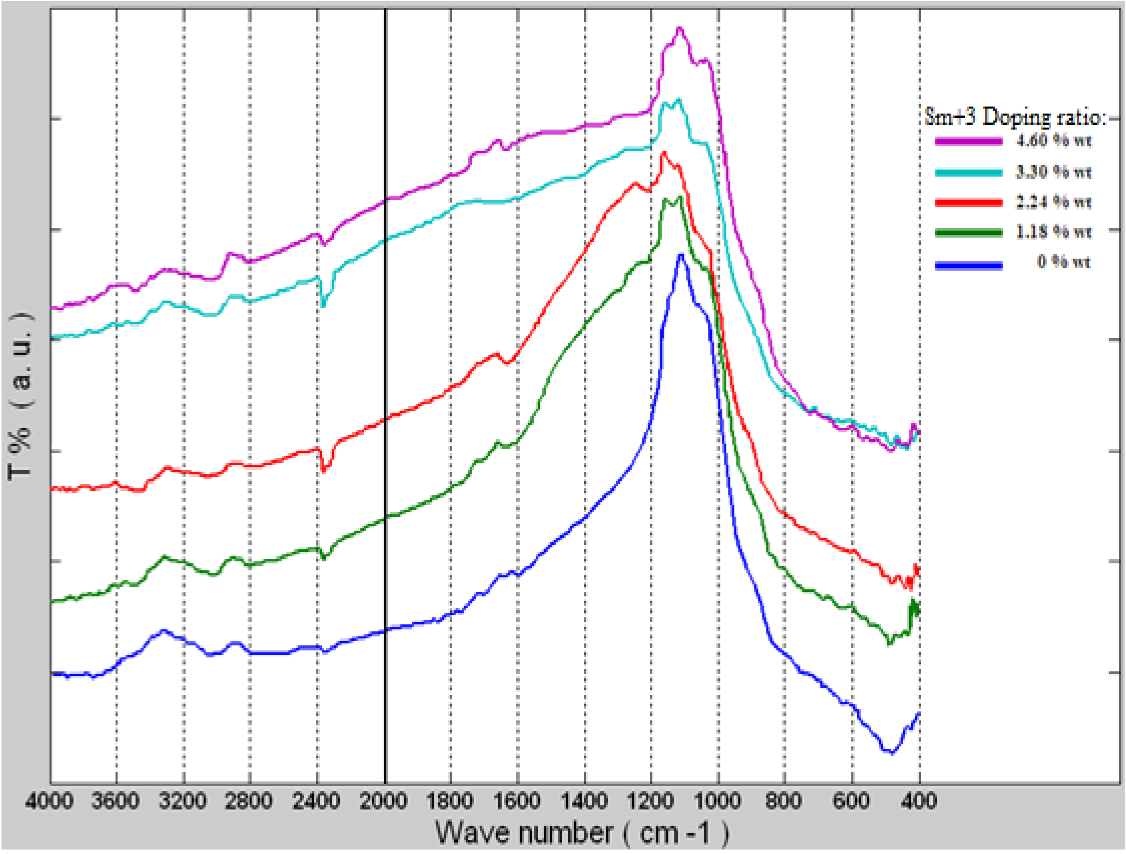

Figure 3: FTIR spectra of Sm:TiO2 samples with different doping ratio after annealing process.

The effect of doping ratio of amplitude, FWHM of the bandwidth and wavelength of high ratio transition peak is listed in Table 1. Table 1 shows that the two parameter peak wavelength and FWHM are varying with increasing of doping ratio, that means those two parameters affected by the amount of doping ratio [3, 4]. This result gives good indication about using Sm:TiO2 to prepare of optical band-pass filter with wavelength around 8.960µm (~1116 cm-1).

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

Sm3+ Doping Ratio, % ppm	Wave Number(cm-1)	Wave Length (µm)	Bandwidth range From → to (cm-1)	Bandwidth range From → to (µm)	Bandwidth (µm)
0 %	1109	9.017	1462 □ 947.7	6.839 □ 10.551	3.711
1.18 %	1116	8.960	1629 □ 948.5	6.138 □ 10.542	4.404
2.24 %	1119	8.936	1844 □ 952.7	5.422 □ 10.496	5.073
3.30 %	1113	8.984	2832 □ 922.8	3.531 □ 10.836	7.305
4.60 %	1113	8.984	2964 □ 905.6	3.373 □ 11.042	7.668

Conclusion

The FTIR analysis of all doped samples after annealing showed a wide single peak sample, the position of the maximum transmission of these peaks have a blue shift when the TiO2 samples doped with low ratio of Samarium ions with no further response at the higher doping ratio with Sm3+ ions. Thus, it could strongly advice to synthesis of optical band-pass filter at Mid-IR optical filter (wavelength around 8.960µm ~1116 cm-1) by using Sm:TiO2 nanoparticles (1.18% ppm doping ratio) prepared by the sol gel method.

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