

Synthesis of SnO₂ and C,N,S-tridoped SnO₂ nanoparticles by Precipitation method

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ABSTRACT

Metal oxides play a very important role in many areas of chemistry, physics and materials science. The metal elements are able to form a large diversity of oxide compounds. In this investigation, we synthesis SnO₂ and C,N,S-tridoped SnO₂ nanoparticles by precipitation method. The synthesized SnO₂ and C,N,S-tridoped SnO₂ nanoparticles were characterized by instrumental analysis methods, such as X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) techniques. The observed results showed particle size 30 nm.

Keywords: SnO₂ nanoparticles; Synthesis; Characterization; Doping

INTRODUCTION

Nanometer range semiconducting materials have been a subject of intense study for last several years due to their size dependent physical and chemical properties below a critical size characteristic of the material. In recent years, the interest in the physical properties of semiconducting metal oxide, such as SnO₂, TiO₂ and ZnO, has significantly increased due to their potential applications, in special when they are intentionally doped with magnetic elements [1,2].

Wide energy-band-gap semiconductors have attracted a great deal of interest

because of their potential applications in next-generation electronic and optoelectronic devices, such as field-effect transistors, organic bistable memory devices (OBDs), light-emitting diodes (LEDs), and solar cells [3–7]. Among the various kinds of wide energy-band-gap semiconductor nanoparticles, SnO₂ semiconductors have been particularly interesting due to their excellent optical and electrical properties, as well as their high chemical stability [8–10]. SnO₂ nanoparticles have been formed using several methods of sol-gel process, spray pyrolysis, pulsed laser ablation, chemical

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vapor deposition, and thermal evaporation [11–15]. In this work, we synthesized SnO₂ and C,N,S-tridoped SnO₂ nanoparticles by chemical precipitation method because this method has some advantages such as precise control over the stoichiometry, low temperature synthesis, high purity and high chemical homogeneity.

EXPERIMENTAL METHODS

Materials

The chemical reagents used included Tin (IV) chloride pentahydrate (Sigma), Thiourea (Aldrich), ammonia solution (Merck) and distilled water. All were used without further purification.

Preparation of SnO₂ and C,N,S-tridoped SnO₂ nanoparticles

N,S,C-SnO₂ powders were prepared using a precipitation method. The thiourea was mixed with Tin (IV) chloride pentahydrate in 500 ml distilled water to form a 0.25 M aqueous solution. Then, the solution was stirred for 3 h. Following complete dissolution, the solution was treated with an ammonia solution (25%) until the solution pH reached to 7. The resulting solution was stirred for another 2 h. Precipitated products were separated from the solution by centrifugation, and dried at 100°C for 24 h. The obtained products were ground and calcined at an elevated temperature for 3 h with a heating rate of 5°C/min. The SnO₂ nanoparticles has synthesized by this method without thiourea solution.

Characterization

The X-ray diffraction (XRD) patterns obtained on a X-ray diffractometer (type HZG41B-PC) using CuK α irradiation. The surface morphology was examined using scanning electron microscopy (SEM) (JSM 6701F—6701). The particle size of the nano powders was measured using

Transmission Electron Microscope (TEM) (Zeiss EM-900).

RESULT AND DISCUSSIONS

X-ray diffraction (XRD)

The XRD patterns of SnO₂ and C,N,S-tridoped SnO₂ nanoparticles are shown in Fig. 1. All diffraction peaks are well assigned to tetragonal crystalline phase of SnO₂ (with the reference pattern; ICDD card No. 41-1445) and the Ni doping does not change the tetragonal structure of SnO₂. From Figure 1, it is noted that the intensity of the C,N,S-tridoped SnO₂ peaks decreases with increasing C, N, S- content and the full-width at half-maximum (FWHM) of the peaks increases with increasing C, N, S- content. The average particle size of samples are calculated using Scherrer's formula,

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (1)$$

where (λ) is the wavelength of CuK α radiation (= 0.154056 nm), β is the full width at half maximum (FWHM) of the (*hkl*) peak at the diffracting angle 2θ [12], all the peaks are used to calculate the average particle size of samples.

The average particles size of samples are found to be 32 and 29 nm for SnO₂ and C,N,S-tridoped SnO₂ nanoparticles using scherrer's formula, respectively.

Fourier transform infrared spectroscopy (FTIR)

The FTIR transmittance spectra of different samples are given in Fig. 2, in which the band located at 3180 cm⁻¹ is owing to the vibration of O-H and the band located at 1636 cm⁻¹ is due to the H-O-H vibrating mode of the absorbed water, while the bands at around 578 and 1630 cm⁻¹ can be attributed to the Sn-O stretching vibration and the O-Sn-O bending vibration in SnO₂.

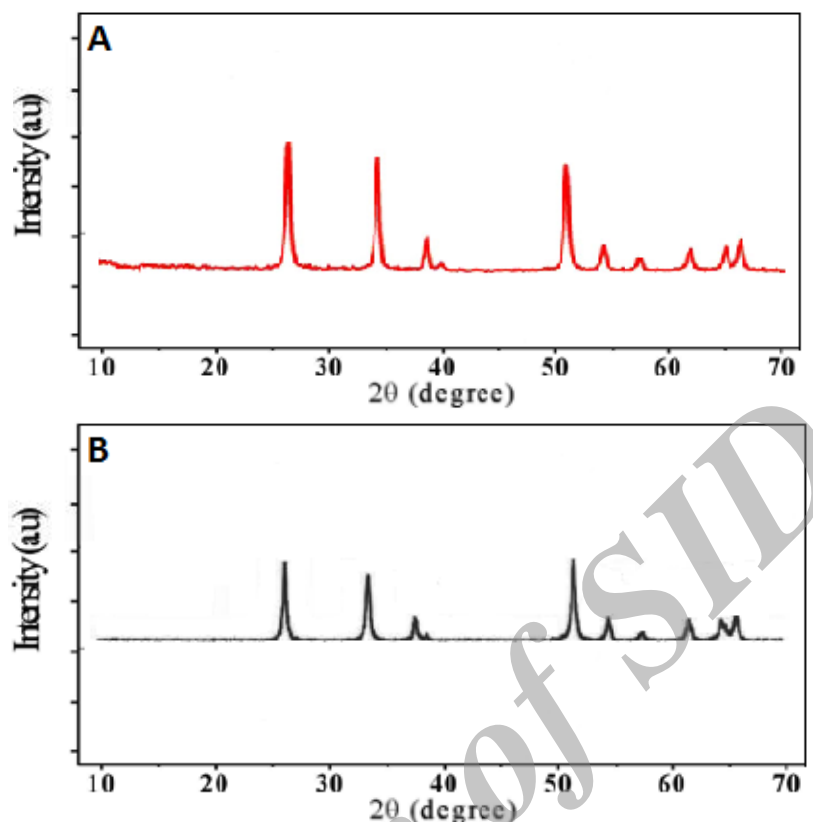


Fig. 1. XRD patterns of SnO₂ (A) and C,N,S-tridoped SnO₂ (B) nanoparticles.

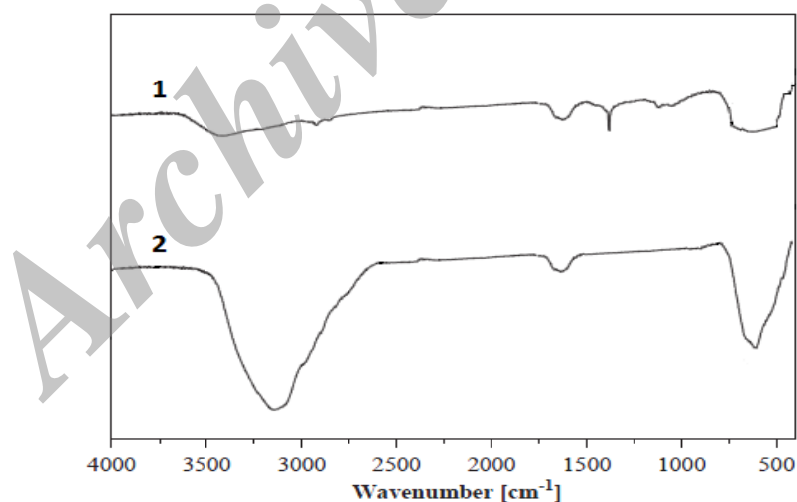


Fig. 2. FTIR spectra of SnO₂ (1) and C,N,S-tridoped SnO₂ (2) nanoparticles.

Scanning electron microscopy (SEM)

Figs. 3A and 3B show the SEM images of the as synthesized SnO₂ and C,N,S-tridoped SnO₂ nanoparticles, respectively, indicating products consist of nanoparticles structures. Statistical analysis of different SEM images showed that the average

diameter of these nanoparticles was in the range of below than 50 nm. The SEM images present the morphological evolution where Fig. 3 shows the as synthesized nanoparticles have aggregation to form spherical particles.

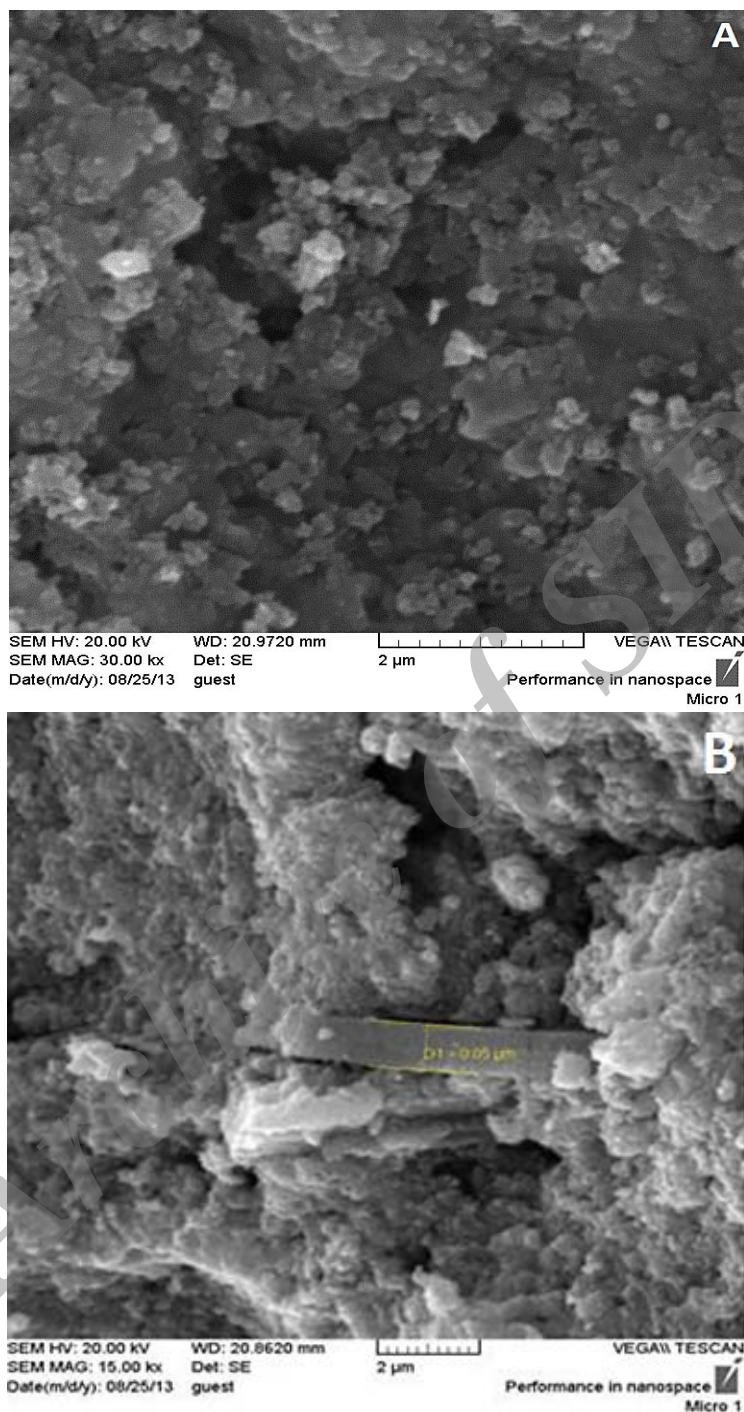


Fig. 3. SEM image of SnO₂ (A) and C,N,S-tridoped SnO₂ (B) nanoparticles.

Transmission Electron Microscope (TEM)

TEM micrograph of SnO₂ and C,N,S-tridoped SnO₂ shown in Fig. 4 reveals the morphology and particles size.

The monodispersed particles with spherical shape and size <50 nm with an

equable distribution, except for a little aggregated particulate, is observed in TEM image. On the other hand, the TEM photographs show that nano powder is nanometer scale, which is follow with the results obtained from the XRD patterns.

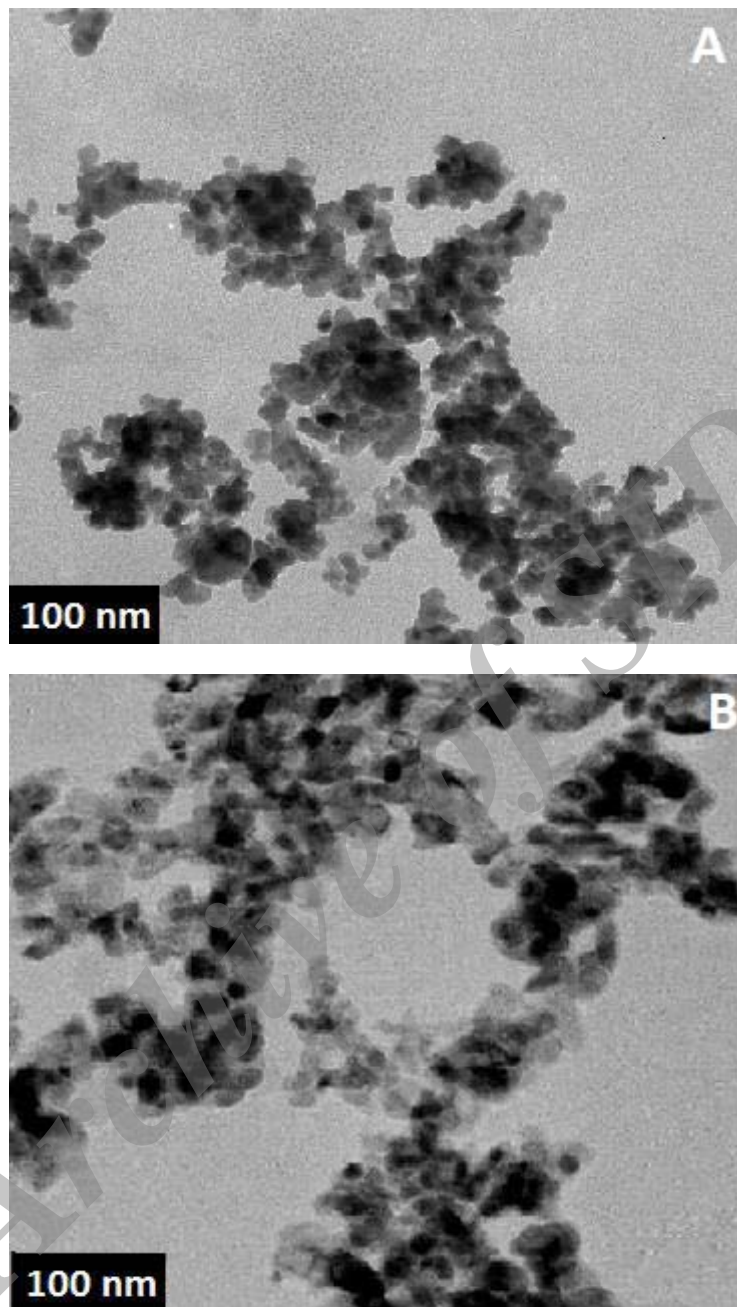


Fig. 4. TEM image of SnO₂ (A) and C,N,S-tridoped SnO₂ (B) nanoparticles.

CONCLUSIONS

C,N,S-tridoped SnO₂ nanoparticles powders were synthesized by the precipitation method from thiourea and tin (IV) chloride pentahydrate. XRD results show that the average particles size is in the range from 29 to 32 nm. It is shown that the average particles size of the

nanoparticles decreases with increasing amounts of Doping C,N and S. TEM images of SnO₂ and C,N,S-tridoped SnO₂ powders shows that the particle agglomerated with an average diameter <50 nm, confirming the reduction in particles size as a result of C,N,S-doping in

SnO₂. Other techniques, including SEM and FTIR, were employed to characterize all of the synthesized materials.

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