Simple size-controlled fabrication of Zn_2SnO_4 nanostructures and study of their behavior in Dye sensitized solar cells

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ABSTRACT

Zn_2SnO_4 nanostructures were obtained via facile and rapid co-precipitation approach in presence of amines with different long chain as a novel basic and capping agents. The effect of different amines such as NH_3 , ethylenediamine, triethylenetetramine, tetraethylenepentamine on the size of Zn_2SnO_4 nanostructures were investigated. The results demonstrated that applying the appropriate amount of organic amine could be effective in particle size control. The obtained nanostructure products were specified by powder X-ray diffraction (XRD), Fourier transform infrared (FT-IR) spectra, scanning electron microscopy (SEM), transmission electron microscopy (TEM), energy dispersive X-ray analysis (EDX) and ultraviolet-visible (UV-vis) spectroscopy. Dye sensitized solar cells (DSSC) were created by the constructed electrodes as working electrode and then were studied by current density-voltage (J-V) curve. It was found that incorporating of TiO_2 nanoparticles to optimized Zn_2SnO_4 nanostructures has significant role on the constructed DSSCs photovoltaic properties.

Keywords: Nanostructures; Semiconductor; Solar cell; Electron microscopy; Size controller

1. Introduction

Zinc stannate (Zn_2SnO_4) as a ternary semiconductor is well-known for various applications such as dye-sensitized solar cells [1], photocatalysis [2, 3], lithium ion batteries [4] and gas sensing systems [5]. Zn_2SnO_4 has

a broad band gap of 3.6 eV with great optical **property**, good chemical sensitivity and low visible absorption. Recently, photovoltaic usage for dye sensitized solar cells has become the focus of significant research attempts [6,7]. It has been specified that the energy conversion performances of the photovoltaic sets that benefit nanocrystalline Zn_2SnO_4 are critically dependent on the size and morphology of the materials [1, 8]. **There are different preparation methods and factors for synthesis of various types of nanostructures with great properties [9-11].** Different synthetic parameters such as using capping agents and surfactants [12, 13], reaction temperature, **reaction** time [14] and pH [15, 16] can affect Zn_2SnO_4 morphology and particle size. A wide variety of routes have been proposed for the synthesis of nanocrystalline zinc stannate nanostructures including hydrothermal [17], co-precipitation [18], microwave-assisted hydrothermal [2], solid state calcinations [19] and thermal evaporation [20]. Among of these methods, co-precipitation **approach** is facile, rapid, cost effective method, potential for large-scale production and simply control the morphology and particle size for synthesis of nanostructures [21]. For size and morphology detection, many attempts have been carried out. Beside the common surfactants, recently our group **have** focused on the result of different amines as a capping agent on the final properties of obtained products [22–24]. The purpose of this study is investigating the role of different amines on the size, morphology and uniformity of the Zn_2SnO_4 nanocrystals and the efficiency of corresponding dye-sensitized solar cells. The selected amines are chosen in a way that we consider the effect of various amines with different nitrogen active sites. The long carbon chain of amines can supply excellent steric hindrance to control the size of nanoparticles [25, 26]. In this paper, Zn_2SnO_4 nanoparticles were synthesized using four different amines of NH_3 (monodentate amine), ethylenediamine (bidentate amine), triethylenetetramine (tridentate amine) and tetraethylenepentamine (**tetradentate amine**) **for shape and size controlling via** a co-precipitation method. The results demonstrated that applying the appropriate amount of amines could be effective in particle size control. Finally, the dye-sensitized solar cells **are** performed to study effect of incorporating of

titanium dioxide **nanoparticles** with different ratios on the photovoltaic properties of as-produced optimized Zn_2SnO_4 nanostructures.

2. Experimental

2.1. Materials and physical measurements:

All the chemical reagents for the synthesis of zinc stannate nanostructures such as $\text{Zn}(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$, $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$, NH_3 , ethylenediamine (en), triethylenetetramine (TETA), tetraethylenepentamine (TEPA) were commercially available and employed without further refinement. Fourier transform infrared (FT-IR) spectrum was recorded on Nicolet Magna-550 spectrometer in KBr pellets. X-ray diffraction (XRD) patterns were detected by a Philips-X'pertpro, X-ray diffractometer using Ni-filtered $\text{Cu K}\alpha$ radiation. GC-2550TG (Teif Gostar Faraz Company, Iran) were used for all chemical analyses. Scanning electron microscopy (SEM) image was prepared on LEO-1455VP equipped with an energy dispersive X-ray spectroscopy. The EDX analysis with 20 kV accelerated voltage was carried out. Transmission electron microscopy (TEM) image was created on a Philips EM208 transmission electron microscope with an accelerating voltage of 200 kV. Optical analysis was performed using a V-670 UV-Vis-NIR spectrophotometer (Jasco).

2.2. Synthesis of Zn_2SnO_4 nanostructures:

Zn_2SnO_4 nanostructures were prepared **via** co-precipitation method. First, $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ solution was added dropwise into the zinc nitrate solution (with molar ratio of $\text{Zn}:\text{Sn} = 2:1$) under magnetic. Then, amine as a novel basic and capping agent was added dropwise into metal solutions. The pH solution was adjusted to 7-8 to preserve the hydroxide phases of Zn and Sn. The obtained precipitate was collected by centrifugation at 5000 rpm for 4 min, washed repeatedly with distilled water. The as-prepared products were dried at 80°C under vacuum for 4 h and then were calcined at 1000°C for 3 h. The preparation conditions of Zn_2SnO_4 nanostructures have been illustrated in [Table 1](#). To study the effect of capping agent on the morphology and size of the zinc stannate products, an experiment as blank **test was** carried out without capping agent at room temperature.

2.3. Fabrication of DSSC

According to the procedure reported by Grätzel [27], the Zn_2SnO_4 and TiO_2 pastes were prepared. The slurry was produced by mixing and grinding 6.0 g of the nanometer sized powder with ethanol and water in several steps. The grinded slurry was merged with ultrasonic horn (Sonicator 3000; Bandeline, MS 72, Germany) and then was mixed with terpineol and ethyl cellulose as binders. After eliminating the ethanol and water with a rotary-evaporator, the final paste was prepared. Firstly, the TiO_2 paste was deposited on the clean substrates of fluorinated tin oxide glass (FTO glass, Pilkington Glass, TEC7) by a doctor blade technique. The Zn_2SnO_4 paste was deposited on the TiO_2 by a doctor blade technique. The active area of the electrodes was 0.25 cm^2 . After natural drying at room temperature, the electrodes coated with the TiO_2 and Zn_2SnO_4 pastes were slowly heated under an air flow at 325°C for 5 min, at 375°C for 5 min, at 450°C for 15 min and 500°C for 15 min. At 80°C in the cooling, the prepared electrodes were immersed into a 0.5 mM cis-di(thiocyanato)-N,N-bis(2,2-bipyridyl-4-carboxylicacid-4-tetrabutylammonium carboxylate)ruthenium(II) (N-719) dye solution in ethanol and kept at room temperature for 24 h to complete the sensitizer uptake. The dye-adsorbed electrodes were then rinsed with ethanol. A Pt coated FTO glass electrode was prepared as a counter electrode. The Pt electrode was located over the dye-adsorbed electrode and the edges of the cell were sealed with $50 \mu\text{m}$ thick sealing sheet (Surlyn). Sealing was accomplished by pressing the two electrodes together on a double hot-plate at a temperature of about 110°C . The electrolyte material was prepared according to the literature [27]: a solution 0.6 M BMII, 0.03 M I_2 , 0.1 M guanidinium thiocyanate and 0.50 M 4-tert-butylpyridine. The electrolyte was introduced into the cell via two small holes drilled in the counter electrode. Finally, these two holes were sealed by a small square of sealing sheet.

2.4. PV measurements

Photocurrent-voltage (I–V) curve was evaluated by using computerized digital multi meters (Ivium-n-Stat Multi channel potentiostat) and an unstable load. A 300 W metal xenon lamp (Luzchem) was served as a simulated sun

light source, and its light intensity (or radiant power) was controlled to simulated AM 1.5 radiation at 100 mW/cm² with a filters for this purpose.

3. Results and discussion

3.1. X-ray diffraction pattern

For confirming the crystal structure and purity of the zinc stannate product, XRD analysis was carried out. XRD pattern of the product synthesized in presence of NH₃ as a basic agent and capping agent after calcination at 1000 °C is shown in Fig. 1. The diffraction peaks observed in Fig. 1 can be indexed to pure cubic phase Zinc Tin Oxide (JCPDS card no. 24-1470) with space group of Fd-3m and cell constants $a = b = c = 8.6574 \text{ \AA}$. The 2θ value of peaks in the XRD pattern of Zn₂SnO₄ nanostructure clearly brought out. the crystallite diameter (D_c) of Zn₂SnO₄ nanostructure obtained using the Scherrer equation [28]: $D = K\lambda/\beta\cos\theta$; where β is the width of the observed diffraction line at its half intensity maximum, K is the so-called shape factor, which usually takes a value of about 0.9, and λ is the wavelength of X-ray source used in XRD. Computed crystalline domain size has been detected to be 31 nm.

3.2. SEM and TEM images

The result of using different amines as size controller on size, morphology and uniformity of the obtained Zn₂SnO₄ products is shown in Fig. 2. Fig. 2a-d show prepared Zinc Tin Oxide nanostructures by NH₃, en, TETA and TEPA (sample No.1-4) as novel basic and capping agents, respectively. The used different amines for preparation of product are indicated in Scheme 1. The average particle sizes of Zn₂SnO₄ obtained from SEM images are listed in Table.1. As shown in Fig 2a-d, the morphology of the synthesized Zn₂SnO₄ samples by all of the amines is spherical nanoparticles. Although morphologies of the samples No.1-4 are the same, the size and uniformity of nanoparticles are different. These results show that using NH₃ basic agent lead to synthesis of agglomerated product. By increasing steric hindrance of amines, a decrease in particle size occurs. The Zn₂SnO₄ product synthesized by TEPA amine is optimum product because nanoparticles have smaller size and more

uniformity. The Zn_2SnO_4 surfaces are occupied by hydroxy groups. The nitrogen atoms in the NH_2 groups of amine will form hydrogen bonds with hydroxyls of Zn_2SnO_4 surface. As the amine molecule has a long chain with several amine groups and zinc stannate has many hydroxy groups on the surface, amine will entwine the zinc stannate surface tightly as shown in Scheme 1. The probable mechanism of different amines role as basic and capping agents on the properties of products are shown in Scheme 1.

The exact morphology and size of Zn_2SnO_4 nanostructures prepared in presence of the TEPA amine was illustrated by TEM and HRTEM images. Fig. 3a, b show the TEM images of sample No.4 which contains homogenous ellipsoidal and spherical nanoparticles with the size of about 10-15 nm. Fig. 3c shows representative HRTEM image of the specimen. In the HRTEM image, the distance between the two neighbor planes, d , is measured to be 0.29 nm. The opaque parts in HRTEM image show that probably a small amount of TEPA amine is attached to the surface of nanoparticles which may have an advantageous role in inhibiting nanoparticles agglomeration.

3.3. FT-IR and EDX analysis

To determine the chemical interactions and bonding structures, Fig.4 shows the FT-IR spectrum of the Zn_2SnO_4 nanoparticle (sample No. 4). The absorption from 3451 cm^{-1} can be assigned to the stretching vibration of the hydrogen bonded OH groups of the adsorbed water. The absorption around 1620 cm^{-1} is due to the bending vibration of water molecules. The bands at around 2920 and 2850 cm^{-1} are dedicated to the antisymmetric and symmetric C-H stretching vibrations of TEPA amine, respectively. The broad band 574 cm^{-1} in the FT-IR spectrum belongs to the characteristic vibrations of the inorganic SnO_4 groups. The band at around 421 cm^{-1} can be attributed to the vibrations of the ZnO_4 groups, respectively. The FT-IR result confirms existence of TEPA amine for preventing nanoparticles agglomeration which is already evidenced by HRTEM image.

The purity of the nanostructures was also indicated by energy-dispersive X-ray spectroscopy. EDX analysis of Zn_2SnO_4 nanoparticle (sample No. 4) is showed in Fig. 5. The lines of Zn, Sn and O are obviously observed.

3.4. Optical *property*

The UV-Vis Diffuse Reflectance spectroscopy of Zn₂SnO₄ *nanoparticle* (sample No. 4) is shown in Fig. 6. The fundamental absorption edge in most nanostructure semiconductors follows the exponential law. Using the absorption data, the band gap was measured by Tauc's equation:

$$\alpha = \frac{\alpha_0(h\nu - E_g)^n}{h\nu}$$

where α is absorption coefficient, $h\nu$ is the photon energy, α_0 and h are the constants, E_g is the calculated optical band gap of the nanostructure material, and n depends on the type of electronic transition and can be any value between $\frac{1}{2}$ and 3 [29]. The energy gap of the sample has been specified by extrapolating the linear portion of the plots of $(\alpha h\nu)^2$ against $h\nu$ to the energy axis. The E_g *value* is determined 3.6 eV for the *sample* No. 4.

3.5. DSSCs *performance*

The typical photocurrent density (J) – voltage (V) curves of the DSSCs based on N719 sensitized Zn₂SnO₄ and TiO₂ nanoparticles photoelectrodes are shown in Fig.7 and the photovoltaic parameters derived from the J–V curves are summarized in Table. 2. It is observed that the J_{sc}, V_{oc} and η for the Zn₂SnO₄: TiO₂ = 1: 1 photoelectrode are 6.43 mAcm⁻², 0.79 V and 3.64%, respectively, which are much higher than other photoelectrode. As Zn₂SnO₄ / TiO₂ photoelectrode has a good *result*, we are very interested in how it works. The possible mechanism is shown as follows. According to previous works [1], the conductive bond potential of the TiO₂ coating is ca. 0.09 eV more negatively than the Zn₂SnO₄ nanostructures, such a band-structure-matched Zn₂SnO₄ –TiO₂ hybrid photoelectrode can be imaged as the “bridge” for the electron transport from Zn₂SnO₄ to FTO electrode, which can inhibit the recombination reaction between the electron in the CB of Zn₂SnO₄ –TiO₂ and I₃⁻ in the electrolyte resulting in longer electron lifetime. To study the effect of incorporating of TiO₂ on the photovoltaic parameters, DSSCs. 4 and 5 were tested *as reference tests*.

Zn₂SnO₄ nanostructures were prepared by different methods with various size and morphology, already. For the first time, in this paper zinc stannate nanoparticles with very fine size and high uniformity were synthesized via simple and rapid co-

precipitation method via different amines as capping and basic agents without using any surfactants. In comparison to other similar works, shown in Table 3, this co-precipitation method is more promising in terms of low cost, simply control the particle size, short reaction time, simplicity and potential for large scale production. In Table 3, photovoltaic properties of ZTO samples were compared.

4. Conclusions

In summary, Zn_2SnO_4 semiconductor nanoparticles with different sizes were prepared using the simple and rapid co-precipitation method and their performances as the photoelectrode material of dye-sensitized solar cells were investigated. Four different amines with various steric hindrance as novel basic and capping agents were examined to investigate the growth control. The selected amines including short-chain and long-chain were novel in this approach. Here, it was shown that using a long chained amine as a capping agent and size controller is beneficial in producing the particles with smaller size. The results contribute to understanding the significant effect of the chemical structure of the capping agents on the size and agglomeration control of the Zn_2SnO_4 products as well as their photovoltaic properties. It was found that incorporating of TiO_2 nanoparticles to optimized Zn_2SnO_4 nanostructures have significant role on the photovoltaic properties.

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Figure captions:

Fig. 1. XRD pattern of the Zn_2SnO_4 nanostructure product synthesized by NH_3 basic agent after calcination at 1000°C .

Fig. 2. SEM images of Zn_2SnO_4 nanostructures prepared by different amines a) NH_3 , b) en, c) TETA and d) TEPA.

Fig. 3. (a and b) TEM image at different scales and (c) HRTEM of Zn_2SnO_4 nanostructures (sample No. 4).

Fig. 4. FT-IR spectrum of Zn_2SnO_4 nanoparticles (sample No. 4).

Fig. 5. EDS pattern of the Zn_2SnO_4 nanoparticles (sample No. 4).

Fig. 6. UV–Vis diffuse absorption spectrum and linear portion of the plots of $(ah\nu)^2$ against $h\nu$ of sample No. 4.

Fig. 7. I–V characteristics of the DSSC for different samples.

Scheme. 1. Schematic illustration for the growth mechanism of Zn_2SnO_4 nanoparticles in presence of different amines.

Scheme. 2. Schematic diagram of the band structure in $\text{Zn}_2\text{SnO}_4 - \text{TiO}_2$ nanostructures.

Table. 1. Reaction conditions for Zn_2SnO_4 nanostructures.

Table. 2. Important cell parameters collected from J–V curves of prepared samples.

Table. 3. Characterization comparison of zinc stannate nanostructures with other similar works.

Table 1. Reaction conditions for Zn₂SnO₄ nanostructures.

Sample No.	Type of amine	Source of Zn	solvent	Time (h)	Temperature (°C)	Morphology	Particle Size (nm)
1	NH3	Zn(NO ₃) ₂ .4H ₂ O	H ₂ O	1	25	nanoparticle	50-350
2	en	Zn(NO ₃) ₂ .4H ₂ O	H ₂ O	1	25	nanoparticle	25-200
3	TETA	Zn(NO ₃) ₂ .4H ₂ O	H ₂ O	1	25	nanoparticle	25-180
4	TEPA	Zn(NO ₃) ₂ .4H ₂ O	H ₂ O	1	25	nanoparticle	20-150

Table. 2. Important cell parameters collected from J–V curves of prepared samples.

No	DSSCs	J_{sc} (mA cm ⁻²)	V_{oc} (V)	FF	η (%)
1	Zn ₂ SnO ₄ : TiO ₂ = 0.5 : 1	5.45	0.82	0.68	3.05
2	Zn ₂ SnO ₄ : TiO ₂ = 1 : 1	6.43	0.79	0.71	3.64
3	Zn ₂ SnO ₄ : TiO ₂ = 1 : 0.5	3.84	0.78	0.65	1.95
4	Zn ₂ SnO ₄	3.79	0.763	0.67	1.94
5	TiO ₂	4.62	0.769	0.70	2.52

Table 3. Characterization comparison of zinc stannate nanostructures with other similar works.

Morphology	method	Efficiency of of DSSCs	SEM	Ref
Nanosheets consisting o nanoparticles	Hydrothermal	ZTO 4.82% ZTO/TiCl ₄ 5.44%		[1]
Nanoflower	Hydrothermal	3.34%		[8]
Nanoplate/ micro-octahedron	Hydrothermal	3.1%		[30]
Nanosheet-Hierarchical Microspheres	Solvothermal	3.43%		[31]
Nanocactus	Hydrothermal	ZTO 2.21% ZTO/TiO ₂ 6.62%.		[32]
Nanoparticle	Simple and rapid c precipitation	ZTO 1.94% ZTO/P25 3.64%		This paper

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