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Synthesis of ZnO Nanostructures Using Sol-Gel Method

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Abstract

Zinc oxide plays an important role in current industry due to its special characteristics such as anti-corrosion, anti-bacteria, has low electrons conductivity and excellent heat resistance. Therefore, the objective of this study is to synthesize zinc oxide nanostructures with the most practical ways by using sol-gel method and characterize the nanostructures. Sol-gel method is the simplest method and has the ability to control the particle size and morphology through systematic monitoring of reaction parameters. ZnO nanoparticles were synthesized via sol gel method using Zinc acetate dehydrate ($Zn(CH_3COO)_2.2H_2O$) as a precursor and ethanol (CH_2COOH) was used as solvent, Sodium hydroxide (NaOH) and distilled water were used as medium. ZnO nanoparticles were characterized by using XRD, EDX, FESEM, and nano-particles analyser. Result of EDX characterization shows that the ZnO nanoparticles has good purity with (Zinc content of- 55.38% and; Oxygen content of- 44.62%). XRD result spectrum displays mainly oxygen and zinc peaks, which indicate the crystallinity in nature as exhibited. FESEM micrographs shows that synthesized ZnO have a rod-like structure. The obtained ZnO nanoparticles are homogenous and consistent in size which corresponds to the XRD result that exhibit good crystallinity. ZnO nanoparticles were successfully synthesized by sol-gel method in nanosize range within 81.28nm to 84.98nm.

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1. Introduction

Synthesis of metal nanoparticles with specific properties is a newly established research area that attracts a great deal of attention. There are several methods that have been put forward for synthesis of these materials, namely chemical vapor condensation, arc discharge, hydrogen plasma-metal reaction, and laser pyrolysis in the vapor phase, microemulsion, hydrothermal, sol-gel, sonochemical, and microbial processes taking place in the liquid phase, and ball milling carried out in the solid phase[1,2]. The properties of metal nanoparticles depend largely on their synthesis procedures. The variety of metal oxide is great and their range of properties and possible applications appear to be enormous. Zinc Oxide is very suitable for sensor and transducer usage with its relatively bio-safe and biocompatible material. Besides, nanostructured metal oxide has been found to display appealing nano-morphological, functional, biocompatible, non- toxic and catalytic properties[3]. The market demand for the ZnO nanopowders is increasing and widely used in industries due to their ultraviolet filtering, catalytic, anti-corrosion and anti-bacterial properties. Recently, they have mainly been used in sunscreens as an ultraviolet-resistant additive. Other applications of zinc oxide nanopowder include electrophotography, photoprinting, capacitors, protective coatings, anti-microbial, and conductive thin-films in LCDs, solar cells, and blue laser diodes[4].

2. Experimental

Zinc Oxide nanostructure was synthesized by using sol-gel method. In order to prepare a sol, 2 g of Zinc Acetate Dihydrate and 8 g of Sodium Hydroxide were weighted using a weighting balance. Then, 10 ml and 15 ml of distilled water were measured by a measuring cylinder. After that, 2 g of zinc acetate dihydrate was dissolved with a 15 ml of distilled water and 8 g of sodium hydroxide was dissolved in a 10 ml of distilled water. The solutions were stirred with a constant stirring for about five minutes each. After well mixed, sodium hydroxide solution was poured to the solution containing zinc acetate with a constant stirring by magnetic stirrer for about five minutes. Then, a burette was filled with 100 ml of ethanol and tiltrate dropwise to the solution containing both sodium hydroxide solution and zinc acetate. After the reaction, white precipitate was formed.

2.1 Synthesis of Zinc Oxide Nanostructure

Synthesizing Zinc Oxide nanostructure via sol gel technique in this research includes the use of several materials such as Zinc Acetate Dihydrate ($Zn(CH_3COO)_2.2H_2O$) ≥99% purity (HmbG Chemicals), Sodium hydroxide (NaOH) ≥98% (Sigma Aldrich), Ethanol (CH_2COOH) HmbG Chemicals) and distilled water. Zinc Acetate Dihydrate was used as precursor and Ethanol was used as a reagent. Distilled water was used as a solvent medium.

3. Results and Discussion

3.1 Zinc Oxide Nanostructures

Based on the experimental work that has been done, there are series of chemical reaction that takes place. The complete hydrolysis of zinc acetate with the aid of NaOH in an ethanolic solution should result in the formation of a ZnO colloid. The final product was obtained as a result of the equilibrium between the hydrolysis and condensation reaction. Due to the heating, Zinc Acetate within the solution undergoes hydrolysis forming acetate ions and zinc ions. The abundance of electrons in the oxygen atoms makes the hydroxyl groups (-OH) of alcohol molecules bond with the zinc ions[5]. The overall chemical reaction to form ZnO nano-powder when sodium hydroxide was used as solvent stated as follow in Eq. (1):

$$(Zn(CH_3COO)_2.2H_2O) + 2NaOH \longrightarrow ZnO + 2NaCH_3COO + H_2O \quad (1)$$

Zinc hydroxide acetate is an intermediate product of the hydrolysis reaction, formed in the presence of H_2O and OH- ions. It can be easily transformed into ZnO at higher temperature and with prolonged refluxing. Sodium acetate is water soluble and could therefore be removed from the end product. High purity ZnO nano-powder could therefore be obtained successfully by sol gel technique[6].

3.2 Field Emission Scanning Electron Microscope (FE-SEM)

There are varieties of ZnO nanostructures that had been discovered, which are in a form of nanorods, nanotubes, nanobelts, nanosprings, nanospirals, nanorings and many more (Bahadur et al. 2008). In addition, rod like structure is the best nanostructure compared to others due to their one-dimensional nanostructures (example: nanorods, nanowires, and nanotubes) that can facilitate more efficient carrier transport because of decreased grain boundaries, surface defects, disorders, and discontinuous interfaces (Morkoç & Ozgur, 2009; and Moezzi, et al., 2012). Morphology study has found out synthesized ZnO are in form of rod like structure as the result of zinc acetate were used as precursor. It was supported by other findings, which Bahadur et.al (2007) claimed that the morphology of ZnO nano-powder using zinc acetate is smoother than in zinc nitrate. Moreover, precursor concentration plays a great role on morphological features of nanorod. According to Pourrahimi et al. (2014), ZnO can be form in different structures due to the type of precursors that has been used, such; Zinc acetate nano rod like structure, zinc chloride and sulphate-nano-prism structure and zinc nitrate-prism flower shape structure. Kashif et al. 2012 claimed that increase in the concentration of precursor would increase the growth and density of ZnO nanorod, when length of nanorod is reduced. In addition, rod like structure is the best nanostructure compared to others due to their one-dimensional nanostructures (example: nanorods, nanowires, and nanotubes).

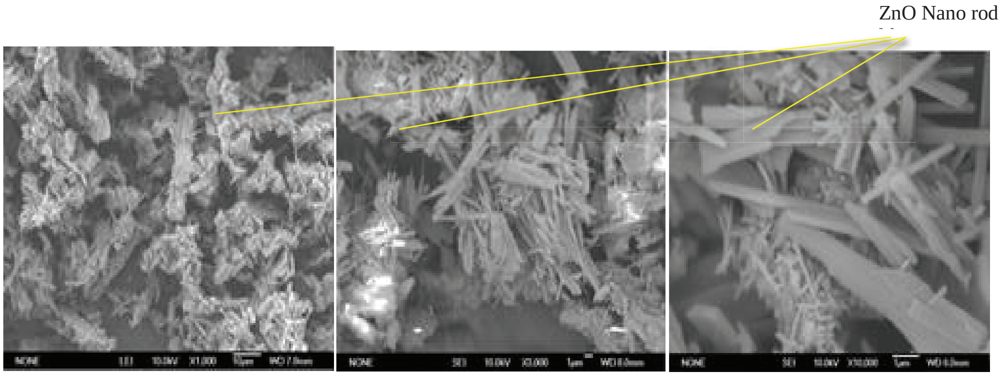

Fig 1. FE-SEM micrograph of zinc oxide synthesized in different magnification.

3.3 Nano particle analyzer

As showed in Fig.2 and Table 1, for particle size analysis it has absolutely confirmed that the synthesized ZnO powder is in nanosize form. The particle size of synthesize ZnO powder is about 84.98 nm. This result is based on their length. When measure the particle size analyzer, the assumption is based on the lenght of structure. This result correspond to the XRD which indicated that the synthesized ZnO nano-powder exhibit good crystallinity. It is considered as a good result because the particle size of synthesized ZnO is below than 100nm as agreed by Farley et al. (2010).

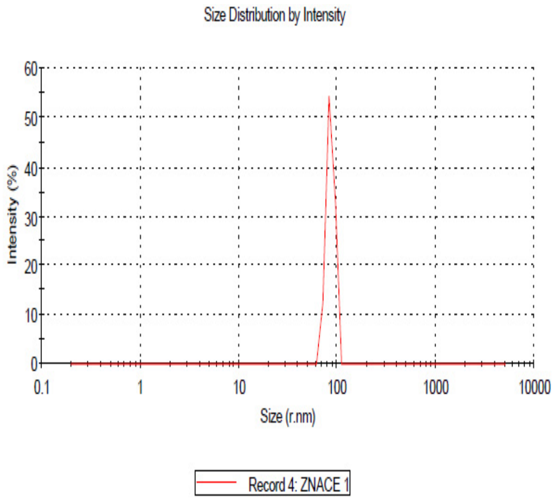

Fig. 2: Particle size analyser for ZnO using Zn Acetate as precursor

Table 1: Data for ZnO nano-powder using Zn Acetate as a precursor

Size (r.nm)	% Intensity	Width (r.nm)
84.98	100.0	7.945

3.4. Energy Dispersive X-ray Spectroscopy (EDX)

Fig. 3 and Table 2 show results obtained from the EDX characterization suggested that the ZnO powder has good purity (Zinc content - 55.38%; Oxygen content - 44.62%), in which very little impurities can be seen. Theoretically, expected stoichiometric mass per-cent of Zn and O are 80.3% and 19.7%. (Bari et al. 2009 & Tarwal et al. 2011). Some findings found that, by using sodium hydroxide as a solvent, the EDX graph shows near to stoichiometry in value compared to ammonium hydroxide (Bari et al. 2009). The composition of zinc element is higher than in synthesized ZnO nano-powder. This result is correspond to the finding done by Tarwal et al. (2011), claimed that ZnO nano-powder are nearly stoichiometric.

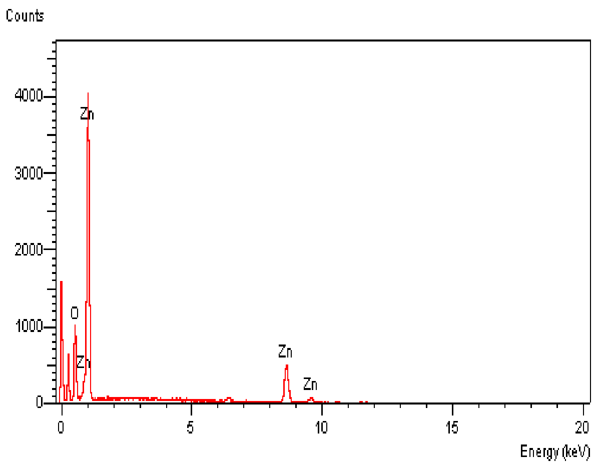

Fig. 3. EDX graph of ZnO nanopowder using Zinc Acetate as a precursor.

Table 2. TEM machine (JEOL 2010F) instrument specification.

Element	Spectrum Type	Intensity Corrn.	Standard Corrn.	Element %	Sigma %	Atomic %
O K	ED	1.153	0.65	44.62	0.87	76.70
Zn K	ED	0.891	1.00	55.38	0.87	23.30

3.5 Differential Scanning Calorimetry (DSC)

The ZnO nano-powder was further characterized by Differential Scanning Calorimetry, or DSC. Thermal analysis technique was used to study on how a material's heat capacity (Cp) is changed by temperature. Fig. 5 below is the DSC curve characterization based on ZnO nano-powder. Based on the DSC thermograph, each sample show endothermic peak but there is slightly exothermic peak at temperature 26°C for ZnO nano-powder. The endothermic peaks are primarily caused by the vaporization of the ethanol in the solution. The exothermic peak is from the abrupt change of heat transfer between the sample and pan and also from distortion of sealed pan due to vapour pressure of the sealed pan due to vapour pressure of the sample.

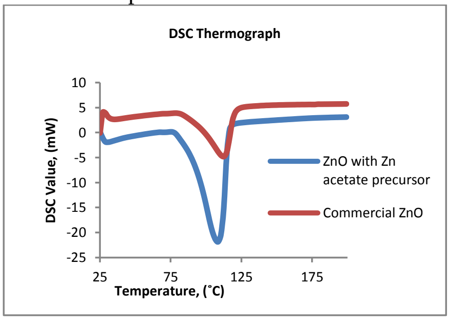

Fig. 4. DSC thermogram for synthesised ZnO nano-powder.

3.6 X-Ray Diffractometer (XRD)

XRD analysis determined the phase's presence in nanopowder. XRD result as shows in Fig. 6 are the resulting pattern of ZnO nano-powder in various profiles of peak and diffraction angle 2θ, which represent the diffraction of ZnO nano-powder using Zinc Acetate as precursor. The XRD pattern of the powder was recorded in the fraction angle range 20° to 80°. The figure shows sharp peaks of ZnO, which indicate the crystallinity in nature. The synthesised powder, ZnO nano-powder showed a single phase with clear diffraction peaks; which correspond to the reported data (JCPDS card no 36-1451). The highest peak is at angle 33.8616° at (101) plane with 4847.45 intensity and at angle 36.2695° with intensity 4069.75. The produced ZnO nano-powder is the compound of Zinc Oxide with 63%.

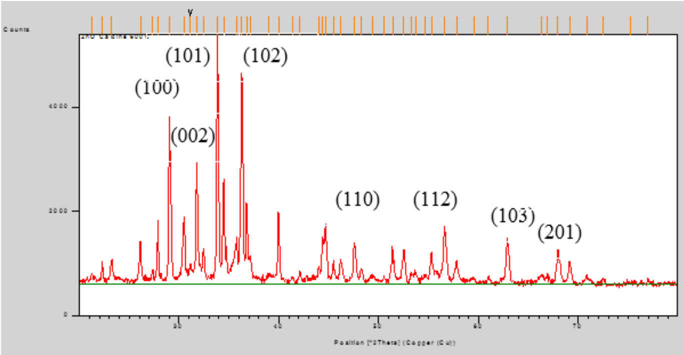

Fig. 5. XRD patterns of zinc oxide.

4. Conclusion

The zinc oxide nanostructure was successfully synthesized by using sol-gel method. The results showed that the ZnO rod like structure was successfully synthesized by sol gel method in nanosize range about 84.98nm. The synthesized ZnO nano-powder obtained exhibit good crystallinity.

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References

1. Kargari, A., Tavakoli, A., & Sohrabi, M. (2007). A review of methods for synthesis of nanostructured metals with emphasis on iron compounds.Chemical Papers, 61(3), 151-170.
2. Research and market. (2013). The Global Market for Metal Oxide Nanoparticles to 2020. Retrieved from http://www.researchandmarkets.com/reports/2488811/the_global_market_for_metal_oxide_nanoparticles
3. Wahab, H.A & Salama, A.A et al. (2013). Optical, structural and morphological studies of ZnO nano-rod thin film using sol-gel.3, 46-51.
4. Chai, C. (2012). The Global Market for Zinc Oxide Nanopowders 2012. New Report on Global Zinc Oxide Nanopowder Market, 135-140.
5. Yung, K., Ming, H., Yen, C. & Chao, H., (2012). Synthesis of 1D,2D and 3D ZnO Polycrystalline Nanostructures Using Sol-Gel Method.Journal of Nanotechnology, 1-8.
6. Bari, A. R., Shinde, M. D., Vinita. D. & Patil, L. A. (2009). Effect of Solvents on the Particle Morphology of nanostructured ZnO. *Indian Journal of Pure & Applied Physics*, *47*, 24-27.
7. Morkoç, H., &Özgür, Ü. (2009). Chapter 1 General Properties of ZnO. In Zinc Oxide: Fundamentals, Materials and Device Technology. Wiley-VCH.
8. Moezzi, A., McDonagh, A.M, & Cortie, M.B. (2012). Zinc oxide particles: Synthesis, properties and applications. Chemical Engineering Journal, 185–186, 1–22.