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SYNTHESIS AND CHARACTERIZATION OF ZNO NANOPARTICLES BY SOL-GEL METHOD WITH VARIOUS ADDITIVES

Sherly Kasuma Warda Ningsih*, Miftahul Khair, Silvi Veronita
Department of Chemistry, Faculty of Mathematics and Natural Sciences
Padang State University, Kampus Air Tawar, Jl. Prof. Dr. Hamka, West Sumatera 25161, Indonesia
**Corresponding author: sherly_kasuma@yahoo.com*

ABSTRACT

Preparation of zinc oxide (ZnO) nanoparticle with various additives by sol-gel process has been studied. ZnO nanoparticles were obtained by using zinc sulfate heptahydrate as the precursor, ethanol as the solvent. Ethylene glycol and urea were used as the additives by the addition of potassium hydroxide solution to adjust the pH value. The powders were formed by drying in the temperature of 105 °C for 60 minutes and after heating at 500 °C for ± 1 hour. The products were obtained in white powders. The synthesized ZnO were characterized by X-ray diffraction (XRD) and Scanning Electron Microscopy (SEM). The XRD patterns showed ZnO forms were produced generally in hexagonal structure (*wurtzite*). Crystallite sizes of ZnO were estimated by using Scherrer equation. The particle size of ZnO prepared by using ethylene glycol and urea obtained in the range 18-70 nm and 26-75 nm respectively. SEM micrograph of ZnO shows agglomeration of hexagonal nanoparticles and the the distribution size is 0.1-1.0 μm approximately.

Index Terms— *ZnO nanoparticle; additive; hexagonal; sol-gel; agglomeration*

1. INTRODUCTION

Zinc oxide (ZnO) is one of the pupular of semiconductor that have a wide band gap of 3.37 eV with large exciton binding energy of 60 meV[1,2]. ZnO have extensive application doe to its electrical and optical properties. ZnO can be applied in many applications, including gas sensors[3,4], generators[5], field emission transistors[6], ultraviolet photodetectors[7], in biomedical systems[8], biosensors[9], electric materials[10], light emitting diode[11], solar cells[12] and piezoelectric tranducer[13].

There are several ways to produce ZnO nanoparticles such as thermal decomposition14,15, carbothermal reduction process16, solid sate method17, hydrothermal process18, sonochemical methods19, chemical vapor deposition (CVD)2, 20,21, metal organic chemical vapor deposition (MO-CVD)11, polymerization method22, precipitation process223,24, and sol-gel method25,26,27. Sol-gel process is one of the simplest and lowest cost (inexpensive)28.

Here, the synthesized ZnO nanoparticles prepared by sol-gel method. Zinc sulfate heptahydrate was used as precursor, ethanol as solvent, and potassium hydroxide as pH adjuster. The various additives were used in this research including ethylene glycol and urea. Sol-gel method has a number of advantages over other methods such as inexpensive equipment, will produce small particle size and uniform distribution particle with highest homogenity.

2. MATERIAL AND METHODS

2.1. Material

All the reagents were analytical reagent grade and were used without further purification. The various additives used in this research were ethylene glycol and urea, zinc sulfate heptahydrate ($ZnSO_4.7H_2O$) as the precursor. Ethanol (pa) was used as the solvent and potassium hydroxide was used as pH adjuster.

2.2. Synthesis of ZnO nanoparticles

In a typical process, 0.5 M zinc sulfate heptahydrate ($ZnSO_4.7H_2O$) was dissolved in appropriate amount of ethanol at room temperature. Aqueous solution of ($ZnSO_4.7H_2O$) was kept on magnetic stirrer for 1.5 hour and 2.0 mL ethylene glycol was added and followed by addition of KOH solution drop-wise till the turbidity occurred (pH=8) for 3 hours. Thus, the white precipitations were formed and which were washed several times with distilled water. The samples were allowed to dry in an oven at 105 °C for 60 minutes and followed with calcinations in a furnace at 500 °C for 1 hour. Finally, the dried powder was ground in agate mortar. The synthesized ZnO nanoparticles were analyzed by using X-ray Diffraction (XRD) with a diffractometer by using monochromatic CuKα with λ = 1.5406 and morphological study was carried out by Scanning Electron Microscopy (SEM).

3. RESULT AND DISCUSSION

3.1. Sol ZnO preparations

Table 1. Observation data of sol ZnO with various additives

Additives	Observation
Ethylene glycol	Zinc sulfate heptahydrate was easily dissolved in ethanol, after addition of 2

	mL ethylene glycol produced homogenous sol and followed by addition of KOH solution approximately 10 drop wises, addition of KOH solution followed until the pH value reached 8.
Urea	Zinc sulfate heptahydrate was easily dissolved in ethanol solvent, after addition of 1.0 g of urea produce homogenous sol of ZnO and followed by addition of KOH solution around 12 mL will produce milky white solution, the pH solution reached 8.

3.2. Characterization of ZnO nanoparticles

3.2.1. XRD patterns

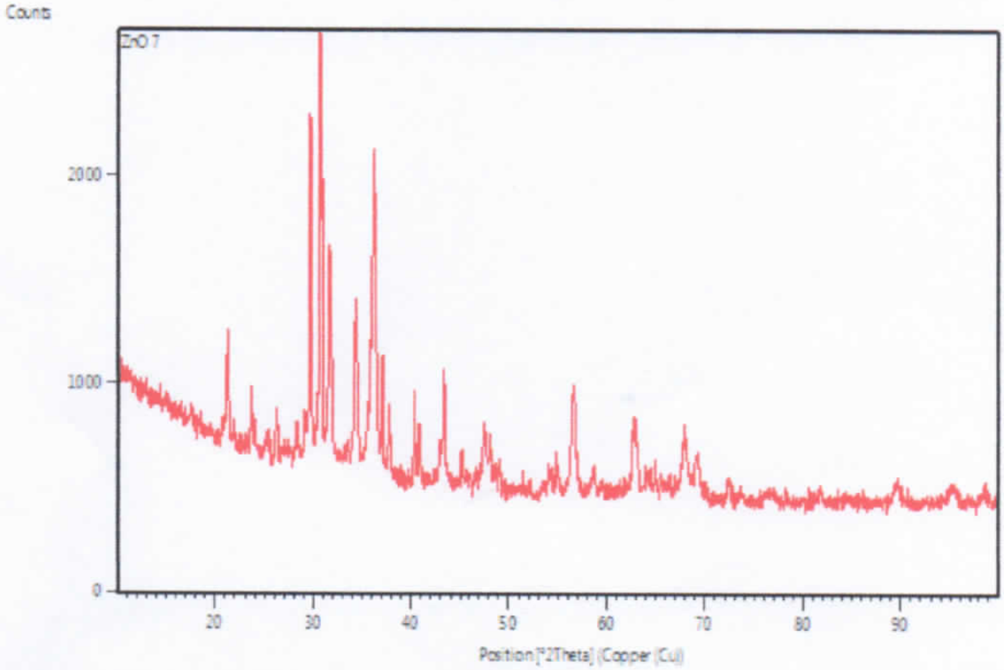

Fig.1. XRD pattern of ZnO nanoparticle synthesized by using ethylene glycol as additive

The X-ray diffraction pattern of the ZnO synthesized by using ethylene glycol as additive was shown in Fig.1. This data clearly shows distinct peaks at 2θ = 31.72; 34.42; 36.25; 47.54; 56.58; 62.82; 67.90 and 69.23. The peaks have been identified as peaks of hexagonal ZnO (*wurtzite*) crystallites with various diffracting planes [100], [002], [101], [102], [110], [103], [112] and [201], respectively. ZnO nanoparticle posses a high crystallinity since all the peaks was very sharp. All of the reflections in this pattern can be readily indexed to a hexagonal phase of ZnO which is in good agreement with the literature result(Pdf Card No. 01-078-2595). The other peaks observed at an angle 2θ of 21.30 and 30.74 which have been identified as potassium sulfate (Pdf card No. 00-005-0613). The average crystalline size of the synthesized ZnO nanoparticle prepared by using ethylene glycol as additive was calculated by using Scherrer to be about 18- 70 nm (Table 2).

Table 2. XRD data of ZnO nanoparticle prepared by using ethylene glycol as additive

Angle (2θ)	Height (counts)	FWHM	d-value [Å]	Relative intensity (counts)	Crystallite size (nm)
31.72	1056.69	0.1279	2.8209	50.68	63.9
34.42	813.74	0.1791	2.6056	39.03	46.0
36.25	1550.09	0.2558	2.4779	74.35	32.6
47.54	277.82	0.2558	1.9125	13.33	33.6
56.58	507.10	0.1279	1.6266	24.32	69.9
62.82	355.78	0.3852	1.4792	17.06	23.9
67.90	304.83	0.2558	1.3803	14.62	37.0
69.23	179.22	0.5117	1.3572	8.60	18.7

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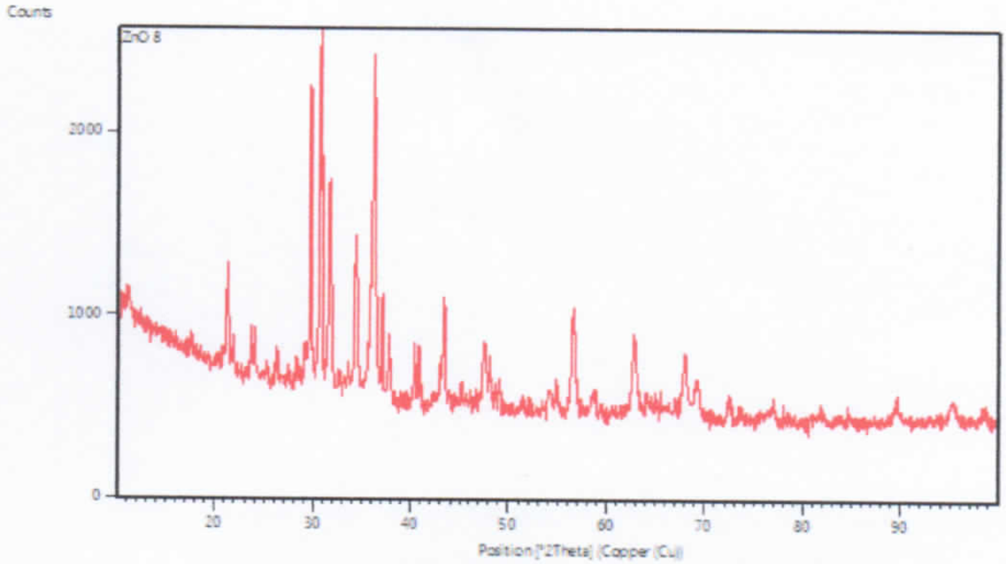

Fig.2. XRD pattern of ZnO nanoparticle synthesized by using urea as additive

The crystallinity and phase of the products were examined by X-ray Diffraction. The X-ray diffraction pattern of the product synthesized was shown in Fig.2. This data clearly shows distinct peaks at 2θ = 31.78; 34.43; 36.28; 47.55; 56.62; 62.82; 67.94 and 69.36. The peaks have been identified as peaks of hexagonal ZnO (*wurtzite*) crystallites with various diffracting planes [100], [002], [101], [102], [110], [103], [112] and [201], respectively. ZnO nanoparticle posses a high crystallinity since all the peaks was very sharp. All of the reflections in this pattern can be readily indexed to a hexagonal phase of ZnO with space group *P63mc* which is in good agreement with the literature result (Pdf Card No. 01-078-3322). The other peaks observed at an angle 2θ of 21.32 and 30.74 which have been identified as potassium sulfate (Pdf card No. 00-044-1414).The avarage crystalline size of the synthesized ZnO nanoparticle prepared by using urea as additive was calculated in the range of 26-75 nm (Table 3).

Table 3. XRD data of ZnO nanoparticle synthesized by using urea as additive

Angle (2θ)	Height (counts)	FWHM	d-value [Å]	Relative intensity (counts)	Crystallite size (nm)
31.78	1121.55	0.2047	2.8154	59.18	39.9
34.43	848.27	0.2303	2.6047	44.76	35.7
36.28	1724.93	0.2303	2.4764	91.01	35.9
47.55	346.74	0.2558	1.9121	18.30	33.5
56.62	577.46	0.1535	1.6255	30.47	58.2
62.82	450.98	0.1279	1.4793	23.80	72.0
67.94	302.06	0.3582	1.3798	15.94	26.5
69.36	199.03	0.1279	1.3548	10.50	74.9

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3.2.2. SEM images of ZnO nanoparticles

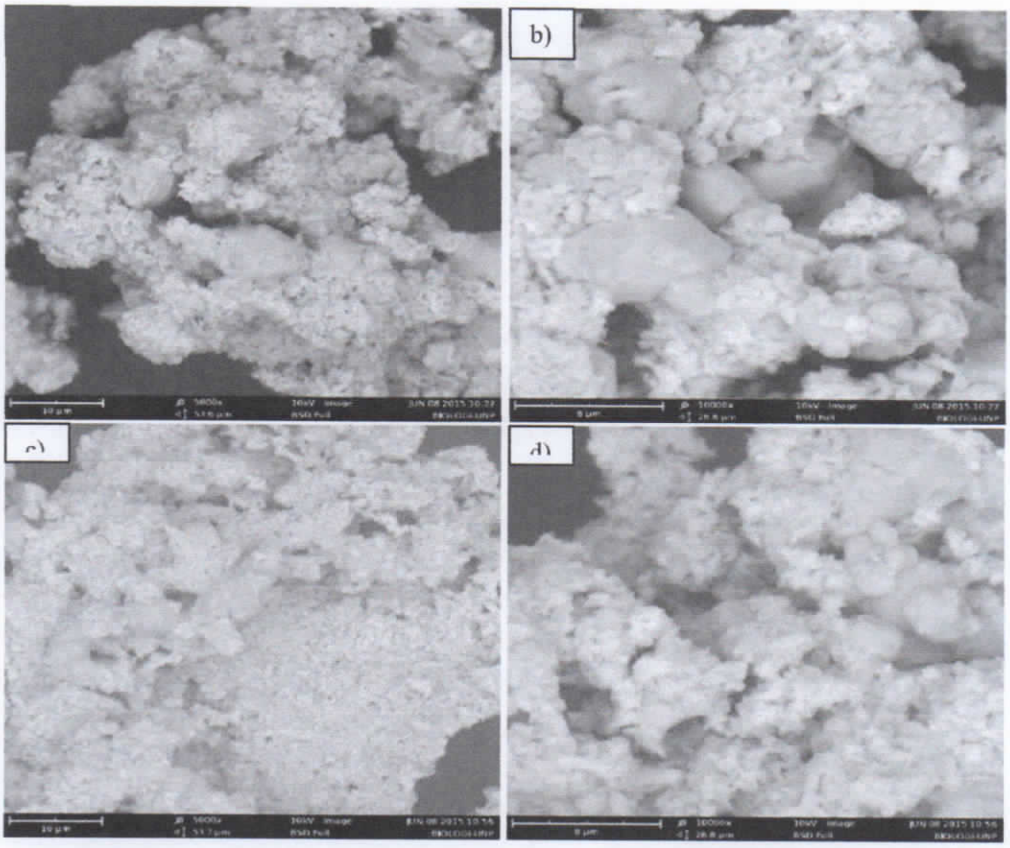

Fig.3. SEM images of ZnO nanoparticles synthesized by using a) and b) ethylene glycol as additive, c) and d) urea as additive

Fig. 3 depicts the representative SEM micrographs of ZnO nanoparticles. The effect of various additives was studied in order to obtain the smallest particles size of ZnO. The synthesized ZnO nanoparticle prepared by using ethylene glycol (Fig 3.a and b) exhibited a small agglomeration of hexagonal nanoparticles. ZnO nanoparticles were prepared by using urea (Fig 3.c and d) shows agglomeration of hexagonal nanoparticles. The additives have been influenced to the structure and the particle size of the synthesized ZnO.

4. CONCLUSION

ZnO nanoparticles were successfully prepared by sol-gel method with various additives. The additives play a significant role on the crystalline size and morphology of the ZnO nanoparticles. XRD data for ZnO prepared by using ethylene glycol shows the hexagonal (*wurtzite*) structure of ZnO with crystalline sizes in the range of 18-70 nm. The structure of ZnO prepared by using urea as additive shows the hexagonal (*wurtzite*) structure of ZnO with crystalline sizes in the range of 26-75 nm. SEM images of the synthesized ZnO confirmed the agglomeration of hexagonal nanoparticles. Ethylene glycol is the best additives for preparation of ZnO nanoparticles.

5. ACKNOWLEDGEMENTS

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6. REFERENCES

[1] Dutta S, Chattopadhyay S, Sarkar A. Role of defects in tailoring structural, electrical and optical properties of ZnO. *Prog Mater Sci* 2009; 54:89–136.

[2] J.J. Wu., S.C. Liu. Low temperature growth of well-aligned ZnO nanorods bychemical vapor deposition. Adv. Mater; 2002; 14: 215–218.

[3]N. Hongsith., C. Viriyaworasakul., P. Mangkorntong., N. Mangkorntong., S. Choopun. Ethanol sensor based on ZnO and Au-doped ZnO nanowires; Ceramics International; 2008; 34: 823–826.

[4] Y. Zong., Y. Cao., D. Jia, S. Bao., Y. Lu. Facile synthesis of Ag/ZnO nanorods usingAg/C cables as templates and their gas-sensing properties; Mater. Lett; 2010; 64: 243–245.

ISBN 978-602-19877-3-5

[5] X. Wang., J. Song., Z.L. Wang. Nanowire and nanobelt arrays of zinc oxide from synthesis to properties and to novel devices; Journal of Materials Chemistry; 2007; 17: 711–720.

[6] M.S. Arnold., P. Avouris., Z.W. Pan., Z.L. Wang. Field-effect transistors based on single semiconducting oxide nanobelts; Journal of Physics Chemistry B; 2003; 107: 659–663.

[7] J.H. Jun., H. Seong., K. Cho., B.-M. Moon, S. Kim. Ultraviolet photodetectors based on ZnO nanoparticles; Ceramics International; 2009; 35: 2797–2801.

[8]N. Kumar., A. Dorfman., J.I. Hahm; Ultrasensitive DNA sequence detection using nanoscale ZnO sensor arrays; Nanotechnology; 2006; 7: 2875–2881.

[9] Zhao Z, Lei W, Zhang X, et al: ZnO-Based Amperometric Enzyme Biosensors. Sensors 2010, 10:1216–1231.

[10]B.B. Straumal., A.A. Mazilkin., S.G. Protasova., A.A. Myatiev., P.B. Straumal., E. Goering. Amorphous grain boundary layers in the ferromagnetic nano grained ZnOfilms; Thin Solid Films; 2011; 520: 1192–1194.

[11] D.C. Kim., W.S. Han., B.H. Kong., H.K. Cho., C.H. Hong. Fabrication of the hybridZnO LED structure grown on p-type GaN by metal organic chemical vapordeposition; Physica B; 2007; 401-402: 386–390.

[12] A. Belaidi., T. Dittrich., D. Kieven., J. Tornow., K. Schwarzburg., M. Kunst., N.Allsop., M. Ch Lux-Steiner., S. Gavrilov. ZnO-nanorods arrays for solar cellswith extremely thin sulfidic absorber; Sol. Energy Mater. Sol. Cells; 2009; 93: 1033–1036.

[13] Rasmussen JW, Martinez E, Louka P, Wingett DG: Zinc Oxide Nanoparticles for Selective Destruction of Tumor cells and Potential for Drug delivery Applications. Expt Opin Drug Deliv;2010, 7:1063–1077.

[14] S. Baskoutas., P. Gabournis., S.N. Yannopoulus., V. Dracopoulus., L. Toth., A. Chrissanthopoulus., N. Bourupoulus. Preparation of ZnO nanoparticles by thermal decomposition of zinc alginate; Matter.lett: 2008; 62: 1890-1892.

[15] P. Jajarmi. Fabrication of pure ZnO nanoparticles by polymerization method; Materials Letters:2009; 63: 2646–2648.

[16] Y.S. Lim., J.W. Park., S.T. Hong., J. Kim. Carbothermal synthesis of ZnO nanocomb structure; Materials Science and Engineering; 2006; 129 (1): 100-103.

[17] Manoj Pudukudy., Zahira Yaakob. Facile solid state synthesis of ZnO hexagonal nanogranules with excellent photocatalytic activity; Applied Surface Science; 2014; 292: 520-530.

[18]X. Fang., Y. Bando., U.K. Gautam., T. Zhai., H. Zeng., X. Xu., M. Liao., D. Golberg. ZnO and ZnS nanostructures: ultraviolet-light emitters, lasers, and sensors; Critical Reviews in Solid State and Materials Sciences; 2009; 34: 190–223.

[19] P. Banerjee., S. Chakrabarti., S. Maitra., B.K. Dutta; Zinc oxide nano-particles sonochemical synthesis, characterization and application for photoremediation of heavy metal; Ultrasonics Sonochemistry; 2012; 9:85–93.

[20] R. Yousefi., M.R. Muhamad., A.K. Zak. Investigation of indium oxide as a selfcatalyst in ZnO/ZnInO heterostructure nanowires growt; Thin Solid Films; 2010; 518: 5971–5977.

[21] R. Yousefi., M.R. Muhamad., A.K. Zak. The effect of source temperature on morphological and optical properties of ZnO nanowires grown using a modified thermal evaporation set-up; Current Applied Physics; 2011; 11: 767–770.

[22] P. Jajarmi. Fabrication of pure ZnO nanoparticles by polymerization method ; Materials Letters; 2009; 63:2646–2648.

[23] R. Song., Y. Liu., L. He. Synthesis and characterization of mercaptoacetic acidmodified ZnO nanoparticles; Solid State Sciences; 2008; 10: 1563–1567.

[24] E. Tang., B. Tian., E. Zheng., C. Fu., G. Cheng. Preparation of zinc oxide nanoparticle via uniform precipitation method and its surface modification by methacryloxypropyltrimethoxysilane; Chemical Engineering Communications; 2008; 195: 479–491.

[25] Wu YL, Tok AIY, Boey FYC, et al: Surface modification of ZnO nanocrystals. Appl Surf Sci; 2007, 253:5473–5479.

26. F. Bigdeli., A. Morsali. Synthesis ZnO nanoparticles from a new Zinc(II) coordination polymer precursor; Materials Letters; 2010; 64: 4–5.

[27] J. Zhong., J. Li., Z. Xiao., W. Hu., X. Zhou., X. Zheng. Improved photo catalytic performance of ZnO prepared by sol–gel method with the assistance of CTAB; Mater.Lett; 2013; 91: 301–303.

[28] Ningsih, S.K.W. Effect Of Various Solvent On The Synthesis Of NiO Nanopowders by Simple Sol-Gel Methods and Its Characterization; Indones.J.Chem, 2015; 15: 50-55.

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