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ORIGINAL ARTICLE

Hydrothermal Synthesis of Tm^{+3} Doped Sb_6O_{13} Nanocrystals

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ABSTRACT

A simple hydrothermal method has been developed for synthesizing of $Tm_xSb_{6-x}O_{13}$ nanocrystals at a high yield at 180 ℃ that is a new method for preparing these nanocrystals. Antimony oxide often promotes the activating catalysts used for selective partial oxidations and for related reactions such as oxidative coupling. Antimony oxide can be used as a catalyst, retardant, dielectric, fining agent and optical material. XRD patterns indicate that the $Tm_xSb_{6-x}O_{13}$ nanocrystals with 0-2 % mol are isostructural with Sb_6O_{13}. SEM shows the size of particle changes with doping Tm^{+3} ions into Antimony sites and the size of particles decrease. Floresance specta also shows this result. FT-IR, XRD, and SEM ,TEM were used to analysis the structure characteristics of $Tm_xSb_{6-x}O_{13}$ nanocrystals.
Keywords: Antimony Oxide, Hydrothermal, Thulium, Optical Properties.

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INTRODUCTION

Nanocrystalline materials have attracted considerable attention due to their distinguished electrical, optical, optoelectronical, electronic and magnetic properties [1]. Investigations on semiconductor nanostructures have recently been in the focus of intensive research activities because of intrinsic fundamental interest and manifold possibilities for applications. Antimony oxide with defect pyrochlore structure (Sb_6O_{13}) crystallize in the cubic system with space group *Fd3m* The structure of sb_6o_{13} ($Sb_2{}^{+3}O_3$ .2 $Sb_2{}^{+3}O_5$) can be regarded as the two cationic sub lattices belonging to the first two compounds, brought together to form one anionic framework ,with random distribution of the eight Sb^{+3} captions in the (32e) position. The formula of the oxide Sb_6O_{13} ($Sb_2{}^{+3}Sb_4{}^{+5}O_{13}$) can be represented in the form $Sb^{+3}Sb_2{}^{+5}O_6O_{1.5}$, in accordance with the pyrochlore structure AB_2O_6X [2-3].

. Antimony oxide often promotes the activating catalysts used for selective partial oxidations and for related reactions such as oxidative coupling. Antimony oxide can be used as a catalyst, retardant, fining agent and optical material. Recently antimony oxide was found to have high proton conductivity, which is likely to be a promising humidity –sensing material [4-7]. On the other hand, during the past few years the synthesis process and spectroscopic properties of lanthanide- doped nanoparticles have attracted considerable attention, since they are considered as potentially useful active component in lamps and displays, lasers, and new optoelectronic devices [8].

EXPERIMENTAL

Synthesis of $Tm_xSb_{6-x}O_{13}$ nanocrystals

Nanocrystalline $Tm_xSb_{6-x}O_{13}$ samples were prepared by a hydrothermal method.Sb_2O_3, $Ce(NO_3)_2$, Tm_2O_3, were used as starting material. In a typical procedure,(Sb_2O_3) and $Ce(NO_3)_3.6H_2O$ and Tm_2O_3 were dissolved into 10 ml of distilled water at room temperature in beaker and magnetically stirred to obtaining homogeneous solution. The as-obtained product was transferred into a Teflon lined stainless autoclave. The autoclave was sealed and maintained at 180°C for 48h, allowed to cool naturally to room temperature. A white precipitate was collected. The product was washed and filtered several times with distilled water, and dried in room temperature.

Characterization

Powder **X**-ray diffraction (P-XRD) was performed with a Siemens D5000 X-ray diffractometer with Cu-K_α radiation (λ = 1.541 A°) and 2θ was varied 4-70˚.

The Fourier Transformed Infrared (FTIR) transmissions in the region 400-4000 cm^{-1} were recorded using the FTIR Nexus 670 by KBr pellet technique.
The morphology of the particles was examined by a scanning electron microscope (SEM, philips XLC).

RESULT AND DISCUSSION

Figure.1 shows powder X-ray diffraction patterns of $Tm_xSb_{6-x}O_{13}$ nanocrystals with x=0.00, 0.01, 0.015, 0.02. The positions of the peaks are in accordance with JCPDS card (33-0111) for crystalline Sb_6O_{13}. This indicates that the $Tm_xSb_{6-x}O_{13}$ nanocrystals with x=0-0.02 are isostructural with crystalline Sb_6O_{13}.

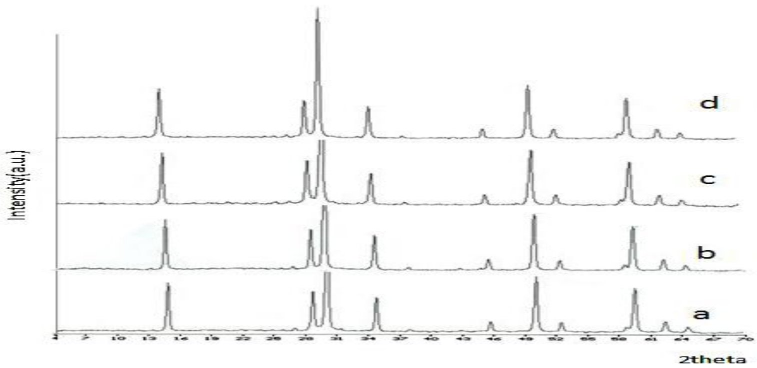

Fig1. XRD patterns of $Tm_xSb_{6-x}O_{13}$;(a: x=0.00, b: x=0.01, c: x=0.015, d: x=0.02)

SEM images of Sb_6O_{13} and $Tm_xSb_{6-x}O_{13}$ nano crystals have been shown in figure 2a and 2b. The size of particle changes with doping Tm^{+3} ions into Antimony sites and the size of particles decrease. The size of particles for Sb_6O_{13} nanocrystals is 60-80nm, but for $Tm_xSb_{6-x}O_{13}$ nano crystals the size of particle is 40-60nm.

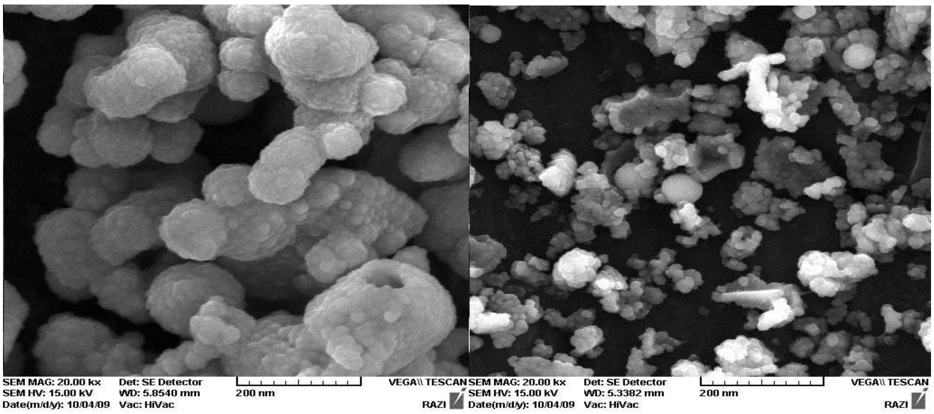

Fig.2 SEM images a) Sb_6O_{13} nanocrystals b) $Tm_xSb_{6-x}O_{13}$ nano crystals

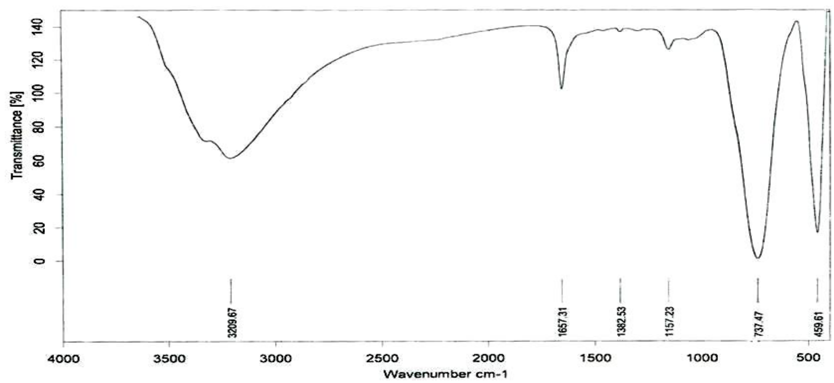

Fig 3 Shows IR spectrum of the as obtained sample at optimized condition. Strong adsorption peaks at459.61,737.47cm $^{-1}$ are correspond to the vibration of Sb -O bond in the Sb_6O_{13} lattice [14]. The bond at 1657cm^{-1} corresponds to the deformation vibration of water molecule. it is significant that there is a small signature at 1382cm^{-1} indicative of the presence of nitrates. Adsorption peaks at463.57cm $^{-1}$ are correspond to the vibration of Tm-O bond in the Tm_2O_3 lattice.

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