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Structural, Optical, and Antifungal Characters of Zinc Oxide Nanoparticles Prepared by Sol-gel Method

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Abstract. The preparation of zinc oxide nanoparticles has been successfully done by employing a sol-gel route. The x-ray diffraction data analysis presented that the zinc oxide particles crystallized as hexagonal wurtzite structure and sized in nanometric scale of 35.8 nm. The scanning electron microscopy image showed that the sample had agglomeration pattern with the particles size of 38.2 nm. Such results were confirmed by a synchrotron small-angle x-ray scattering that the prepared sample agglomerated in 3-dimensional structure with a fractal dimension of about 3. The functional group of the Zn-O as the main component of zinc oxide were observed at the wavelengths of about 454 and 523 cm⁻¹. Moreover, the optical band gap energy of the zinc oxide nanoparticles was of 3.53 eV. Excitingly, the powdered sample in this work had an average inhibition zone diameter of 4.59 mm for *Candida albicans* fungus. Therefore, the prepared zinc oxide nanoparticles exhibited imperative materials for antifungal agent.

Keywords: Zinc oxide, nanoparticle, structure, optical band gap energy, antifungal agent.

1. Introduction

A nanomaterials study to develop medical applications including antibacterial and antifungal applications has been an interest of many experts in the nanoscience and nanotechnology fields. Previous report shows that the nanomaterials having the ability to kill microorganism in the large quantity is known as "wonder of modern medicine" [1]. The high performance of nanomaterials in the application is generally related to the nanomaterials characteristics such as particle size, distribution, and morphology. Furthermore, several previous studies show that nanomaterials have been developed in recent years are able to show a prime performance for various applications especially related to the uniqueness of the catalytic [2], optical [3], electronic [4] and antimicrobial [5] properties.

Of the many nanomaterials being developed by the researchers, zinc oxide has become one of the materials that is interesting to be studied, particularly related to the biomedical application [6]. A number of several previous research showed that zinc oxide nanoparticles gave the highest catalytic efficiency,



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strong adsorption, and act as an antibacterial agent [7] and antifungal agent in the wastewater treatment [8,9]. Interestingly, the particle is designated as one of the many materials that are recognized as safe [10]. In relation to the development of zinc oxide particles for an antifungal application, the fundamental study that covers the nanostructural hierarchy and the optical properties become necessary to be performed, including its correlation with the antifungal performance.

2. Materials and Methods

The materials used were zinc acetate dihydrate, NaOH, H₂O, methanol, DMSO, *Sabouraud Dextrose Agar* (SDA), dan dextrose. The instruments used were the magnetic stirrer, beaker glass, pipette, filter paper, petri dish, pH paper, spatula, mortar, and oven. The synthesis of zinc oxide nanoparticles was done by a sol-gel method began by a dissolution process of *zinc acetate dihydrate* in H₂O using a magnetic stirrer, and then proceeded with a titration process with NaOH until reaching pH 13 to obtain a gel solution. Next, the gel solution was heated at the temperature of 90 °C for 30 minutes which then followed by a washing process with methanol until reaching pH 7. The next method is the filtering which was followed by the heating process at the temperature of 100 °C for 60 minutes to generate zinc oxide particle powder.

The zinc oxide particle powder was then characterized using an x-ray diffractometer (XRD), small angle x-ray scattering (SAXS), scanning electron microscopy (SEM), Fourier transform infra-red (FTIR) spectroscopy, as well as antifungal testing. The XRD, SAXS, SEM, and FTIR characterizations were performed to investigate the structural and optical characters of the sample. Moreover, the antifungal performance on the sample using *Candida albicans* fungus was investigated by means of a well-diffusion method.

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3. Results and Discussion

Figure 1 presents the diffraction pattern of an x-ray for the zinc oxide nanoparticles was fitted through a refinement analysis using Rietica software. In the refinement process, the AMCSD data no 0005203 was used as the zinc oxide data model. The refinement analysis results showed that the experimental data have the highest match with the data model indicated by the statistical parameter value $R_p = 14.7$, $R_{wp} = 20.9$ and $\chi^2 = 1.8$. Based on Figure 1, visually, the zinc oxide phase peaks were identified at 2-theta at angles of $(2\theta) = 31.7^\circ, 34.4^\circ, 36.2^\circ, 47.5^\circ, 56.6^\circ, 62.8^\circ, 66.3^\circ, 67.9^\circ, 69.1^\circ, 72.5^\circ$, and 76.9° . The diffraction patterns have similar patterns to other research that have been reported previously [11]. Generally, the zinc oxide particles have a diffraction pattern with the characteristics that tend to be high and sharp indicating that the sample has a high crystallinity. Furthermore, from the data analysis, it was not found a new phase or impurity indicating that the sample has a high purity level.

The results of the X-ray diffraction data analysis quantitatively show that zinc oxide particles have a hexagonal wurtzite crystal structure with a lattice parameter value of $a = b = 3.251 \text{ \AA}$ and $c = 5.211 \text{ \AA}$, with $\alpha = \beta = 90^\circ \neq \gamma = 120^\circ$. The sample of synthesis result in this research also has the crystal volume of $47.7 \text{ \AA}^3$ with the crystallite size of 35.8 nm . This crystal structure has a $P6_3mc$ space group indicated by two sublattices interconnected including Zn^{2+} and O^{2-} , thus each Zn ion is surrounded by tetrahedral O ions and vice versa. In addition, such wurtzite crystal has the value of the axial ratio of $c/a = 1.6029$ and parameter of $u = 0.379$ whose results are nearing the ideal atomic structure [12]. Based on the data analysis, the values of crystal lattice distortion and bond-length degree of Zn-O on zinc oxide particles are 1.0186 and 1.9783 respectively. Crystallographically, the crystal distortion on zinc oxide particles caused a change in the electronic bond between two nearest atoms which affected the length of Zn-O bond.

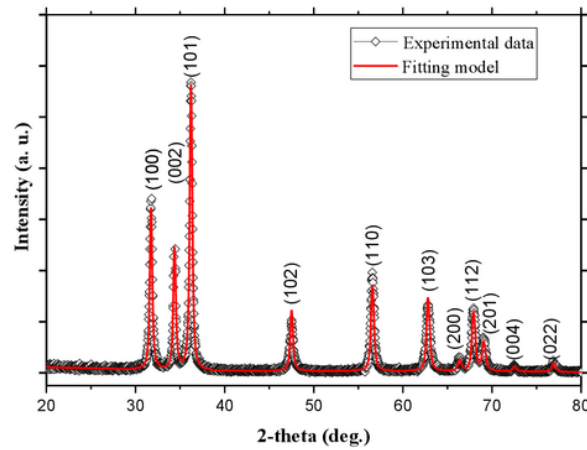


Figure 1. Experimental x-ray diffraction data and fitting model of the zinc oxide nanoparticles

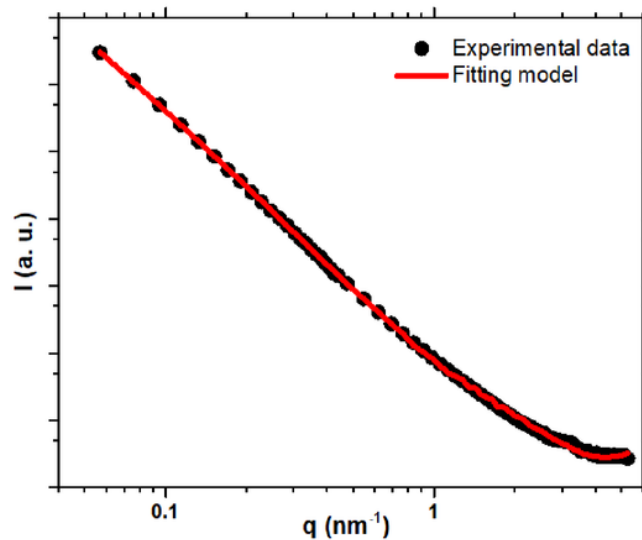


Figure 2. Scattering profile of the zinc oxide nanoparticles

A scattering was employed to determine the size, shape, distribution, and their correlation in a nanometre scale. Based on Figure 2, generally, the zinc oxide nanoparticles were consisted of primary particles forming an aggregation in fractal structure. Therefore, we applied a lognormal distribution combining with mass fractal model to fit the scattering data. The mathematical model for fitting the scattering data was modified from the mathematical model reported in previous work [13].

$$I(q) \approx \int_0^\infty N(R) F_N^2(q, R) dR S(q, \varepsilon, D, R) \quad (1)$$

where F is the amplitude of scattering, N is the total of particle density, $S(q, \varepsilon, D, R)$ is the mass fractal structure factor, q is the scattering vector, ε is the correlation length, D and R are the fractal dimension and particle size.

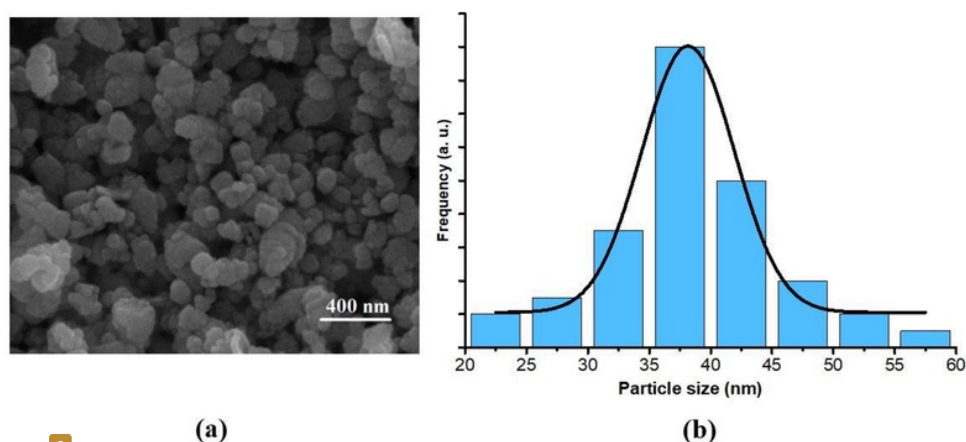


Figure 3. (a) SEM image and (b) particle size distribution of the zinc oxide nanoparticles

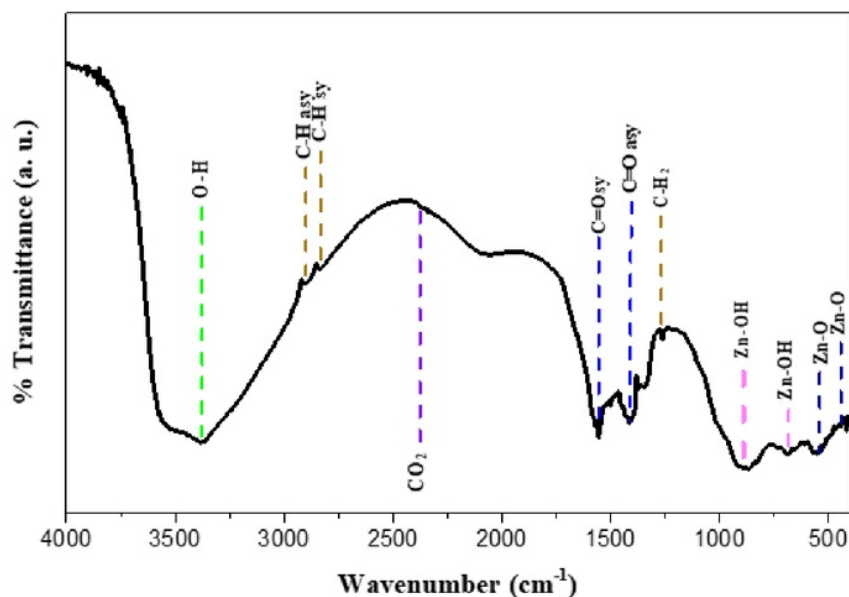


Figure 4. FTIR spectrum of the zinc oxide nanoparticles

The result of data analysis for the scattering data shows that the zinc oxide nanoparticles has a primary particles of approximately 9.14 nm¹ constructing a bigger cluster with the size of 34.39 nm. Furthermore, the fractal dimension of the zinc oxide nanoparticles is about 2.85 which represents that the particles constructed in 3-dimensional structure. These result is in line with the results of data analysis obtained from XRD and SEM characterizations. The morphology of the zinc oxide nanoparticles visually can be seen in Figure 3. The visual results of SEM data analysis on the zinc oxide nanoparticles sample indicated that the shape of the particle tends to be spherical and slightly monodisperse, both for primary and secondary particles. However, generally, zinc oxide nanoparticles

still experienced a slight agglomeration with the particle size of 38.16 nm. Interestingly, the particle size from SEM data analysis results corresponded/approached the results generated from the X-ray diffraction data analysis.

Table 1. Functional groups of the synthesized the zinc oxide nanoparticles

Wavenumber (cm ⁻¹) Experimental data	Functional groups	Wavenumber (cm ⁻¹) References	References
454 and 523	Zn-O	454 and 523	[14], [15]
690 and ~900	Zn-OH	717 and ~900	[16,17]
3443	O-H stretching	3400	[18]
1384 and 1630	C=O asymetris and C=O symetris	1384 and 1630	[19,20]
2376	CO ₂	2376	[21]
2885 and 2937	C-H symetris and C-H asymetris	2885, 2937, 2860 and 2950	[11,22]
1268	CH ₂	1050	[16]

In general, the agglomeration process was caused by the high surface energy and specific surface area on the particle of zinc oxide nanoparticles. Besides that, the agglomeration also enabled a crystal nucleation dominantly due to the higher NaOH saturation, thus producing a fast conversion of the Zn²⁺ ion to be [Zn(OH)₄]²⁻ with low agglomeration directing to the smaller particle size [23]. Furthermore, the crystal growth in zinc oxide nanoparticles was caused by the chemical reaction rate and the Zn²⁺ and OH⁻ ion concentration. When noted on SEM figure closely, it is very apparent that there is crystal growth. The important roles which caused the crystal growth and nucleation among them are the role of ion OH⁻ in the solution and during the process of sample heating.

The functional group formed on the zinc oxide nanoparticles sample was reviewed using FTIR spectroscopy as shown in Figure 4. Based on Figure 4 and Table 1, it is apparent that the functional group of zinc oxide nanoparticles from the synthesis results in the research showed a similar result with the previous research. At the low wavenumber, the Zn-O functional group was formed at the wavenumbers of 454 and 523 cm⁻¹. These results showed that the zinc oxide particles have been well configured as presented in the x-ray diffraction data. Besides that, the Zn-OH functional group appeared at the wavenumbers of 690 cm⁻¹ and approximately 900 cm⁻¹. Furthermore, at the wavenumber of 3443 cm⁻¹, the hydroxyl stretching vibration of O-H often occurred. The appearance of the O-H bond showed the existence of water absorbed in the zinc oxide particles surface [24].

The carboxyl C-O bond was also identified at the wavenumber of 1384 cm⁻¹ and 1630 cm⁻¹ which possibly came from the reactive carbon during the synthesis process [20]. The appearance of C-H₂ and the symmetrical and asymmetrical C-H functional group bonds at the wavenumbers 1268, 2885, and 2937 cm⁻¹ identified the existence of the monoacetate group as the intermediate product [22]. Meanwhile, the functional group of CO₂ at the wavenumber of 2376 cm⁻¹ was predicted coming from the outside air. To study the other important characteristics related to the optical properties of band gap energy, the UV-Vis characterization was performed at room temperature. The characterization results showed the absorbance relation with the wavelength in the range of 200-800 nm as presented in Figure 4.

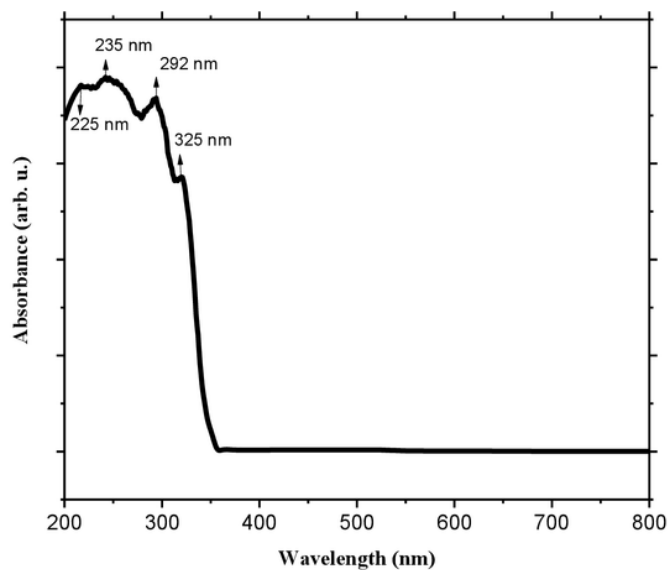


Figure 5. The relation between wavelength and absorbance of the zinc oxide nanoparticles

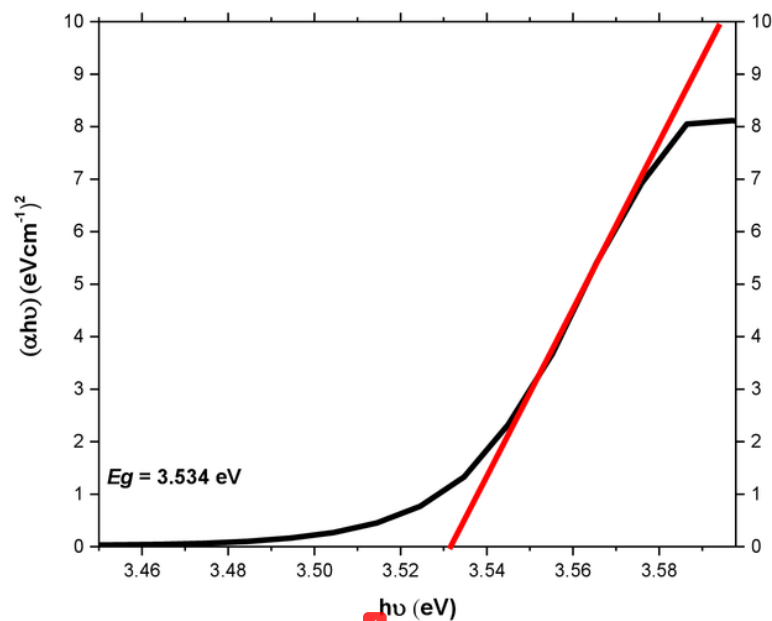


Figure 6. Band gap energy of the zinc oxide nanoparticles

Based on Figure 5, the absorption spectrum of the zinc oxide nanoparticles was identified at the wavelength of 200–380 nm. The maximum absorption peaks of zinc oxide nanoparticles were identified at the wavelength of 225, 235, 292, and 325 nm. Interestingly, the absorption of zinc oxide nanoparticles at the obtained wavelength was far lower compared to that of the absorption of zinc oxide nanoparticles with bulk size that occurred at 388 nm. The emerged absorption at the wavelength of 225 nm can be

related to the inter-band transition on the metal electron from the basic level of valence band [25]. On the particle of the sample, a direct transition occurred when the electron transitioned from the valence band to the conduction band directly. Mathematically, the band gap energy of the zinc oxide nanoparticles was obtained from the linear fitting by using a Davis & Mott's equation as on Equation (2) [26] through the graphic relation of $h\nu$ and $(ah\nu)^2$ as shown in Figure 6.

$$(ah\nu)^{1/n} = A(h\nu - E_g) \quad (2)$$

where $h\nu$ is the photon energy (eV) related to the material, E_g is the band gap energy, A is the contradiction constant and, n is the exponential value of $1/2$ for direct band gap energy. Based on the fitting linear results in Figure 7, it was obtained the band gap energy of 3.534 eV. This experimental data corresponds to the previous study conducted by Preethi *et al* (2016) which shows that the value of band gap energy of zinc oxide nanoparticles from the synthesis results with a sol-gel method was approximately 3.5–3.9 eV [27]. In physics, the value of band gap energy identified that the zinc oxide nanoparticles belong to semiconductor material category.

Table 2. The results of antifungal activity test on the zinc oxide nanoparticles

Treatment	Inhibition Zone Diameter (mm)		
	Zinc oxide nanoparticles	Antibiotic (Positive control)	H ₂ O (Negative control)
1	4.30	26.00	0
2	5.20	25.05	0
3	4.00	25.50	0
4	4.85	24.85	0
Average	4.59	25.35	0

In this research, the antifungal activity test on the zinc oxide nanoparticles was conducted using *Candida albicans* fungus through a well-diffusion method. The antifungal activity test results on zinc oxide nanoparticles (concentration of 54%) were presented in Table 2. The average value of antibiotic inhibition zone diameter (positive control) on *Candida albicans* fungus was 25.35 mm. Meanwhile for aquadest (negative control) did not show any change in the inhibition zone diameter of *Candida albicans* fungus. An adequate significant change occurred on ZnO sample with an antibiotic as the positive control. Based on the results obtained, it showed that the ZnO particles gave a significant effect in inhibiting the growth of *Candida albicans* fungus. The growth inhibition of *Candida albicans* fungus by ZnO particle was likely caused by the ZnO particle has a small size (nanometer scale) which could go through the cell walls due to the direct contact between particles with fungal membrane cells, thus it was able to inhibit the performance of fungal cells which caused the fungal growth inhibition even the fungal cell death. The results of the inhibition zone diameter are almost close to the previous study conducted by Sharma *et al.* which are about 1.5–11.4 mm [28]. Therefore, the powdered zinc oxide nanoparticles prepared in this work becomes imperative materials to be applied as antifungal agent.

4. Conclusion

We conclude that the ZnO sample was successfully fabricated using a sol-gel method. The crystal structure of the ZnO sample was in hexagonal wurtzite and crystallized in nanometric scale of 35.8 nm. Furthermore, the visual morphology of the ZnO sample identified a spherical particle shape with the particle size distribution formed by several crystallites of 38.16 nm. The functional group of the ZnO sample was identified at the wavenumber of 454 and 523 cm⁻¹. The optical properties of ZnO showed that the sample has a gap energy value of 3.534 eV. Meanwhile, antifungal activity of the ZnO sample has an inhibit zone diameter of 4.59 mm.

Acknowledgments

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