IOP Conf. Series: Journal of Physics: Conf. Series **853** (2017) 012041 doi:10.1088/1742-6596/853/1/012041

# Fabrication and characterization of Zinc Oxide (ZnO) nanoparticle by sol-gel method

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**Abstract**. Currently, nanomaterial is an interestingfield of study. This is due to its chemical and physical properties that are superior to that of large-sized materials.One nanomaterial widely studied is zinc oxide (ZnO). In this study, a synthesis of ZnO nanoparticles made by Sol-Gel method was conducted.The process parameters used are variations in pH, in increasing order, of 7; 8; 9; 10; 11; and 12. There are two principal reactions to produce a compound oxide, namely hydrolysis and condensation. NaOH is an agent for the hydrolysis of $(CH_3COO)_2$ Zn resultingin Zn $(OH)_2$. Subsequently, condensation produces ZnO. Calcination was carried out at a temperature of $80^0$C for 1 hour. The ccharacterization of the samples showed that the condition of pH 12 produced the best sample with a size of 73.8 nm and ZnO percentage of 100%. Although pH 7 produced a particle size of 1.3 nm, the percentage of ZnO formed was only 42.9%. The calcination process was performed to remove $CH_3COONa$. However, the process can lead to aggregation of ZnO particles to each other, which increases the particle size.

## 1. Introduction

Nanoscale material is a material which is very attractive because it has properties that are very different than those shown on the macroscopic scale. Various attractive quantum phenomena arise from the downsizing of material into nanoscale dimensions. Platinum bulky metal, known as inert material, can be turned into a catalytic material if its size is reduced to the nanoscale. Stable materials such as aluminum can be flammable and insulating materials turn into a conductor[1]. With nanotechnology, any substance or material will allow for a weight reduction accompanied by increased stability and improved functionality.

There are two methods of synthesis of nanoparticles, namely top down and bottom up. Top down synthesis process does not involve a chemical reaction. The process that occurs is only major material splitting into nanometer-sized material; the obtained new material that has better performance and is different from the original bulk material. Meanwhile, the bottom-up synthesis process involves a chemical reaction of a starting material to produce another material which is nanometer-sized.. Top down methods include mechanical milling, repeated quenching, and lithography, whereas bottom up methods include the sol gel process, aerosol-based process, chemical vapor deposition (CVD), atomic condensation, gas phase condensation, and fluid supercritical synthesis[3].

One of the many synthesized materials into nano-sized particles is Zinc Oxide. Zinc Oxide is a material that is currently widely studied. This is because ZnO exhibit optical, acoustic, and electrical properties allowing it a number of potential applications in the fields of electronics, optoelectronics, and sensors[2].Zinc Oxide is widely applied as an anti-reflection coating material, transparent electrodes in solar cells, gas sensors, varistors, electroluminescence material, and material Photoluminescence. Semiconductor ceramic materials are attractive because of commercial demand for optoelectronics such as transparent conductivity electrode, ultraviolet (UV) light emitters, and electron spin that are semiconductors with a wide energy gaps and binding energies on optical processes. Additionally, Zinc Oxide has several advantages including its stable chemical structure, non-toxicity, and its utility as an additive in a variety of materials, as well as its abundant availability in nature that makes its price cheaper.

From a number of Zinc Oxide nanoparticles synthesis method, the sol-gel method is a method that is relatively simple and works at lower temperature when compared to other methods such as chemical vapor deposition (CVD), hydrothermal, and plasma which require high temperatures in the process of synthesis. The sol-gel method has a principle similar to the solid state reaction. The process involves heating the mixture to stoichiometric reagents to make the ions move so that the desired solid product is obtained.

Materials that are commonly used in the sol-gel process is metal oxide. Furthermore, sonicator is used for mixing, and Zinc Oxide particles break. Zinc Oxide precipitates further heat in order to obtain a powder of nanoparticles of Zinc Oxide. The sol-gel method uses a lot of chemicals so it works on varying levels of acidity (pH) in the solution. The effect of pH contribute to the effects of hydrolysis and condensation during the process of forming a gel and the morphology of the resulting Zinc Oxide [5]. In addition, the pH can change the value of atomic nuclei and the development unit. The result of the synthesis of zinc oxide nanoparticles will be characterized using Particle Size Analyzer for observation of particle size and X-Ray diffraction for composition observation.

## 2. Materials and methods

The materials used in this study include: Zn $(CH_3COOH)_2$ (zinc acetate dihydrate), NaOH (sodium hydroxide), $CH_3OH$ (methanol), and aquabidest. Some $(CH_3COOH)_2Zn.2H_20$ powder as much as 4.39 grams was dissolved in 100 ml of methanol and stirred with 750 watt sonicator for 30 minutes to obtain a homogeneous solution with a concentration of 0.2 M. Separately, 1.0 Mof NaOH were dissolved in 500 ml aquabidest. Furthermore, the optimization of NaOH titration was performed. The results of the titration optimization was used for titration of NaOH into a solutionof $(CH_3COOH)_2Zn.2H_20$, thereby changing the pH value. In order to determine the effect of pH on the synthesis results, the researchers then made a colloid with five variations in pH of 7, 8, 9, 10, 11, and 12.When the solution has formed a milky white, they were then stirred again by using a sonicator for 30 minutes. Then, they were allowed to stand for several days for observation. pH 7 and pH 12 has the fastest time and the longest colloids to settle, and then the results of both were analysed by the PSA. They were then centrifuged to separate the sediment at 3000 rpm for 30 minutes, and thesediment was taken. The precipitate was heated so that $H_2O$ precursors and other compounds can be lost using the oven at 800C for 1 hour. Furthermore, ZnO solids were crushed using a mortar and the powder obtained consist ZnO nanoparticles. Then, ZnO sample was analyzed by the XRD to determine the composition of the sample.

## 3. Results and discussion

The mix $(CH_3COO)_2Zn.2H_20$ and $NaOH.H_20$ used titration. Optimization of titration was used in terms oftime of hatching, temperature, and stirrer speed. The optimization of the time variation of hatching was performed at 0, 1, 20, and 60 seconds. The optimization results show that the longer the time of hatching,the longer the time a solution to settle will be. The optimization of temperature was performed at $0^0$C, $25^0$C, and $50^0$C.The results showed that the longest deposition occurs at room temperature ($25^0$C). The third optimization focused on variations of the magnetic stirrer speeds of 100 rpm, 300 rpm, and 600 rpm. The results showed that the solution takes most time to settle when magnetic thestirrer speed of 600 rpm. Thus titration parameters are optimized to hatching time of 60 seconds, the room temperature $25^0$C, and 600 rpm speedof magnetic stirrer. Then, the titration of

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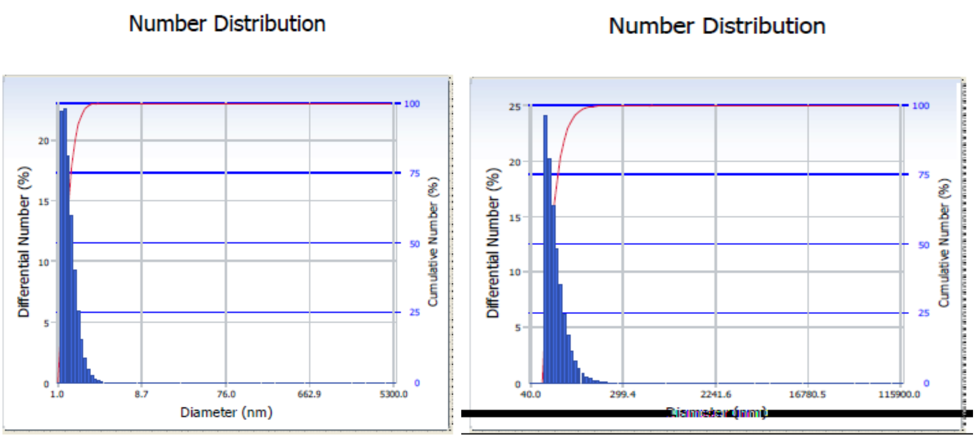
NaOH into 100 ml of $(CH_3COO)_2Zn.2H_20$ was performed manually using a pipette until the pH of the solution turned into 7, 8, 9, 10, 11, and 12. The colloidal ZnO formed was of milky whitecolor. The colloidal ZnO is fastest and most long to settle that at pH 7 and pH 12 (Table 1)

Tabel 1 The relationship between pH with deposition time

| pH | Deposition time (minutes) |
|---|---|
| 7 | 4320 |
| 8 | 1440 |
| 9 | 600 |
| 10 | 480 |
| 11 | 240 |
| 12 | 120 |

Furthermore, the solution has had various pH 7, 8, 9, 10, 11, and 12 centrifuged at 3000 rpm for 30 minutes. Centrifuges were used to separate the precipitate from the solution. The position of each solution was taken and placed in crucible, then calcined in an oven heated to 800C for 1 hour. The calcination of the Zinc Oxide will produce a white blob and then the white blob was crushed using a mortar to produce Zinc Oxide powder. Calcination in this case serves to remove the element contained in the compound, and the compound resulting from shrinking volume.The depreciation of compounds that happen cause re-unification of these elements so that the particles get bigger. Furthermore, ZnO powder were tested using PSA to determine its size and XRD to determine the composition of the compounds contained in the ZnO.

The results showed that among the variations of the deposit of zinc oxide, the one from the solution of pH 12 obtained the fastest settling time, whereas the solution of zinc oxide obtained at pH 7 solution was the slowest to settle. Therefore, we tested the colloidal ZnO of pH 12 and pH 7 using PSA (Figure 1). The average particle size of ZnO of the pH 7solution is 1.3 nm while for the pH 12 solutionit is73.8 nm. The greater the pH of the solution, the less time it takes to settle so that the solution of the resulting particle size increases. Conversely, the smaller the pH of the solution, the longer time it takes to settle and the smaller is its resulting particle size (Figure 2)




(a) (b)

Figure 1. Number distribution of sample at (a) pH 7 and (b) pH 12

The XRD observation results for the sample pH 7, 8, 10, and 12 were shown in Figure 2. XRD observation data were analyzed using two approaches, namely by fitting the data using Microsoft Excel and by the X-powder program. Let us turn first to fitting the data using Microsoft Excel. In this case, the test results of XRD to pH 7, 8, 10, 12 are matched to the database manually with Microsoft Excel, i.e.by matching peaks of database ICDD Pcpdfwin 1998 ZnO with peaks that are indicated by ZnO synthesized at various pH. From the results of Figure 2 it appears that at pH 7 ZnO nanoparticles are still not fully formed, same with pH 8 which also has not formed ZnO and other compounds. At a pH of 10 formation ZnO nanoparticles began, whereas at pH 12 pure ZnO nanoparticles had formed.

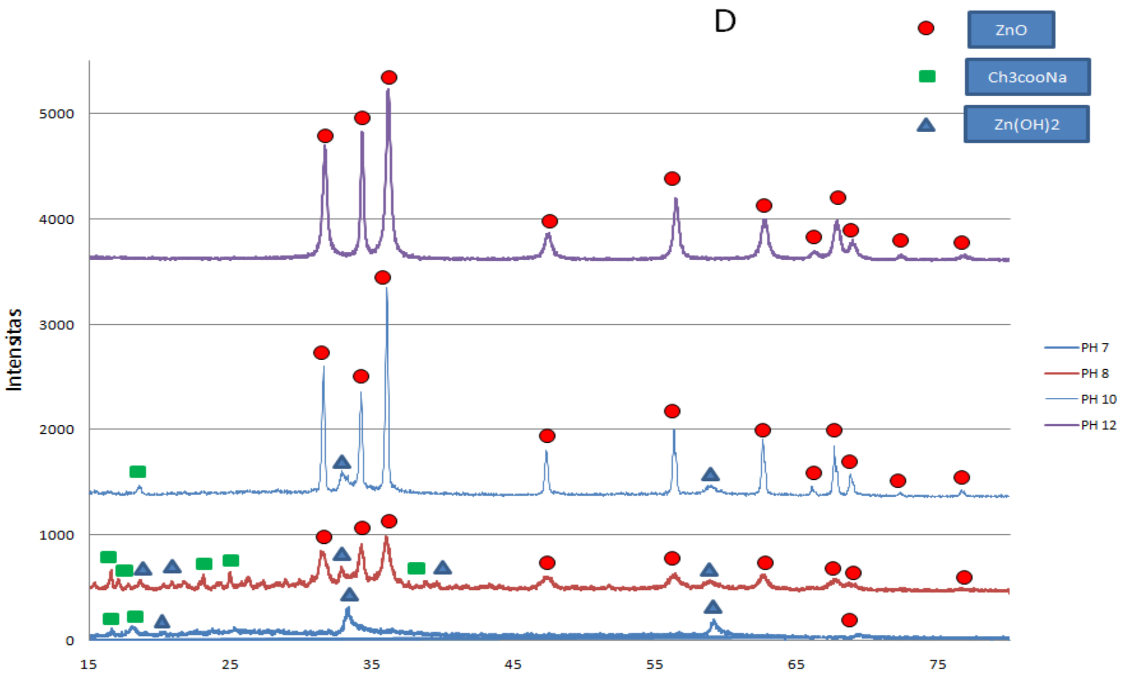



Figure 2. Quantitative analysis of the sample at pH 7, 8, 10, dan 12.

The second approach was performed using the X-powder. The diffraction patterns in the form of test results of the XRD spectra provide information about the diffraction angles (2θ) on the horizontal axis and the intensity generated on the vertical axis. The XRD testing aims to determine the composition of the compounds formed in ZnO. Determining the composition of these compounds is done using a search match with X-powder program. The search results matched the X-powder in a way fitting the data obtained by the percentage weight of the compound of synthesized ZnO at pH 12, 10, 8, and 7 which are shown in Table 2.

Table 2. Influence of pH to the procentage of ZnO

| pH | % ZnO |
|---|---|
| 7 | 42.9 |
| 8 | 62.2 |
| 10 | 64.7 |
| 12 | 100 |

From the XRD observation data, using the program X-powder and Williamson-Hall plot analysis he crystal size can also be determined (Table 3). From table 3, it appears that the greater the pH value, the greater the size of the crystal. Conversely, the smaller the pH value, the smaller the size of the crystalis. This is consistent with the results of the particle sizes of ZnO that were formed. The size of the crystals can cause dilated calcination so that the particle agglomeration will grow back.

Tabel 3. Relation between pH with size of crystal

| pH | Ukuran kristal (nm) |
|---|---|
| 7 | 10.94±0.99 nm |
| 8 | 17.44±5.36 nm |
| 10 | 38.27±2.14 nm |
| 12 | 74.04±41.77 nm |

## 4. Conclusion

One of the factors that influences the size of the diameter of the nanoparticles using thesol-gel method is pH. The greater the pH of the sol-gel process, the greater the agglomeration so that the particle size gets bigger and vice-versa. The ZnO particle size characterization of the results obtained by the PSA at pH 7 and pH 12 was 1.3 nm and 73.8 nm. The results of XRD analysis with X-Powder program obtained the degree of purity of ZnO produced at pH 7, pH 8, pH 10 and pH 12 at42.9%, 62.2%, 64.7%, and 100%, respectively. The study posits that using sol-gel method is the best conducted ata pH of 12 which resulted in a particle size of 73.8 nm with a composition of 100% ZnO.

## 5. References

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