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# PROCEEDING



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ICDMSET 2015

*Education, Mathematics, Science and Technology for  
Human and Natural Resources*

**October 22, 2015**

Inna Muara Hotel and Convention Center  
Padang, Indonesia

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Faculty of Mathematics and Science  
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**The International Conference on  
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and Technology**

**(ICOMSET 2015)**

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

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### 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  $\mu\text{m}$  approximately.

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

### 1. INTRODUCTION

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

There are several ways to produce ZnO nanoparticles such as thermal decomposition<sup>14,15</sup>, carbothermal reduction process<sup>16</sup>, solid state method<sup>17</sup>, hydrothermal process<sup>18</sup>, sonochemical methods<sup>19</sup>, chemical vapor deposition (CVD)<sup>20,21</sup>, metal organic chemical vapor deposition (MO-CVD)<sup>11</sup>, polymerization method<sup>22</sup>, precipitation process<sup>23,24</sup>, and sol-gel method<sup>25,26,27</sup>. Sol-gel process is one of the simplest and lowest cost (inexpensive)<sup>28</sup>.

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 homogeneity.

### 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 ( $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ ) 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 ( $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ ) was dissolved in appropriate amount of ethanol at room temperature. Aqueous solution of ( $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ ) 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  $\text{CuK}\alpha$  with  $\lambda = 1.5406$  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 wise, addition of KOH solution followed until the pH value reached 8.

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.

Urea Zinc sulfate heptahydrate was easily

### 3.2. Characterization of ZnO nanoparticles

#### 3.2.1. XRD patterns

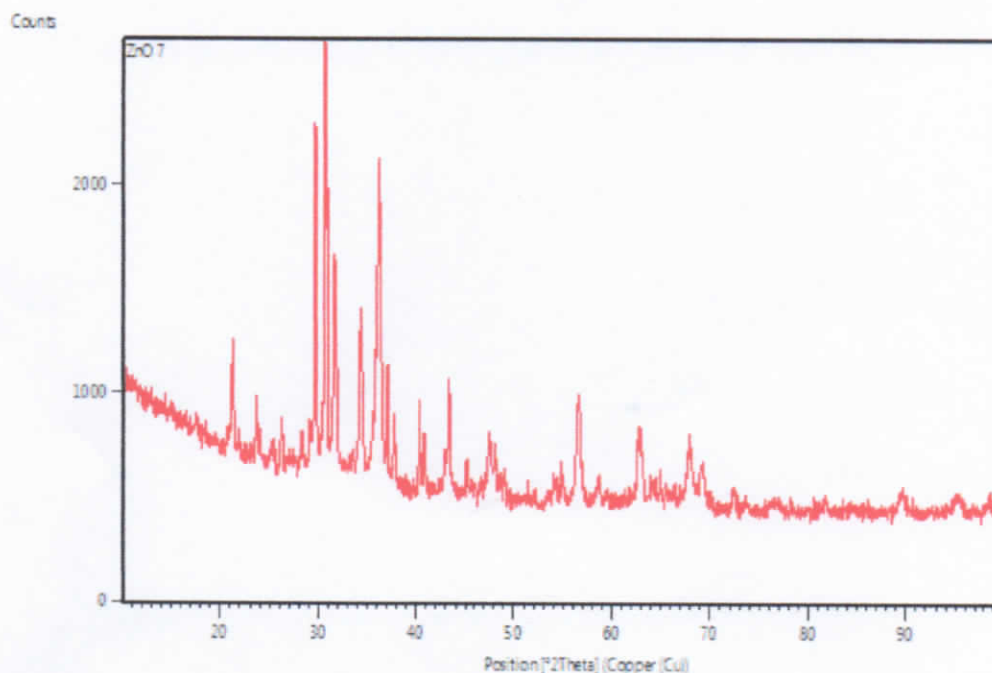


Fig.1. XRD pattern of ZnO nanoparticle synthesized by using ethylene glycol as additive

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  $2\theta = 31.72; 34.42; 36.25; 47.54; 56.58; 62.82; 67.90$  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  $2\theta$  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 ( $2\theta$ )	Height (counts)	FWHM	d-value [ $\text{\AA}$ ]	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



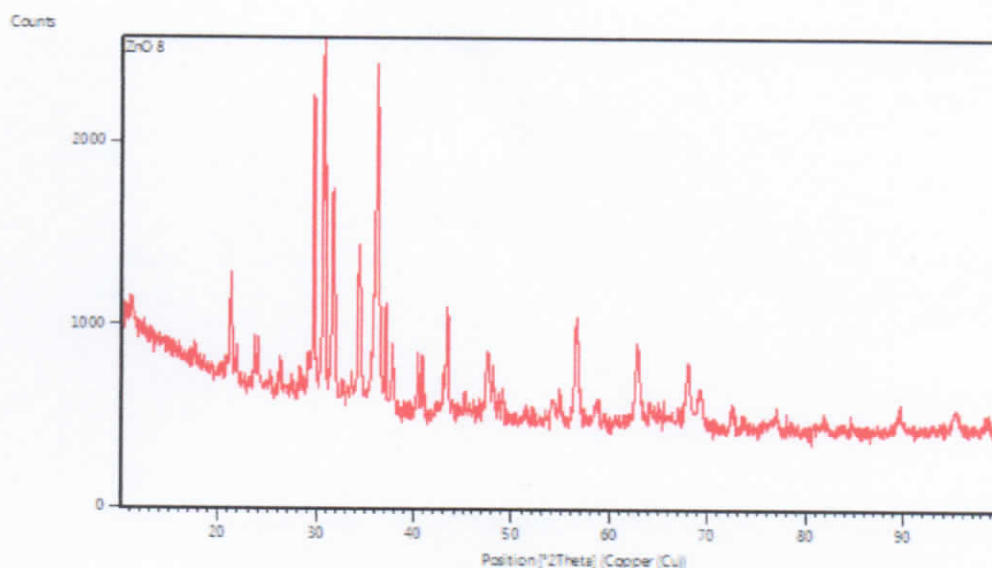


Fig.2. XRD pattern of ZnO nanoparticle synthesized by using urea as additive

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  $2\theta = 31.78$ ;  $34.43$ ;  $36.28$ ;  $47.55$ ;  $56.62$ ;  $62.82$ ;  $67.94$  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 possesses a high crystallinity since all the peaks were 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  $2\theta$  of  $21.32$  and  $30.74$  which have been identified as potassium sulfate (Pdf card No. 00-044-1414). The average crystallite 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 ( $2\theta$ )	Height (counts)	FWHM	d-value [ $\text{\AA}$ ]	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

## 3.2.2. SEM images of ZnO nanoparticles

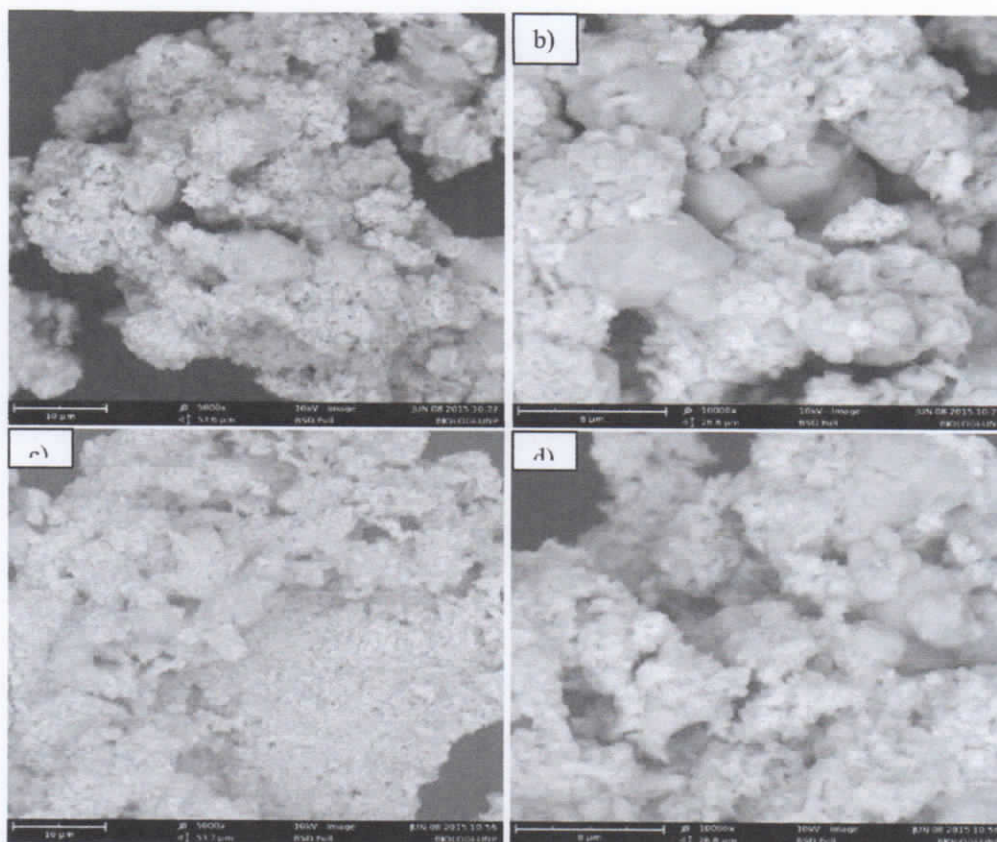


Fig.3. SEM images of ZnO nanoparticles synthesized by using a) and b) ethylene glycol as additive, c) and d) urea as additive

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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STATE UNIVERSITY OF PADANG**

*Certificate*

Is Hereby Given To

**Sherly Kasuma Warda Ningsih**

**As Presenter**

at "The International Conference on Mathematics, Science, Education and Technology"  
in Padang, West Sumatera, Indonesia

22<sup>nd</sup> October 2015



**Prof. Dr. Lufri, M.S.**  
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