The Synthesis of Photocatalyst Material ZnO using the Simple Sonication Method

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Abstract. ZnO is well known as photocatalyst material therefore potentially to applied in many purposes. The particle size of photocatalyst material influenced the catalytic activities. In this research, ZnO was synthesized using the simple sonication method to obtain the the smaller particle with sonication time variation respectively: 30, 60, 160, 360 minute. X-ray diffraction data showed that the synthesized material have wurtzite structure with space group $P\overline{6}3$mc. The synthesized ZnO with 30 minutes sonication time produced the smallest particle size and have the lowest band gap energy (2.79 eV). The photocatalytic test at methylene blue also showed that the optimum activity was gained from ZnO which synthesized at 30 minute sonication time (degradation percentage of methylene blue is 77.93%).

1. Introduction
The dye pollutant from textile industries have become environmentally problem. Photocatalysis is one of technology have potentially to degrade the dye pollutant so it can applied to solve the dye pollutant problem. Photocatalysis is a process of chemical reaction which involving photocatalyst material and light as initial reaction [1]. ZnO is one of material that used for photocatalyst material and have some advantages such as high photocatalyst activity with a band gap 3.20-3.37 eV, cheap, more stable structure, and non-toxic [2].

The particle size of photocatalyst material influenced to the photocatalytic activities [3]. So it is important to develop the synthesis method of ZnO to obtain the smaller particle as results enhancing the photocatalytic activities of ZnO. There are many method to produce ZnO. Sonochemical synthesis is more effective as well as simple method to produce the sample with smaller particle size [3,4]. The mechanism of sonochemical method involved: (a) the formation, (b) growth, and (d) collapse of bubbles in liquid and then can produce high temperature and pressure. This condition can use to chemical reaction [3-5]. Therefore, in this research we synthesized ZnO using sonochemical method to obtain the smaller particle and in this method, we use the simple ultrasound reactor with low frequency to synthesized ZnO.
2. Experimental
Zinc acetate dihydrate was dissolved of double-distilled water and then aqueous solution of 1 M NaOH was poured drop wise in this solution while stirring it continuously at room temperature until pH 12. The result solution was transferred into ultrasonic reactor for 30 (ZnO 30 min), 60 (ZnO 60 min), 180 (ZnO 180 min), and 360 minutes (ZnO 360 min) at room temperature. The white precipitate was aged for 24 h and filtered, washed with water and ethanol for several times and then dried. The frequency of sonicator is 42 kHz with power output 100 W.

The sample results were characterization using X-ray diffraction (XRD), scanning electron microscopy (SEM), and diffuse reflectance spectroscopy (DRS). The test of photocatalytic activities applied to methylene blue (MB) using ultra violet (UV) light source with wavelength 365 nm from 0 to 60 minutes.

3. Result and Discussion
Figure 1 shows XRD patterns of synthesized ZnO at time variation of sonication. The all of XRD pattern sample matched with standard ZnO wurtzite (JCPDS No. 36-1451). All the reflection can be indexed to the pure hexagonal phase of ZnO with space group $P6_3mc$ with the crystal lattice $a = 3.2495$ (Å), $c = 5.2069$ (Å). The XRD pattern also showed that at 30 minutes sonication, ZnO is successfully formed and there are no change when the sonication time was longer.

![Figure 1. The X-ray powder diffraction pattern of ZnO](image-url)

The micrograph of SEM are shows in Figure. 2. ZnO morphology which synthesized using sonication method has better distribution than without sonication method. It indicated that the time of sonication influenced to the morphology of particle [6]. Optimum distribution achieved at 30 minutes of ultrasonic irradiation because the morphology of other time variation and without sonication method is not uniform.
and produce agglomeration on ZnO morphology. It indicated at more 30 minutes of ultrasonic irradiation, the smaller particle attracted each other and produces the agglomerates.

![SEM images](image1.png)

**Figure 2.** SEM image of synthesized ZnO at time variation of sonication: (a) 30 (b) 60 (c) 180, and (d) 360 minutes.

Figure 3 shows the DRS data of synthesized ZnO. As can be seen that the reflectance value was decreased in visible area for ZnO at sonication time 30 minutes if compared with others. The increasing of absorbance in visible range gave opportunity to work in visible light range, so it is expected to have more effective photocatalytic activity. The value of bandgap energy was obtained from % reflectance and calculated using Kubelka-Munk equation and was shown in Table 1. The lowest bandgap energy which obtained at ZnO 30 minutes sonication is 2.79 eV which equivalent with wavelength 444.44 nm. It indicates that ZnO 30 min has more effective potential to utilized as photocatalyst material.
Figure 3. DRS spectra of samples (inset: % absorbance spectra data)

Table 1. The bandgap energy of ZnO

| Material          | Bandgap Energy (eV) | Wavelength (nm) |
|-------------------|---------------------|-----------------|
| ZnO 30 min        | 2.79                | 444.44          |
| ZnO 60 min        | 3.18                | 389.93          |
| ZnO 180 min       | 3.10                | 400.00          |
| ZnO 360 min       | 3.16                | 392.40          |

Figure 4 shown that degradation MB without UV and ZnO catalyst less than 4%, while degradation MB without ZnO and using UV until 5%. The optimum degradation MB using ZnO catalyst UV was obtained from ZnO 30 min at value 77.94%. It relates to ZnO 30 min is (a) smallest particle size (b) lowest bandgap energy, and (c) light absorption at visible light. And also it indicated that the size of particle influenced to the photocatalytic activity [7].
4. Conclusion
Photocatalytic material ZnO was synthesized using sonochemical method have wurzite structure with space group $P6_3mc$. The morphology particle of ZnO which synthesized using sonication method has better distribution than without sonication method, while the optimum distribution achieved at 30 minutes of ultrasonic irradiation.

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