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Effects of Structure and Morphology of Hydrothermally Grown ZnO Particles on the Photovoltaic Performance of Dye Sensitized Solar Cell

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ABSTRACT

Highly ordered microporous ZnO materials with crystalline structure were synthesized hydrothermally from three different Zn-precursorsnamely: Zinc-acetate, chloride, and nitrate. XRD investigations revealed that all the obtained powders have single phase zincite structure. The powder morphology was investigated by SEM which showed that the starting Zn-precursor affects the shape as well as the size of the obtained particles. A photoelectrodes fabricated using the three different synthesized microporous ZnO particles showed variation in photocurrent density (Jsc),dye loading and hence the efficiency with variation in structure texture, morphology and particle size. DSSC built with photoelectrodesmade from ZnO particles synthesized from Zn-acetate as precursor has the highest efficiency (5.4%) which is attributed to having lowest particle size and largest surface area available for dye loading. This increases light harvesting at the photoelectrode which in turn increases short circuit current density. On the other hand, the lowest current density (5.65mA/cm^2) is obtained for DSSC with photolelctrodes made from Zn-nitrate as precursor.

Indexing terms/Keywords

ZnO, nanoparticles, hydrothermal treatment, DSSC

Academic Discipline And Sub-Disciplines

Materials Science

SUBJECT CLASSIFICATION

Physics

TYPE (METHOD/APPROACH)

Experimental Approach

1. INTRODUCTION

Finding energy sources satisfy the world's growing demand is one of the foremost research challenges. One of the most promising technologies is the dye sensitizedsolar cells (DSSCs), which have attracted much attention due to their low cost, flexibility, high efficiency [1,2].

The energy conversion process in DSSCs relies on injection of electrons from the photo-excited dye molecules into the conduction band of mesoporous semiconductor photoanode deposited on transparent conducting oxide (TCO) electrode. The oxidized dye molecules are regenerated by the electrons in electrolyte which is simultaneously reduced at the counter electrode [3, 4]. The semiconductor used in DSSCs must have a wide bandgap, high charge carrier mobility and film fabricated from these materials should have high surface area to obtain an efficient dye-sensitization and high light harvesting. This can best found in nanostructured material, most commonly are titanium dioxide (TiO_2) and zinc oxide (ZnO) [5-8].

Many processing techniques include sol-gel processing, hydrothermal growth, electrochemical and electrophoretic deposition, and self-assembly are used for synthesis of nanostructured materials. Dye-sensitized solar cells (DSSCs) with different photoelectrode nanostructures were studied [9]. The main nanostructural forms developed during the past several decades include nanoparticles, nanorods, nanotubes, nanobelts, nanosheets and nanotips. Considerable efforts have been devoted to the synthesis of one dimensional structures such as nanowires, nanotubes or nanorods, which have low growth temperatures, good potential for scale up and significantly enhance the electron transport speed in the photoelectrode as they provide a direct conduction pathway for the rapid collection of photo generated electrons [10].

Popcorn-style dye-sensitized solar cells with the porous electrodes made of submicron-sized aggregates (or popcorns) of ZnO nanocrystallites with enhanced power-conversion efficiencywas reprted [11-12].

Efficient ZnO-based DSSCs have been demonstrated by modifying the surface of the ZnO surfaces, where the stability of the ZnO in the electrolyte solution is found to reduce their efficiency and makes it out of competition with the best achievement of the TiO_2 dye-sensitized solar cells. So that it is very important to modify the surface of the ZnO surfaces or to grow new nanostructured architecture of ZnO, such as nanowires, nanotubes or nanorods [13-14] , which have low growth temperatures and good potential for scale up [15-16].

In this work, the effect of the chemical precursors used in preparing ZnO particles on the structure and morphology of hydrothermally grown particles is studied. ZnO particles synthesized with different chemical precursors are used in

preparing photoelectrodes for DSSCs. The performance of DSSCs was investigated through electrochemical measurements.

2. EXPERIMENTAL DETAILS

2.1 Powder Synthesis

Three different zinc-salts namely: zinc acetate dihydrate, reagent $Zn(CH3COO)_2.2H_2O$, Zinc nitrate ($Zn(NO_3)_2$•$6H_2O$) and zinc chloride ($ZnCl_2$), were used as starting precursors to get 0.5 M/L methanolic solutions.. A 3 M aqueous solution of sodium hydroxide (NaOH) was added drop wise to the solution under vigorous stirring forming a white suspension that was left under stirring for 12 h at room temperature. The pH value of the solution was adjusted to 12. The sols were treated hydrothermally for 12 h at 200 ^oC. The white slurry was washed with extra methanol to remove the precursor material and dried in air at 100 ^oC for 12 h. The powder was grinded using a hammer mill to reduce the size of the agglomerates until obtaining a fine powder.

2.2. Paste, Layer Deposition and Sensitization

A 2 g of ZnOpowder was wetted with 0.2 g of polyethylene glycol (PEG 400). The obtained paste was dispersed mechanically in mortar until a very soft uniform paste was obtained. A 5 mL of ethanol was then added to the paste to obtain a suitable viscosity for the coating preparation. ZnO paste was deposited on fluorine-doped SnO_2-(FTO)- coated glass substrates using the ‘‘doctor blade’’ technique to form 0.5 * 0.5 cm layers. The layers were dried at 100 ^oC for one hour, followed by heating in air at 400 $^oC$for 30min.

The dye sensitization was carried out by dipping the electrodes into a solution of 0.5 mM of Ruthenium535 (Solaronix), known as N3, in ethanol for a few hours. The layers were immersed in the dye solution while they were still warm to enhance the coloration and to prevent the capillary condensation of water vapor from ambient air inside the nanopores of the film.

2.3. Cell Assembly and Characterization

To assemble the solar cells, a Pt-coated silicone wafers was placed on the ZnOphotoelectrode separated by a 10 μm thin membrane spacer. The assembled cell was then clipped together. The electrolyte, consisted of a mixture of acetonitrile (ACN, C_2H_3N from Sigma–Aldrich) and propylene carbonate with a ratio of 20:80 vol.% respectively, and a redox system based on the iodine/triiodide system: 0.5 M lithium iodide anhydrous LiI (FluckaChemika) and 0.05 M iodine I_2 (Flucka T Chemika), were injected into the open cell from the edges by capillarity. The structure and morphologies of powders and photoelectrode surfaces were characterized by scanning electron microscopy (SEM, JEOL JSM- 7000), and the BET surface areas was determined using a Brunauer–Emmett–Teller (BET, Quantachrome NOVA 4200e). To measure the adsorbed amount of N3 dye on the ZnO films, the dye was desorbed by immersing the dye sensitized films in an 0.1 M NaOH solution in water and ethanol (with volume ratio of 1:1). An ultraviolet–visible–near-infrared spectrophotometer (UV–VIS–NIR, Perkin Elmer Lambda 900) was employed to measure the dye concentrations of the desorbed-dye solutions. Photovoltaic properties of solar cells were characterized using an EG&G Princeton Applied Research Potentiostat/ Galvanostat model 273. The illumination source was a 450 W Xenon lamp from Mueller ElectronikOptik with an intensity of 200,000 Lux as measured by a light Meter Voltcraft LX-11008. As the performance of DSSC is greatly influenced by many factors, many sets of samples were tested separately and each cell was tested many times (more than 10 times) to ensure that the obtained results are reproducible and valid.

3. RESULTS AND DISCUSSIONS

3.1 Structural ad Morphological Properties

XRD patterns

The XRD patterns of powder synthesized hydrothermally at 200^oC from three different Zn salts are shown in Fig. 1. The patterns show that the powders are highly crystalline with structure in accordance with the Wurtzite hexagonal structure (JCPDS No. 0036- 1451). The XRD data of the coatings (not shown) is not different from those of the corresponding powder as they also show the Wurtzite hexagonal structure. No changes in the structural phase nor the crystallinity are observed because the layers were deposited from already highly crystalline ZnO nanoparticles. Same conclusion is reported by Teesetsopon et al [17].

As it is seen in the figure the (100)/(002) intensity ratio for powder prepared starting with zinc acetate as precursor is considerably higher than that of powders prepared starting with zinc nitrate or zinc chloride. The variation of ratio is due to shear stress on the particles when it is compacted in the XRD holder to induce the particles to arrange their long axis vertical to the direction of the compacting force. The crystallite size calculated for the (100), (002) and (101) peaks with the lattice dimensions are shown in table 1

Table 1: Crystallite size and lattice dimensions of ZnO powder synthesized hydrothermally from three different Zinc-precursors at 200 °C then dried in air at 100 °C.

Zn- precursor	Crystal size (nm)	a (Å)	c (Å)	c/a
Nitrate	165	3.2504102	5.2069596	1.60193
Chloride	95	3.2509338	5.2071202	1.60173
Acetate	101	3.2504781	5.2071286	1.60196

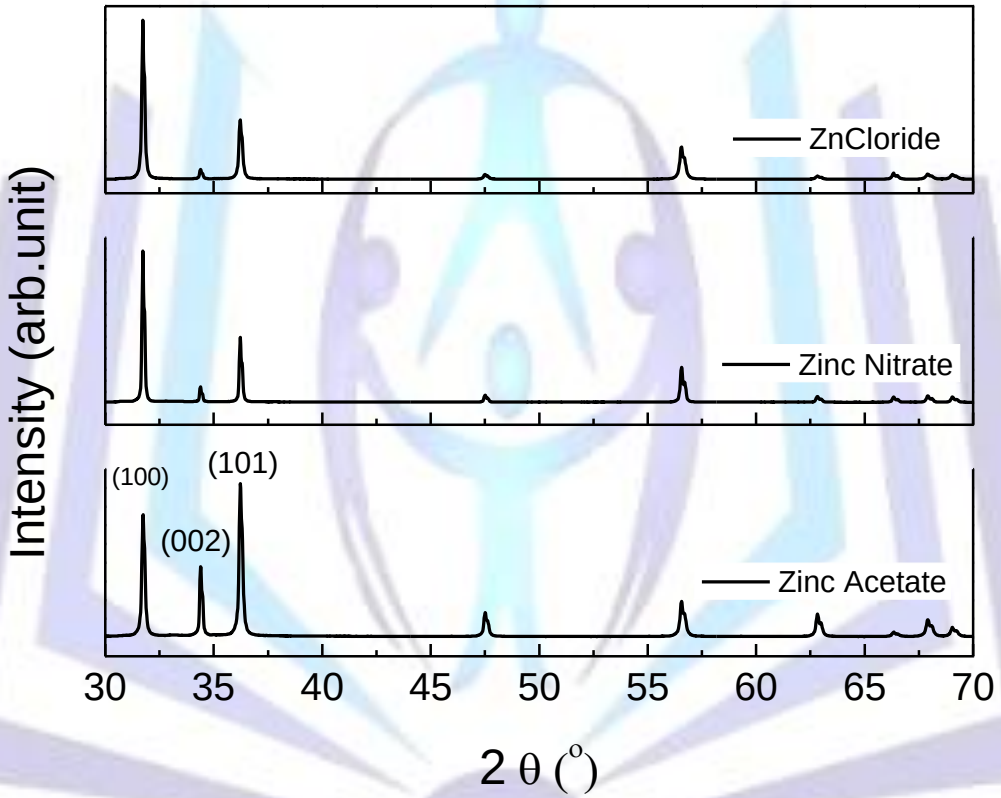

Fig. 1: XRD pattern of the ZnO powder synthesized starting with three different Zn-salts. The peaks refer to the Wurtzite hexagonal structure in the reference data (JCPDS No. 0036–1451).

SEM Micrographs

Figure 2 shows the SEM photographs of ZnO particles prepared hydrothermally from three different Zn-precursors. It is evident that the starting chemicals used for the synthesis of ZnO nanoparticles have a strong influence on the morphology of the obtained powders. As seen in Figure 2.a, particles synthesized using zinc-chloride exhibited large agglomerates with a double head asparagus pack- like shaped structure. The agglomerates are formed by an ordered assembly of hexagonal rod- like structures, 5- 10µm in length and 800nm in diameter. Using Zn-nitrate as precursor results in formation of large agglomerates having hexagonal rod-like shape with very rough surface composed of flakes. The length of the agglomerates is 10–25 µm and their diameter is 3 µm. When Zn-acetate precursor was used, flower-like ZnO agglomerates were obtained. The morphology of single particle is fine structured whisker with crystallites size of about 30 nm and length-to-width ratio of nearly 2 : 1 as depicted in Figure 2.cwhich is in agreement with XRD results.

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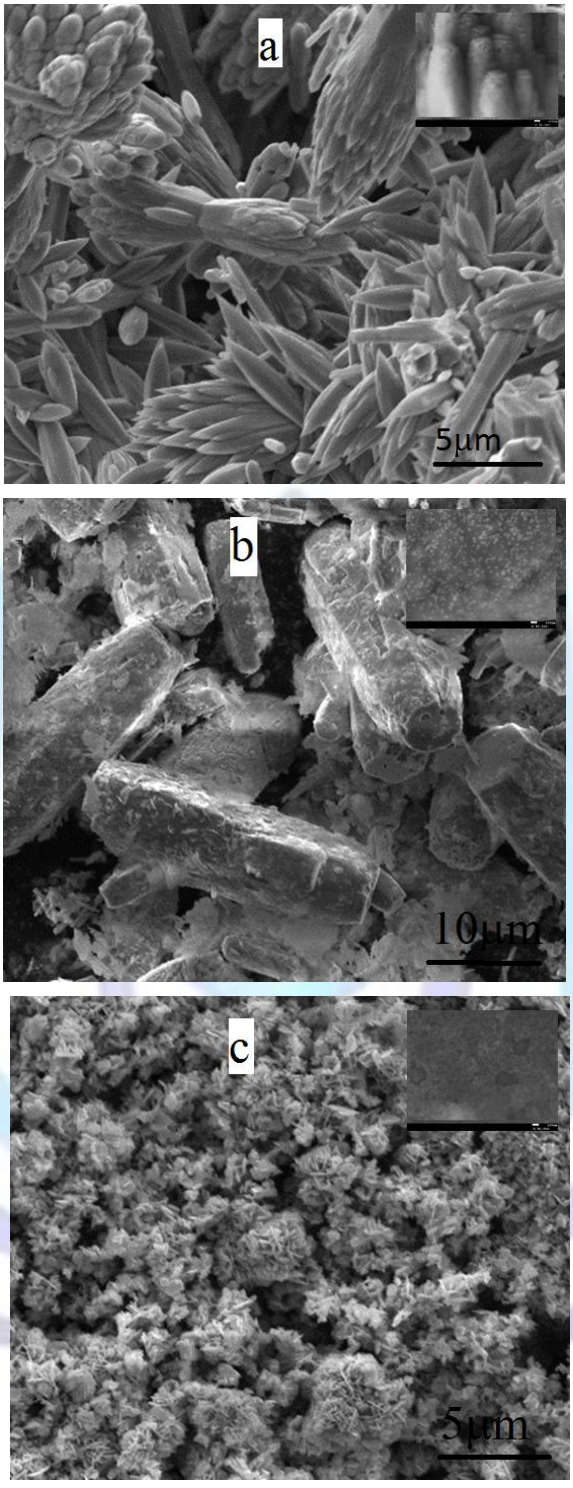

Fig. 2: SEM images of ZnO nanoparticles synthesized hydrothermally at 200^{o}C from Zn-chloride (a), Zn-nitrate (b) and Zn- acetate (c). The insets are SEM surface micrographs of the corresponding photoelectrodes.

The specific surface area and particles size are not the only factors that determine the photovoltaic efficiency of DSSC. Its performance is affected by the geometrical structure of the photoanode which provides particular properties in terms of electron transport and light propagation [18]. Surface SEM pictures of 10 m thick ZnO coating made starting with different Zn precursors, sintered in air at 400^{o}C for 30 min are shown in the inset of Fig.2. The porous morphology of the coatings is clearly seen in the pictures.

3.2. Dye Loading

Generally, DSSC efficiency is strongly dependent on the phase structure, crystallite size, surface areas, and pore structure of used particles. Higher pore volume and larger surface area allow more dye to be absorbed onto the surface of the photoanode. UV- Vis absorption is used to compare the dye loaded on ZnOphotoanodes made from different precursors. Absorption spectra in the visible region are shown in Figure 3. Samples synthesized from Zn-Acetate exhibited the largest absorption compared with the samples made from Zn-Nitrate and Zn-Chloride, which is expected as a result of the differences in particles size and hence surface areas. The surface area of ZnO particles synthesized using Zn-acetate is 6.4m^2/g while 0.67m^2/g is obtained when Zn-nitrate is used. Surface area of 4.5m/g is obtained by using Zn-chloride. This is in addition to dissimilarities in surface roughness and film porosity. The high porosity and roughness of photoanodes made from Zn-acetate as a precursor are convenient for dye loading, leading in turn to higher light harvesting efficiency and accounted for an increase in photocurrent (see Fig. 4 in section 3.3).

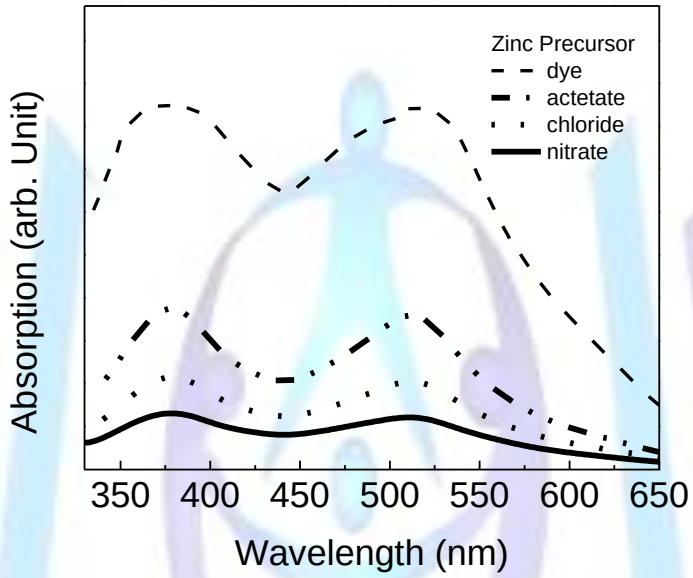

Fig. 3: UV–Vis absorbance of dye de-adsorbed from ZnOphotoanodes prepared from different Zn precursors and annealed at 400^{o}C.

3.3. Electrochemical Characterization

The I–V curves of the corresponding DSSCs are shown in Fig.4. The photovoltaic parameters, short circuit current density (Jsc), open circuit potential (Voc), fill factor (FF), and efficiency value derived from the I–V curves are summarized in Table 2. From the figure it is seen that Zn- salt used in preparing ZnO particles had great influence on the electrical properties of the obtained DSSC. This is due to changes in structure, morphology and particle size of the photoelectrodes. The highest cell efficiency of 5.4% was obtained for cells built with photoelectrodes made from ZnO nanoparticles synthesized from Zn-acetate salts, while efficiencies of only 2.07% and1.6%.were obtained for Zn-chloride and Zn- nitrate salts, respectively. The cell efficiency is strongly dependent on Jsc. The highest Jsc value of 22.4 mA/cm^2 was obtained for the photoelectrodes prepared with Zn-acetate salt, which is about double the value obtained with Zn-Chloride (10.7 mA/cm^2) and 3.5 times the value obtained with Zn- nitrate salts (5.67 mA/cm^2). Meanwhile, the open circuit potential $V_{OC,}$ which is the difference between quasi-Fermi level potential of the photoanode and the potential of the redox couple in the electrolyte value of the cells, does not show drastic changes with changing the zinc precursor. The best value of Voc (552mV) is obtained for cell with Zn-acetate. This indicates higher quasi-Fermi level (higher density of state) due to lower electron recombination and faster transport. These findings imply that the improvement of cell efficiency, of cell made with photoanode prepared from Zn-acetate as precursor, is due to large increase in the photogenerated electrons on the ZnO electrode because of the increased amount of dye adsorbed on ZnO particles with larger surface areas (6.4m^2/g) (Figs. 2a and 3).

Overall FFs shown in this study are low as compared to the reported values of ZnO-based DSSCs. Low FFs may result from a higher series resistance and/or a lower parallel or shunt resistance [19]. The series resistance is caused by the bulk resistance of semiconductor oxide layers, transparent conductive oxide electrode, metallic contacts and electrolyte, thus increased series resistance reduces FF and Jsc. The parallel or shunt resistance results from the leakage path across the dye/oxide interface induced by defects in the bulk and at the surface of the oxide, and decreased parallel or shunt resistance reduces FF and Voc [20].

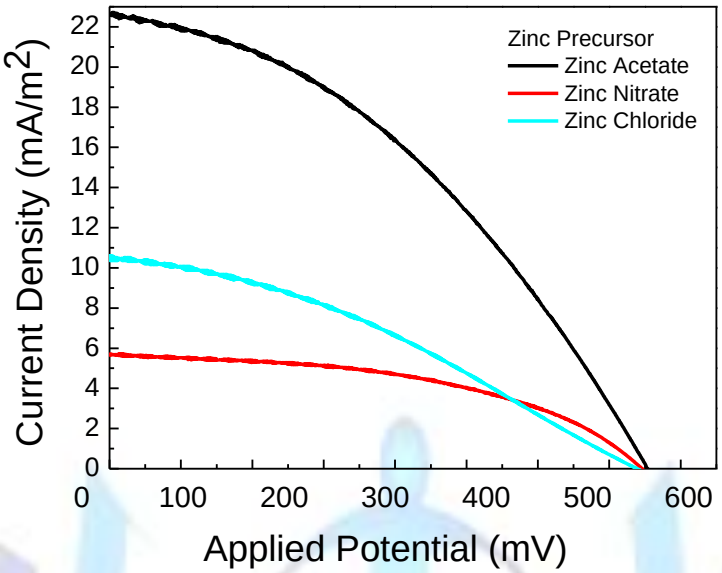

Fig. 4: Current–Voltage characteristics of ZnO- DSSCs with photoelectrodes made from ZnOpodersynthesized starting with different Zn-precursors and annealed at 400°C

Table 2: electrochemical performance of ZnO–DSSC with photoelectrodes made from ZnO particles synthesized hydrothermally from three different Zinc-precursors at 200 °C, sintered in air at 400°C.

Zn-precursor	Jsc (mA)	Voc (mV)	FF	η(%)
Zn-chloride	10.36	550	0.354	2.07
Zn-nitrate	5.65	550	0.523	1.6
Zn-acetate	22.6	552	0.434	5.4

CONCLUSION

Effect of starting Zn- precursor on the structure and morphology of hydrothermally synthesized ZnO microporous particles was studies using three different Zn precursor. Highly order structures with large particle size and small surface area were obtained regardless of the used Zn-precursor. However the smallest particles size (30nm) is obtained when Zn-acetate was used.

DSSC photoanodes were fabricated using the obtained mesoporous ZnO particles and influence of Zn- precursor on the microstructure, dye-adsorption, current density, and power conversion efficiency of ZnO-DSSCs was studies. Generally, hydrothermal synthesis technique is found to be inappropriate for synthesizing particles for DSSC. This is due to large particle size and small surface area of the obtained powder, which hinder the dye adsorption and promote charge recombination, resulting in both low photocurrent density and low open circuit voltage of the DSSCs.

In addition, the large size of photoanode particles results in bad contact between the adjacent particles which hinders electron transport and collection. DSSC built with photoelectrodes made from Zn-acetate has the highest efficiency (5.4%) which is attributed to having lowest particle size and largest surface area available for dye loading.

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- Synthesis of nanocrystalline particles via wet
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- Producing coating with electrochromic properties via sol-gel method, assembling and characterization of electrochromic devices
- Producing transparent conductive coatings on glass from colloidal precursor.
- Producing of coating for solar cell application, assembling and characterization of dye sensitized solar cell.
- Thin film Characterization (electrical, optical, mechanical, structural and surface properties)

Professional honors, awards and fellowships

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Application of the carbon dioxide waveguide laser to photoacoustic and photopiezoelectric detection of essential oils, Keele University, Keel, England, 1999

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