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Synthesis of Nano Zinc Oxide from Galvanized Industrial Waste by Using a Combination Method of SOL-Gel, Sonochemical and Calcination

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Abstract

Nano zinc oxide can be used as catalysts, semiconductors and antimicrobial, and can be applied in the various fields, such as cosmetics, medicines, and packaging. Nano zinc oxide can be produced through synthesis process using zinc acetate as a precursor. Until now, zinc acetate used in Indonesia has to be imported from other countries with a relatively high price. This study aims to obtain nano zinc oxide by using zinc acetate of galvanized industrial waste (zinc dross). The synthesis process of nano zinc oxide was conducted using a combination method consisting of sol-gel process - sonochemical – calcination with variables of ultrasonication time (30 minutes, 45 minutes and 75 minutes) and pH (pH 8, pH 10 and pH 12). Nano zinc oxides produced under these conditions were characterized based on crystal form, as well as the morphology and particle size. The results showed that the best process conditions for the synthesis of nano zinc oxide was identified at aultrasonication time of 60 minutes and a pH of 10. The produced nano zinc oxide has a crystal size of 45.35 nm, more homogeneous morphology. The nano zinc oxide has a particle size of about 50 nm with a zinc oxide content of 87.31 %.

***Keywords**:* nano particle synthesis process; nano zinc oxide; zinc acetate; ultrasonication; calcination; galvanized industrial waste.

1. Introduction

Nanotechnology covers activities that create, manipulate and compose materials with nanometer sizes [1]. The size reduction of materials up to nanometer scale causes nanoscale materials having a different character from the nature of the original material. As results of the size reduction, nanoparticles have a larger surface area per unit volume and therefor they are more reactive than such kind of micro-sized particles. On the nanometer particle size, the applicable laws of physics are dominated by the laws of quantum physics [2]. With these characteristics, nanoparticles have various advantages such structured and controlled arrangement of atoms or molecules, so that the structure and characteristics of the materials can be designed in accordance with the needs, and the utilization of material will be efficient and optimum [3]. Nano science and technology has developed very rapidly. Researches on the development of nanotechnology were recently conducted in various fields, such as food, pharmacy, environmental protection, agriculture, and packaging industries.

A prospective raw material for nanoparticles production are zinc oxide due to its properties as photocatalisator [4], semi-conductor [5], antimicrobial [6], sunscreen [7], antibacterial [8]. Nano zinc oxide is effective as an antimicrobial; the production price is much cheaper than the nano silver [9].

Synthesis of zinc oxide nanoparticles is currently using raw materials such as chemical compounds,ie zinc nitrate, zinc sulfate or zinc acetate. The chemicals are at the moment still imported into Indonesia with a very expensive price. A potential source of raw materials containing high zinc is galvanized industrial waste in the form of zinc dross. Zinc dross contains zinc up to 90% - 98% [10].

Hydrometallurgy process can be used for the utilization of zinc dross. In the process zinc dross reacts with sulfuric acid or hydrochloric acid to form zinc sulfate or zinc chloride. Zinc dross can also react with sodium hydroxide or sodium carbonate to produce zinc hydroxide or zinc carbonate [11]. Synthesis of nanoparticles can in principle be categorized into two methods, namely physical and chemical methods. In the physical method, larger sized material is broken down physically into nanometer-sized materials, whereas the chemical formation of nanoparticles process involves chemical reactions. Sol-gel and sonochemical methods are included in the chemical synthesis of nanoparticles. Sol-gel process is a process of formation of inorganic compounds through chemical reactions in solution. Advantages of the sol-gel method are a good degree of thermal stability, high mechanical stability, and good resistance solvent [12].

Sonochemical a material synthesis method using ultrasonic frequency to create the airwaves in a liquid that then the wave will break down in a short span of time. These pieces of airwave will give pressure and chemical effects [13].Several studies have been conducted in the synthesis of nano zinc oxide, such as zinc acetate synthesis using sonochemical method [14], synthesis of zinc nitrate and urea using precipitation method [15], synthesis of nano zinc oxide using zinc carbonate as a precursor [16], synthesis of nano zinc oxide using a hydrothermal process [17], synthesis of nano zinc oxide with optoelectronic [18], and synthesis of nano zinc oxide by using solvothermal method [19].

In this research, a bench-scale experiment was initiated to investigate the feasibility of synthesis of nano zinc oxide from galvanized industrial waste using the combination method of sol-gel, sonochemical and calcination.

2. Method

2.1. Materials and Equipment

Materials used in this study included zinc acetate of galvanized industrial waste, distilled water, sodium hydroxide (Merck), methanol (Merck) and chemicals for analysis. The instruments and equipmentsware used in this study involving Sonicator CT Chromtech sound enclosure, Memmert ovens, furnaces Yamato muffle furnace fp 32, electric heaters selectamultimatic 5 N, magnetic stirrer magsuda SN 60 N, glassware, thermometers, analytical balance Sartorius BSA 2245-cw, gooch filter, pH meter Horiba D 52, stopwatch seiko, and saucer. Scanning Electron Microscope (SEM) Philips type SEM 515, Transmission Electron Microscope (TEM) JEOL JEM 1400, X-ray Diffraction (XRD) GPC type Emma, Particle Size Analysis (PSA) Vasco.

2.2. Method

Synthesis Process of Nano Zinc Oxide: The synthesis process of nanoparticle is aimed to obtain monodispersion of the controlled nanoparticles in a variety of small sizes (in nanometer) [2]. The chemical synthesis process of nanoparticles was conducted in two types of process, namely sol-gel and sonochemical processes. Ultrasonication process is nanoparticle synthesis procedure which uses ultrasonic energy. Ultrasonic wave was transmitted through a media by using wave pressure from the inducement of molecule vibration movement, resulting in the pressure and chemical effects. In general, the frequency of ultrasonic wave was in 20 kHz – 10 mHz, and comprised of three different sections, covering low ultrasonic frequency (20 - 100 kHz), medium ultrasonic frequency (100 kHz – 2 mHz) and high ultrasonic frequency (2 – 10 mHz). The frequency type used in ultrasonication process was in 20 kHz – 2 mHz. Sol-gel process was realized to form inorganic compound through chemical reaction of a certain solution. The benefits of using sol-gel method are that it generates a good rate of thermal stability, high mechanical stability, good solution resistance, and possibility to stimulate transformation[12].

Ten grams of zinc acetate was dissolved in 100 ml of methanol, stirred for 30 minutes, then filtered. Zinc acetate solution was then sonicated for 30 minutes. Zinc acetate solution was added with 10% sodium hydroxide solution until the desired pH value, ie pH 8, pH 10, pH 12. The solution was then sonicated again for 15 minutes, 30 minutes or 45 minutes. The solution was precipitated for 24 hours, the sediment was the separated from the fluid phase. The precipitate was dried at 100 °C oven for 8 hours, and then followed by calcination at a temperature of 800^{o}C for 3 hours.Figure 1 shows the flow diagram of the synthesis process of nano zinc oxide from galvanized industrial waste

Characterization of Nano Zinc Oxide: Particle size of the synthesized zinc oxide particle size was analyzed using Particle size analysis (PSA), and zinc oxide crystals were characterized by using X-Ray Diffraction (XRD). Scanning electron microscope was used to characterize the surface morphology of the nano zinc oxide. The structure and size of the nanoparticles were characterized by using Transmission Electron Microscope (TEM). The specifications and the producers of the instruments for the physical analysis of nano zinc oxide are presented in Sub-Chapter 2.1.

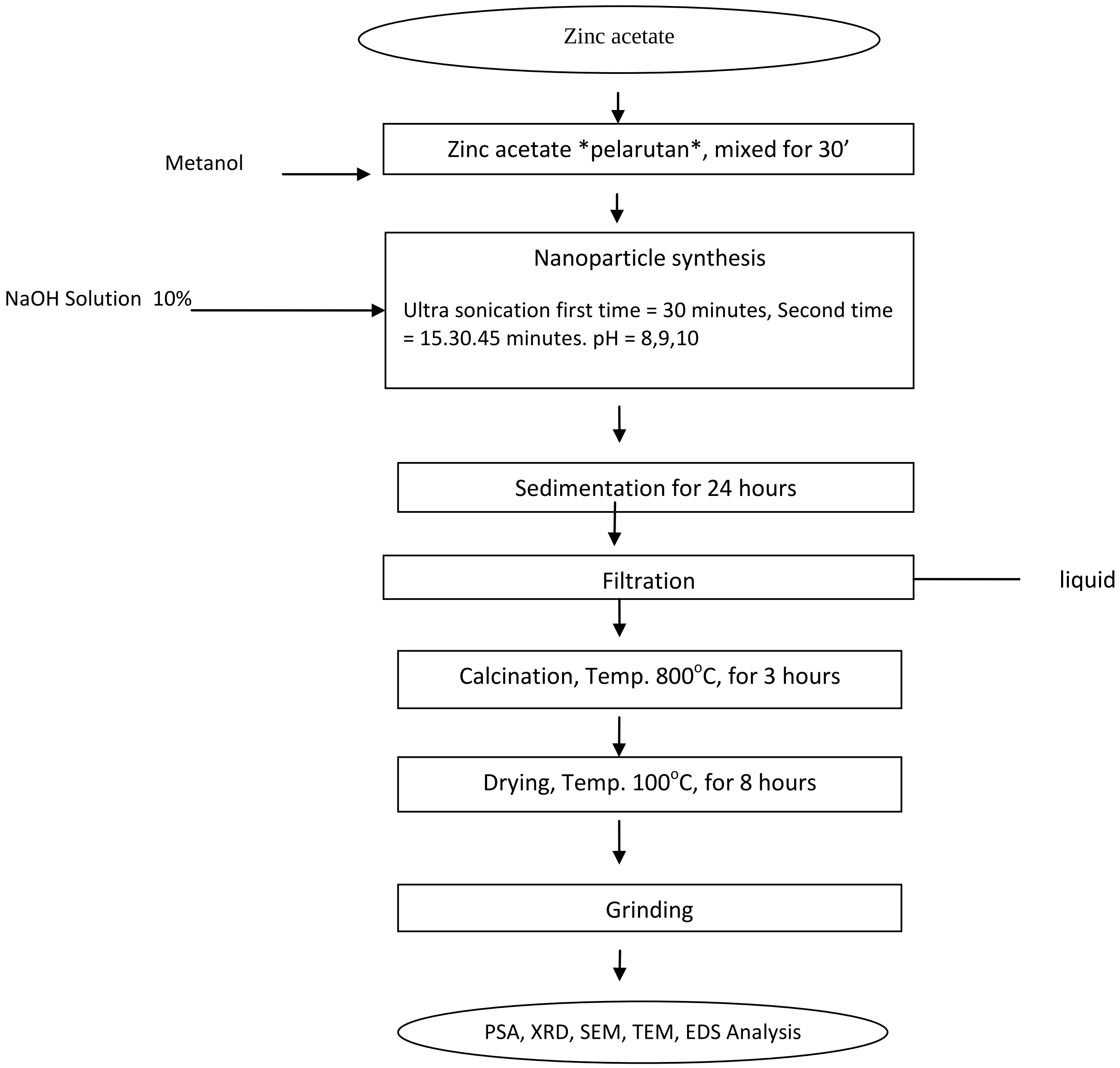


Figure 1: Flow diagram ofthe synthesis process of nano zinc oxide from galvanized industrial waste.

3. Results and discussion

3.1. Synthesis of Nano Zinc Oxide from Galvanized Industrial Waste

The synthesis process of nano zinc oxide in this study used combination processes between sol-gel-sonochemical and calcinations process. Sol-gel process produced some amounts of insoluble zinc hydroxide, while the sonochemical process converted it to nanoparticles of zinc oxide. During the sonochemical process, it involved two-time ultrasonication processes in which methanol was added in order to reduce the size of zinc particles into zinc acetate. Natrium hydroxide was also added for decreasing the zinc size as zinc hydroxide. Last lyit was followed by calcination process for creating nano particles zinc oxide. Zinc acetate used in this study was zinc acetate extracted from zinc dross waste and acetate acid. Acetate zinc produced from this process contained 76.38% of zinc and was able to dissolve in methanol solution.

The reactions involved in the synthesis process are as follows:

$$Zn\,(CH_3COO)_2 + 2\,CH_3OH \longrightarrow ZnOH + 2\,CH_3COOCH_3$$

$$ZnOH + 2NaOH \longrightarrow ZnO + Na_2O + 2H_2O$$

The reaction process of zinc acetate and sodium hydroxide was to create zinc hydroxide with the influences of some variables of pH condition, including pH 8, pH 10 and pH 12. This process showed that pH value resulted in different length of sedimentation time because of the solubility of the sodium hydroxide. In order to get complete sedimentation, the sedimentation process was conducted for about 24 hours. Besides, sonochemical process involved some time variables, namely 45 minutes, 60 minutes, and 75 minutes. The process of ultrasonication showed that the longer time of ultrasonication was applied, the hotter the result was. It occurs because ultrasonication process is an exothermic process, where the calor is produced from the particle collision. Calcination process in 600^{o}C resulted in higher content of carbon, in which the carbon is in form of black-colored sediment obtained from acetate substance as the raw material used in this process. If the process was conducted in 700^{o}C, the product contained less carbon.Further, the process temperature of 800^{o}C resulted in no carbon in the product, making its white-colored sediment.

3.2. Crystal Structure of the Zinc Oxide

Crystal is a form of compact or solid compound which contains atomic molecules, or composed of a certain pattern of ions. The occurrence of crystal was influenced by the speed of crystallization process, supersaturation degrees, temperature, pH control, impurities or other compound addition, types of solvent and stirring process [20]. Furthermore the overall characteristic of a crystal is prominently seen from its size and form [21].

Nanoparticles crystals of zinc oxide could be produced using zinc acetate derived from galvanized industrial waste. These zinc oxide crystals were analyzed by using X-Ray Diffraction (XRD). Figure 3 shows the haracteristics of zinc oxide crystal from process at aultrasunication time of 60 minutes and various pH values, pH 8, pH 10, and pH 12.

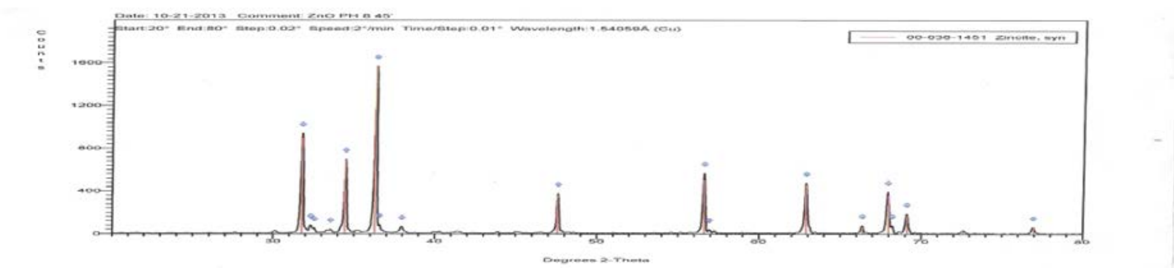
Synthesis of nano zinc oxide at pH 8, pH 10 and pH 12 resulted in crystals with different degrees of crystallinity. The synthesis process at pH 8 produced crystal with a crystallinity degree of 63.59, and the process at pH 10 and pH 12 resulted in a crystallinity degree of 84.65 and 76.95, respectively. In addition, the pH factor also affected the size of the crystals formed. The crystals size of the synthesis processes at pH 8, pH 1o and pH 12 were 45.63 nm, 47.02 nm, and 47.5 nm, respectively. From these results can be drawn a conclusion that the higher the pH, the size of the produced crystals will be larger.

The nanoparticles crystals of zinc oxide are in 2-theta degrees, specifically in $31^{o}$to $77\ ^{o}$. In the synthesis process of nano zinc oxide using pH variables - pH 8, pH10 and pH 12, the resulted crystals had different degrees of crystallization. It showed 63.59 degrees of crystallization for pH 8, 84. 65 degrees of crystallization for pH 10, and 76.95 degrees of crystallization for pH 12. In addition to these, pH variables also influenced the size of the formed crystals. The crystal size produced from pH 8 was in 45.63 nm, from pH 10 was 47.02 nm, and from pH 12 was 47.5 nm. These data made clear that the higher pH condition produced the larger particles.

Synthesis of nano zinc oxide at pH 8, pH 10 and pH 12 resulted in crystals with different degrees of crystallinity. The synthesis process at pH 8 produced crystal with a crystallinity degree of 63.59, and the process at pH 10 and pH 12 resulted in a crystallinity degree of 84.65 and 76.95, respectively. In addition, the pH factor also affected the size of the crystals formed. The crystals size of the synthesis processes at pH 8, pH 10 and pH 12 were 45.63 nm, 47.02 nm, and 47.5 nm, respectively. From these results can be drawn a conclusion that the higher the pH, the size of the produced crystals will be larger.

The synthesis process of zinc oxide nanoparticles was also influenced by ultrasonication time. Figure 3 shows the results of the zinc oxide nanoparticles characterization by X-Ray Diffraction (XRD). Crystalline zinc oxide nanoparticles have degrees 2 theta between 31^{o} - 77^{o}. Process with ultrasonicationtimes of 45 minutes, 60 minutes and 75 minutes resulted in the crystallization degrees of 65.39, 74.18, and of 76.95, respectively. In addition, research showed that ultrasonication time also affected the size of the crystal. The ultrasonication times of 45 minutes, 60 menutes and 60 menutes produced crystal sizesof 44.53 nm, 45.35 nm and 47.5 nm, respectively.

(a)



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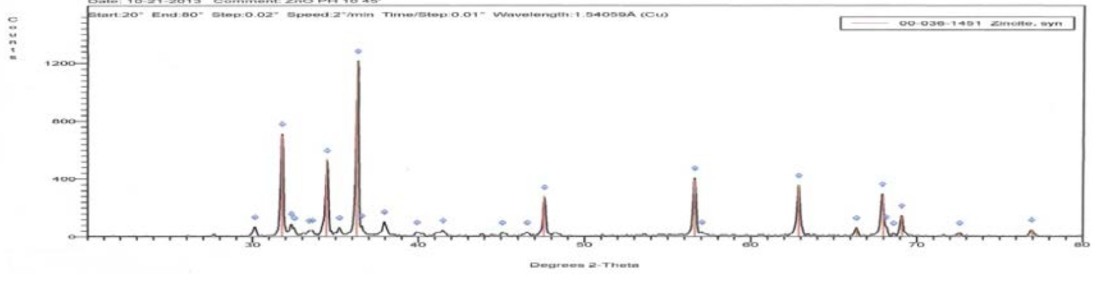

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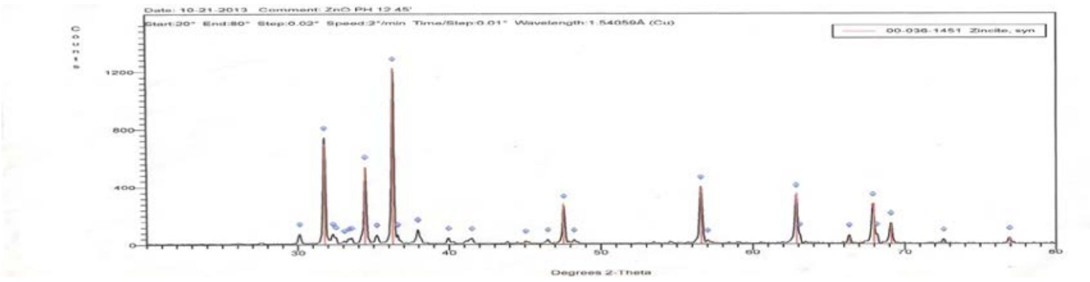
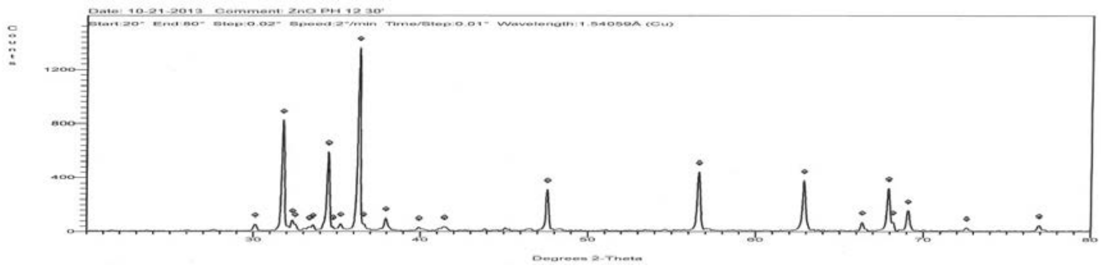


Figure 2: Characteristics of zinc oxide crystal from process at aultrasunication time of 60 minutes and various pH values, pH 8 (a), pH 10 (b), and pH 12 (c)

(a)



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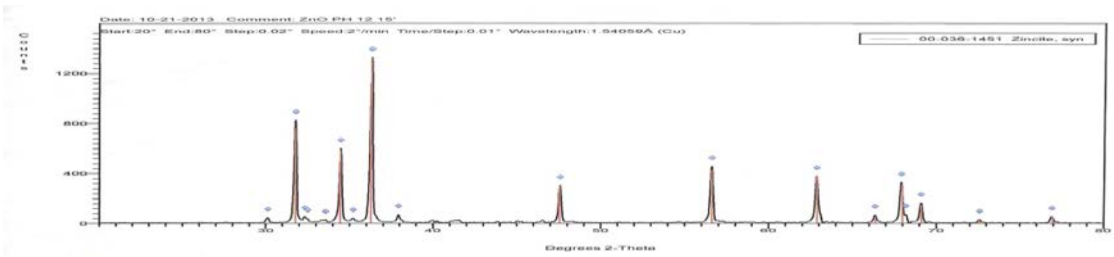


(c)

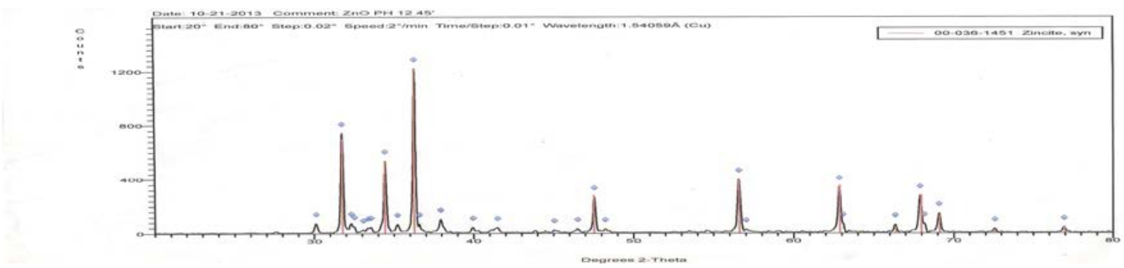
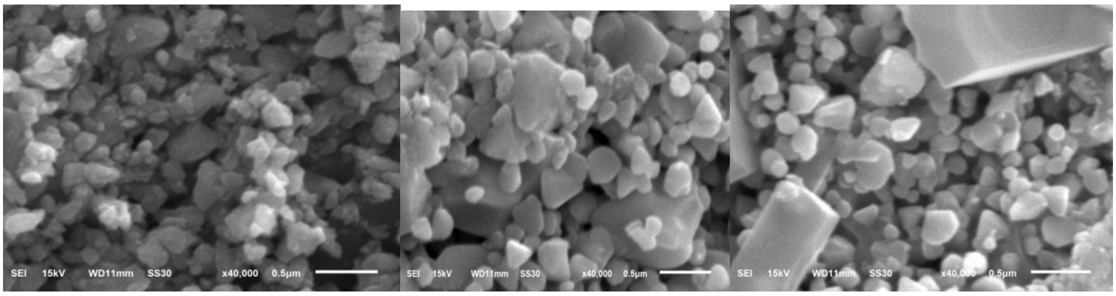


Figure 3: Characteristics of zinc oxide crystal from process at pH 12and various ultrasonicationtime of 45 minutes (a), 60 minutes (b),and 75 minutes (c)

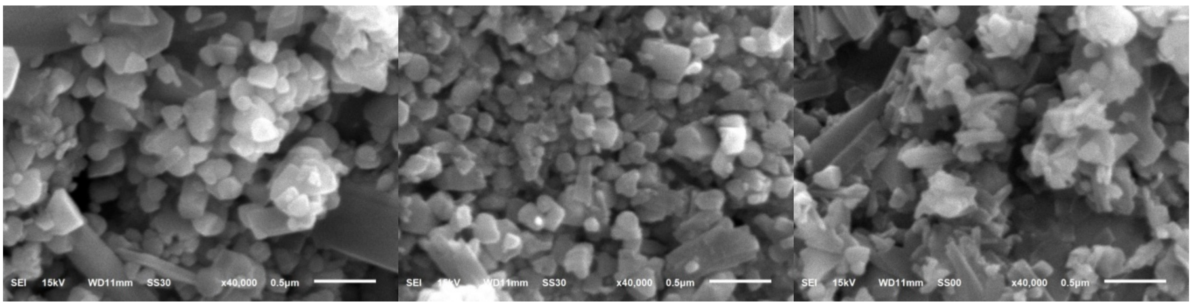
3.3. Morphological Characterization of Nanoparticles of Zinc Oxide

The elements making up the material can be determined by observing the morphology structure using a Scanning Electron Microscope (SEM). In general, homogeneity and regularity of nanomaterials is expected because these properties determine the optimal characteristics and stable in its application [22]. Figure 4 shows the analysis of zinc oxide nanoparticles with SEM at a magnification level of 40,000 times.

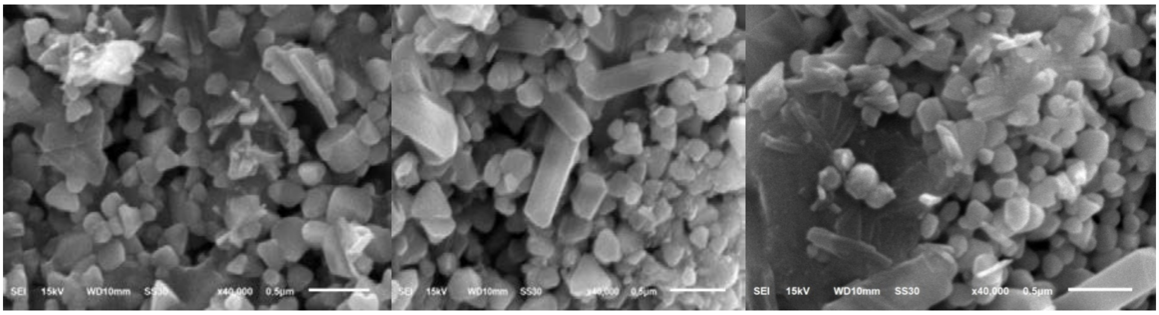
Based on the Figures 4, it is clear that process at pH 8 and ultrasonication of 45 menutes produced nanoparticles of zinc oxide in relatively homogenous form and size. In the 60-minute ultrasonication process natrium and carbon were observed in the nanoparticle. In the 75-minute ultrasonication the number of produced natrium was higher than in 60-minute process. The natrium was also involved in agglomeration process with nanoparticles of zinc oxide. Homogenous form and size of nanoparticles of zinc oxide also occured in pH 10, especially in 60-minute ultrasonication, while 4-minute ultrasonication produces natrium and 75-minute ultrasonication produces carbon and natrium which were involved in agglomeration process with nanoparticles of zinc oxide. The process at pH 12 produced homogenous form and size of nanoparticles of zinc oxide. The amount of produced particles here were less than in pH 8 and pH 10. The other involved compound materials, like sodium and carbon in pH 12 were more than in pH 8 and pH 10. The 75-minute ultrasonication produced less nanoparticles of zinc oxide than 45-minute and 60-minutes ultrasonication processes. In 75-minute nanoparticles of zinc oxide synthesis, the temperature increased.The temperature increase was higher with the longer ultrasonication time. This resulted in the agglomeration or the particles affiliation during the process



(a) pH 8, 45 minutes pH 8, 60 minutes pH 8, 75 minutes



(b) pH 10, 45 minutes pH 10, 60 minutes pH 10,75 minutes



(c)pH 12, 45 minutes pH 12, 60 minutes pH 12, 75 minutes

Figure 4: Morphology of zinc oxide nanoparticles from process at various ultrasonication times and pH values

3.4. Particle Size of Nano Zinc Oxide

Particle size measurement was done by using the Particle Size analyzer (PSA). In this particle size analysis of zinc oxide nanoparticles are dispersed into the liquid medium. The particle size distribution includes intensity, volume and number, so it can be assumed to describe the overall condition of the sample.Figure 5 shows the size of the resulting nanoparticles of zinc oxide.According to Figure 5, it is evident that higher pH produced bigger-sized particles, resulting in shorter sedimentation time. The 60-minute ultrasonicationin pH 8 produced particles in the size of 302.78 nm. Considering the length of ultrasonication time in 45 minutes and 75 minutes, the number of particles decreased. The particle size of 370.96 nm was obtained in pH 10 and 45-minute ultrasonication time, 281.78 nm in 60-minute ultrasonication time. Those bigger size of 360.90 nm was obtained in 75-minute ultrasonication time and pH 10. The PSA measurement showed that the nanoparticles of zinc oxide was considerably large, which was > 100nm. It happened because of the agglomeration between the nanoparticles of zinc oxide and dispersal methanol.

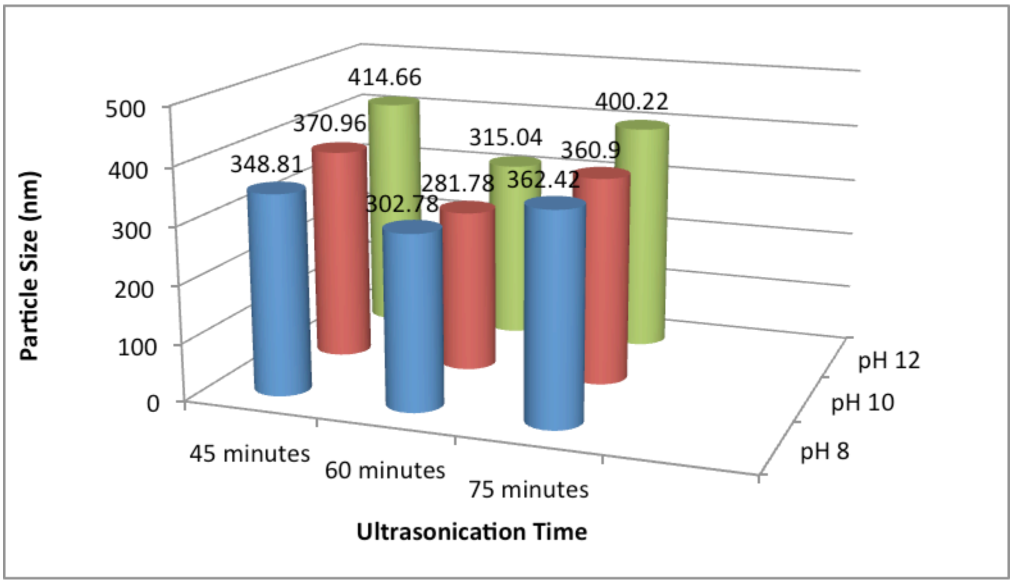


Figure 5: Particle sizes of zinc oxide from various ultrasonication times and pH values

The size of single particle can be analyzed by using *Transmision Electron Microscope (TEM)*. TEM measure accurately the particle size in nanometer scales because of its high resolution (0.1- 0.2 nm) in which the length of de Broglie wave in the electrons (9×10^{-12} m) can penetrate through fragments, and it results on the transformation of data into pictures. Based on the Transmission Electron Microscope (TEM) analysis, the nanoparticles of zinc oxide can be divided into 3 different size categories, namely 50 nm, 100 nm and 200 nm (Figure 6). The figure shows that the nanoparticles of zinc oxide are mostly in size of 50 nm, shown by the measurement of a single particle, and the formed nanoparticles was in form of nanorods. The figure is also displaying 100 nm-sized particles showing the occurrence of agglomeration in some particles.The 200 nm-sized particles also involved in agglomeration process with sodium and carbon. The length of ultrasonication time determined the size of zinc oxide nanoparticles. The figure also describes that 45-minute ultrasonication time produced 50 nm-sized nanoparticles of zinc oxide. There were some impurities which allowed the interparticle agglomeration. The 60-minute ultrasonication time produced 50 nm-sized particles, but the particles were cleaner in which there were no impurities as occurred in 45-minute ultrasonication, so that it resulted in more stable from of nanoparticles. The 50 nm-sized particles produced from 75-minute ultrasonication were somehow also involved in interparticle agglomeration as discussed above, the method used in synthesis process of nanoparticles of zinc oxide determined the size of nanoparticles. Similar results were reported by [23],[24] and [25].

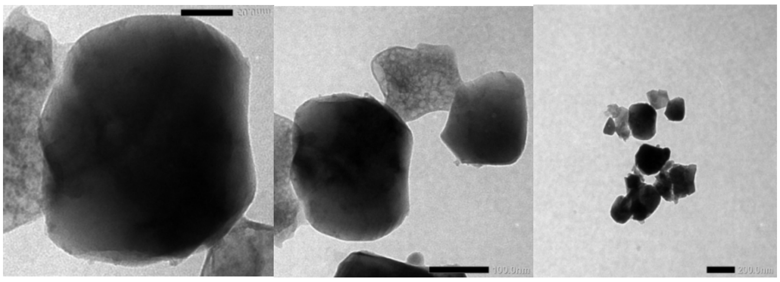


Figure6: Morphological structures of zinc oxide nanoparticles from process at pH 10 and ultrasonication of 60 minute, particle sizes of 50 nm (a), 100 nm (b), and 200 nm (c)

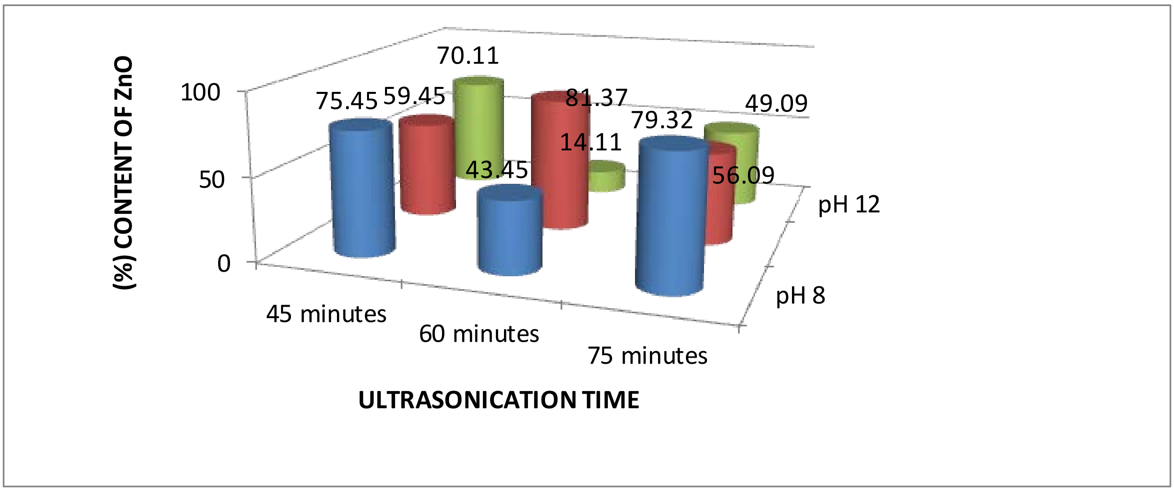
3.5. Composition of Zinc Oxide Products

In the synthesis process of nanoparticles using sol-gel method, there are some stages in the process of mixing up the zinc acetate and methanol. In this stage, there is a compound formation in forms of colloid because methanol is the substance that can lessen the number of α-hydrogen atoms. Adding sodium hydroxide solution increase the value of pH and insoluble zinc oxide is formed. It will result in the production of zinc oxide particles.

In sonochemical method ultrasonication time effecte on particle size. The particle size then affected the particle settling velocity. Furthermore, the calcination process eliminated the organic substances and improved the stability of zinc oxide. The calcination process of organic substances formed carbon compounds. Calcination at a temperature of 600 ^oC showed the presence of carbon compounds, which was marked by the color black zinc oxide compound. Calcination at a temperature of 700 °C resulted in relatively fewer carbon compound, which was marked black less on zinc oxide compound. Calcination at a temperature of 800 ^oC left no carbon compound anymore, which was marked in white zinc oxide compound.

The analysis using *Electron Dispersive Spectroscopy* (EDS) showed that there were some compound compositions in forms of zinc oxide, sodium oxide, and carbon. The results of the analysis are presented in Figure 7a. The highest nanoparticles of ZnO was observed in the process at pH 10 and 60-minute ultrasonication time, in which the nanoparticle content was 81.73% of ZnO. The content of sodium oxide compound is described in Figure 7b.The highest content of sodium oxide was obtained from the process at pH 8 and 60-minute ultrasonication time, namely 45.48%. There were left some amounts of sodium dioxide because this sodium composite was not fully dissolved during the process of pH adjustment. The content of carbonic compound in the nanoparticles of zinc oxide is depicted in Figure 7c. The highest carbonic compound in a particular process condition, specifically in pH 12 and 75-minute ultrasonication, reached 14.09%. The carbonic compound occured in the other involved substances because during the synthesis process of nano zinc oxide, the carbons from the zinc acetate was not fully dissolved in which there were some left over sedimentation of zinc oxide.

(a)



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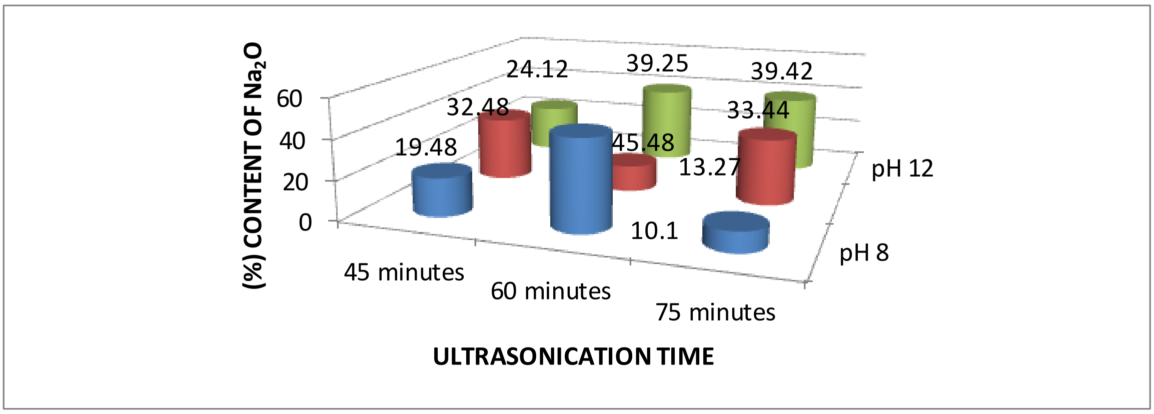

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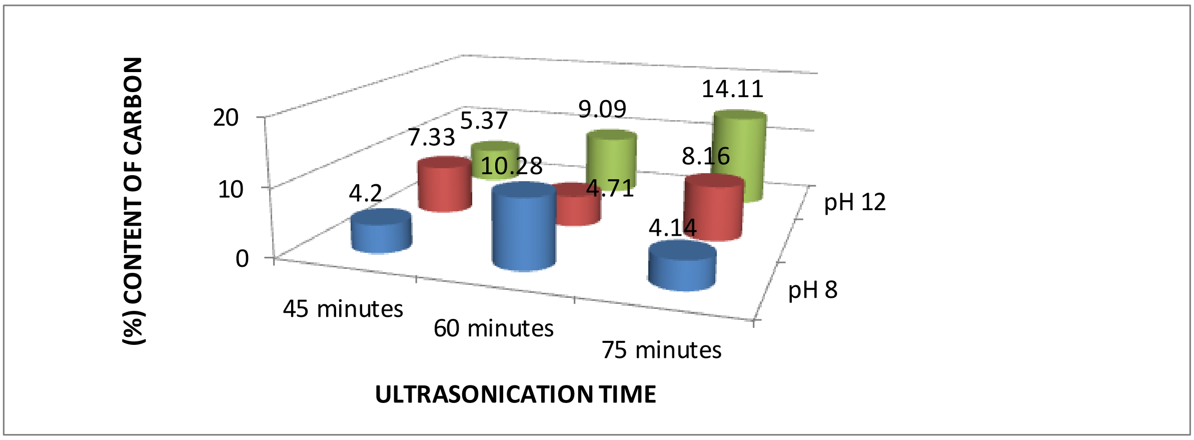

Figure 7: Content of zinc oxide, sodium dioxide, and carbon in the nanoparticle zinc oxide, zinc oxide (a), sodium dioxide (b), and carbon (c)

4. Conclusion

Zinc acetate from galvanized industrial waste can be used as a precursor in the synthesis of zinc oxide nanoparticles by using a combination of sol-gel method, sonochemical and calcination. The optimum process has been identified at pH 10, ultrasonication time of 60 minutes and calcination at a temperature of 800 ^{o}C for 3 hours. Nano zinc oxide produced has a crystal size of 45.35 nm, uniform and homogeneous morphology, nanorod shape with an average particle size of 50 nm. The zinc oxide compound content was 81.73%.

References

[1] H. Joseph, "Polymer Nanocomposites–Processing, Characterizations and Applications," ed: The McGraw-Hill, New York, 2006.

[2] M. Abdullah, Y. Virgus, N. Nirmin, and K. Khairurrijal, "Review: Sintesis Nanomaterial," *Jurnal Nanosains & Nanoteknologi*, vol. 1, pp. 33-57, 2009.

[3] C. P. Poole Jr and F. J. Owens, *Introduction to nanotechnology*: John Wiley & Sons, 2003.

[4] G. Patrinoiu, J. M. Calderón-Moreno, D. C. Culita, R. Birjega, R. Ene, and O. Carp, "Eco-friendly synthetic route for layered zinc compound and its conversion to ZnO with photocatalytical properties," *Solid State Sciences*, vol. 23, pp. 58-64, 2013.

[5] Nuryetti, H. Hermansyah, and M. Nasikin, "Bionanokomposit: Peluang Polimer Alami Sebagai Material Baru Semikonduktor," *Jurnal Riset Industri Online*, vol. 6, 2012.

[6] C. L. Wilson, *Intelligent and active packaging for fruits and vegetables*: CRC Press, 2007.

[7] Y. Cao, H.-J. Wang, C. Cao, Y.-Y. Sun, L. Yang, and Y.-N. Zhang, "Synthesis and anti-ultraviolet properties of monodisperse BSA-conjugated zinc oxide nanoparticles," *Materials Letters,* vol. 65, pp. 340-342, 2011.

[8] T. Gordon, B. Perlstein, O. Houbara, I. Felner, E. Banin, and S. Margel, "Synthesis and characterization of zinc/iron oxide composite nanoparticles and their antibacterial properties," *Colloids and Surfaces A: Physicochemical and Engineering Aspects,* vol. 374, pp. 1-8, 2011.

[9] F. G. Winarno and I. E. Fernandez, *Nanotechnology bagi industri pangan dan kemasan*. Bogor: M-BRIO press, 2009.

[10] P. Baláž, "Mechanochemistry and Nanoscience," in *Mechanochemistry in Nanoscience and Minerals Engineering,* ed: Springer, 2008, pp. 1-102.

[11] S. R. Rao, *Resource recovery and recycling from metallurgical wastes* vol. 7: Elsevier, 2011.

[12] L. Theodore, *Nanotechnology: basic calculations for engineers and scientists*: John Wiley & Sons, 2006.

[13] A. Gedanken and Y. Mastai, "Sonochemistry and other novel methods developed for the synthesis of nanoparticles," *The Chemistry of Nanomaterials: Synthesis, Properties and Applications,* pp. 113-169, 2005.

[14] P. Banerjee, S. Chakrabarti, S. Maitra, and B. K. Dutta, "Zinc oxide nano-particles–sonochemical synthesis, characterization and application for photo-remediation of heavy metal," *Ultrasonics sonochemistry,* vol. 19, pp. 85-93, 2012.

[15] Y. Liu, Z. Jian-er, A. Larbot, and M. Persin, "Preparation and characterization of nano-zinc oxide," *Journal of Materials Processing Technology,* vol. 189, pp. 379-383, 2007.

[16] H. Song, H. Yang, and X. Ma, "A comparative study of porous ZnO nanostructures synthesized from different zinc salts as gas sensor materials," *Journal of Alloys and Compounds,* vol. 578, pp. 272-278, 2013.

[17] N. Ueno, T. Maruo, N. Nishiyama, Y. Egashira, and K. Ueyama, "Low-temperature synthesis of ZnO nanorods using a seed layer of zinc acetate/sodium dodecyle sulfate nanocomposite," *Materials Letters,* vol. 64, pp. 513-515, 2010.

[18] T. Bellezze, S. Saltykov, G. Roventi, M. Malavolta, and R. Fratesi, "Layer-by-layer analysis of hot-dip zinc coating by anodic dissolution in acetate bath," *Surface and Coatings Technology,* vol. 206, pp. 5023-5027, 2012.

[19] P. Tonto, O. Mekasuwandumrong, S. Phatanasri, V. Pavarajarn, and P. Praserthdam, "Preparation of ZnO nanorod by solvothermal reaction of zinc acetate in various alcohols," *Ceramics International,* vol. 34, pp. 57-62, 2008.

[20] A. R. Fachry and J. Tumanggor, "Pengaruh Waktu Kristalisasi Dengan Proses Pendinginan Terhadap Pertumbuhan Kristal Amonium Sulfat Dari Larutannya," *Jurnal Teknik Kimia*, vol. 15, 2008.

[21] K. Sugiyarto and R. D. Suyanti, "Kimia Anorganik Logam," *Yogyakarta: Graha Ilmu*, 2010.

[22] S. Nikmatin, "Biokomposit filler nanopartikel serat kulit rotan sebagai material pengganti komposit sintetis fiber glass pada komponen kendaraan bermotor," 2012.

[23] S. Das, K. Bhattacharjee, S. Maitra, and G. Das, "Effect of oxygen partial pressure on the photoluminescence properties of sol–gel synthesized nano-structured ZnO thin films," *Thin Solid Films*, vol. 550, pp. 65-70, 2014.

[24] M. Z. Ahmad, A. Z. Sadek, K. Latham, J. Kita, R. Moos, and W. Wlodarski, "Chemically synthesized one-dimensional zinc oxide nanorods for ethanol sensing," *Sensors and Actuators B: Chemical*, vol. 187, pp. 295-300, 2013.

[25] A. S. Lanje, S. J. Sharma, R. S. Ningthoujam, J.-S. Ahn, and R. B. Pode, "Low temperature dielectric studies of zinc oxide (ZnO) nanoparticles prepared by precipitation method," *Advanced Powder Technology*, vol. 24, pp. 331-335, 2013.