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Synthesis and Characterization of ZnO with Calcination Temperature Variation and Surfactant Addition using Chemical Coprecipitation Method

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Abstract

This study aims to synthesize ZnO using the coprecipitation method and characterize it using various instrumentation techniques. The calcination temperature variation treatment was carried out to produce particles with good size and crystallinity levels. Adding polyvinyl pyrrolidone (PVP) surfactant during the synthesis of ZnO aims to prevent agglomeration. ZnO characterization used ultraviolet-visible spectroscopy (UV-Vis), X-ray diffraction (XRD), Fourier transform spectroscopy (FTIR), and scanning electron microscopy (SEM). The result showed that the value of the ZnO band gap increased with increasing temperature. ZnO synthesized at a calcination temperature of 800 °C has a hexagonal wurtzite structure. The crystal size of ZnO is 45.64 nm, while ZnO-PVP's is 40.37 nm. The best ZnO crystallinity level is 61.34%. The FTIR spectrum shows ZnO absorption in the fingerprint region (525 cm^{-1}). SEM micrographs showed that ZnO without the addition of PVP agglomerated. The synthesized ZnO has an antibacterial activity, indicated by forming an inhibition zone (7.84 mm).

Keywords: antibacterial, coprecipitation, polyvinyl pyrrolidone, temperature, ZnO

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Abstrak (Indonesian)

Penelitian ini bertujuan menyintesis ZnO dengan metode kopresipitasi dan mengkarakterisasi menggunakan berbagai instrumentasi. Perlakuan variasi suhu kalsinasi dilakukan agar dihasilkan partikel dengan ukuran dan tingkat kristalinitas yang baik. Penambahan surfaktan polivinil pirolidon (PVP) pada saat sintesis ZnO bertujuan mencegah terjadinya aglomerasi. Karakterisasi ZnO menggunakan spektroskopi ultraviolet-tampak (UV-Vis), difraksi sinar-X (XRD), spektroskopi inframerah transformasi fourier (FTIR), dan mikroskop elektron pemayaran (SEM). Hasil penelitian menunjukkan bahwa nilai celah pita ZnO meningkat seiring dengan kenaikan suhu. ZnO yang disintesis pada suhu kalsinasi 800 °C memiliki struktur wurtzit heksagonal. Ukuran kristal ZnO adalah 45,64 nm, sedangkan ZnO-PVP sebesar 40.37 nm. Tingkat kristalinitas ZnO terbaik adalah 61,34%. Spektrum FTIR menunjukkan serapan ZnO pada daerah sidik jari (525 cm^{-1}). Mikrograf SEM memperlihatkan bahwa ZnO tanpa penambahan PVP mengalami aglomerasi. ZnO hasil sintesis memiliki aktivitas antibakteri yang ditunjukkan dengan terbentuknya zona hambat (7,84 mm).

Kata Kunci: antibakteri, kopresipitasi, polivinil pirolidon, suhu, ZnO

INTRODUCTION

ZnO is a transition metal inorganic compound generally a non-toxic white powder. ZnO is a class II-VI semiconductor material in material science because zinc and oxygen are in groups 12 and 16 in the periodic table. ZnO has a high band gap value of 3.37 eV at room temperature and a strong excitation binding energy of 60 meV. ZnO is of great interest to researchers because it has stable physical and chemical properties, such as

electrically stable [1], optical [2], can heal wounds [3], and photocatalytic [4]. ZnO has high optical absorption in the UVA (315-400 nm) and UVB (280-315 nm) regions, which is beneficial in antibacterial response [5].

ZnO synthesis can be done by sol-gel method, precipitation, coprecipitation, mechanical milling, organometallic synthesis, microwave, spray pyrolysis, hydrothermal, and mechanochemical synthesis [6]. In this study, ZnO was synthesized using the coprecipitation method. The coprecipitation method was chosen because it is simple, uses low temperatures relatively quickly, is easy to do, and can produce ZnO with uniform morphology and controllable size [7]. By using the coprecipitation method, the crystal structure of the synthesized sample can be optimized by controlling the synthesis parameters such as temperature, solvent, solution pH, stirring speed, metal salt concentration, precipitant concentration, and surfactant concentration [8].

ZnO can be increased in crystallinity by calcination, which is heating with high temperatures that do not exceed its melting point or the influence of pressure [9]. The higher the calcination temperature, the smaller the band gap energy, and the crystallinity will increase [10]. This study used 400, 600, and 800 °C calcination temperature variations. The calcination process triggers high surface energy that can cause agglomeration. Agglomeration can be prevented by adding a capping agent to the synthesis [11]. This study used 0.5% polyvinyl pyrrolidone (PVP) surfactant as a capping agent. The synthesis results were then characterized using UV-Vis, FTIR, XRD, and SEM.

MATERIALS AND METHODS

Tools and Materials

The tools used to synthesize ZnO are glass cups, measuring cups, drop pipettes, stirring rods, magnetic stirrers, hot plates, centrifugation tubes, ovens, and furnaces. Tools for analysis and testing used a set of UV-Vis instruments (U-2800 Spectrophotometer), a set of X-ray Diffractometer instruments (XRD Empyrean Series 3 Panalytical), a set of Fourier Transform Infrared instruments, a set of Scanning Electron Microscope instruments, and OriginPro 8.5 software. Tools used for antibacterial tests are Petri dishes, sterile cotton sticks, paper discs, and a caliper. The materials used for the synthesis of ZnO are zinc nitrate hexahydrate ($Zn(NO_3)_2{\cdot}6H_2O$) (HIMEDIA), NaOH (Merck), polyvinyl pyrrolidone (PVP) 0.5% (b/v), and distilled water. The material for the UV-Vis test is DMSO solvent. The ZnO antibacterial test materials are MHA (Muller Hinton Agar) agar medium, 10% DMSO, and Gram-positive bacteria *S. aureus.*

Research Procedures

The synthesis of zinc oxide was carried out by dissolving 12 g of zinc nitrate hexahydrate ($Zn(NO_3)_2{\cdot}6H_2O$) into 100 mL of distilled water with constant stirring at 50 °C for 25 min using a magnetic stirrer (For the synthesis of ZnO with the addition of PVP, an amount of 0.5% (b/v) PVP was added to the zinc nitrate hexahydrate solution). A magnetic stirrer dissolved 3.2 g NaOH in 30 mL of distilled water with constant stirring at 30 °C for 10 min. The NaOH solution was then added dropwise into the ($Zn(NO_3)_2{\cdot}6H_2O$) solution while stirring using a magnetic stirrer for 2 hours at 50 °C. The resulting precipitate was centrifuged for 15 minutes at 5000 rpm and washed with distilled water until the pH was neutral. The $Zn(OH)_2$ precipitate was then dried in an oven at 120 °C for 3 hours. In the last step, the dried precipitate was then calcined at 400, 600, and 800 °C in a furnace for 4 hours. The obtained samples were collected in an airtight container at room temperature. The color of the resulting ZnO powder is white. The ZnO formation reaction is as follows [12]:

The precursors $Zn(NO_3)_2{\cdot}6H_2O$ and NaOH were each dissolved in distilled water.

$$2NaOH_{(s)} \rightarrow 2Na^+_{(aq)} + 2OH^-_{(aq)} \quad (1)$$

$$Zn(NO_3)_2.6H_2O_{(s)} \rightarrow Zn^{2+}_{(aq)} + 2NO_3^-{}_{(aq)} + 6H_2O_{(aq)} \quad (2)$$

NaOH solution is dripped into $Zn(NO_3)_2.6H_2O$ solution by heating

$$Zn^{2+}_{(aq)} + 2OH^-_{(aq)} + H_2O_{(aq)} \rightarrow Zn(OH)_2.H_2O_{(s)}\downarrow \quad (3)$$

$$2Na^+ + 2NO_3^- \rightarrow 2NaNO_3 \quad (4)$$

$Zn(OH)_2$ is oven-dried and calcined, resulting in ZnO powder

$$Zn(OH)_2.H_2O_{(s)}\downarrow \rightarrow Zn(OH)_{2(s)} + H_2O_{(g)} \quad (5)$$

$$Zn(OH)_{2(s)} \rightarrow ZnO_{(s)} + H_2O_{(g)} \quad (6)$$

Characterization was performed on ZnO powder. Analysis using UV-Vis spectrophotometer to determine ZnO formation's optimum absorption and wavelength. FTIR analysis was conducted to determine the functional groups of ZnO. XRD analysis was conducted to identify the type of phase and size of

ZnO crystals. SEM analysis was performed to identify the morphology of ZnO. An antibacterial activity test was conducted using a ZnO concentration of 30%.

RESULTS AND DISCUSSION

Effect of Calcination and Surfactant

Calcination temperature affects particle size, where the higher the calcination temperature, the smaller the particle size [10]. Increased calcination temperature will cause atomic diffusion to be faster, thereby increasing crystallinity, and the size of the crystals formed will be smaller [13]. Calcination can also remove impurities on the sample's surface but can cause agglomeration because it triggers high surface energy. Agglomerations can affect the absorption shift and the resulting band gap energy value.

This study used polyvinyl pyrrolidone (PVP) to overcome agglomeration. PVP is a surfactant that can reduce aggregation and particle size [14]. PVP with a uniform and regular chain structure is readily adsorbed on the surface of metal oxide colloids. When the colloidal surface adsorbs PVP, the colloidal activity will significantly reduce, and the colloidal growth rate will be limited. The addition of PVP in the reaction system will modify the particle growth rate, increase the crystallinity of the sample, and change the morphology of the product [15].

Band Gap Analysis

Figure 1(a) is the UV absorption spectrum of ZnO and ZnO-PVP samples with various calcination temperatures. The spectrum of ZnO calcination at 400 °C looks different, which causes the spectra of other samples not to be seen clearly. The calcined ZnO 400 °C absorbance value is greater than the other samples, 2.337 (**Table** 1). This can be caused by several factors, such as the level of ZnO in the samples, where the higher the level of substances contained in a sample, the more molecules will absorb light at a specific wavelength so that the absorbance value will be greater [16]. **Figure** 1(b) is the UV absorption spectrum without the 400 °C calcined ZnO sample.

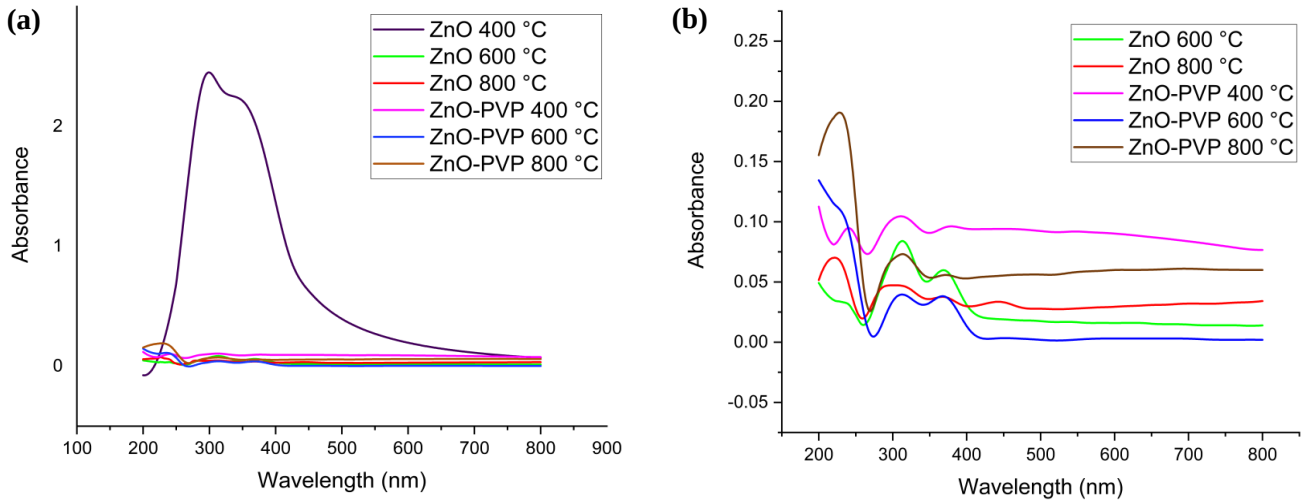

Figure 1. UV-Vis spectra of ZnO and ZnO-PVP (a) with calcination temperature 400 °C and (b) without calcination temperature 400 °C

Table 1. Temperature relationship with band gap value of ZnO

Sample	Calcination temperature (°C)	Band gap (eV)	λ_{max} (nm)	Absorbance
ZnO	400	3.026	301	2.337
	600	3.031	313	0.087
	800	3.370	220	0.070
ZnO-PVP	400	3.506	310	0.104
	600	3.578	312	0.040
	800	3.994	228	0.191

The calcination temperature affects the resulting band gap value. The more significant the calcination temperature, the smaller the band gap [17]. This study produces a band gap that gets bigger as the calcination temperature increases, so the results differ from those of the literature for both ZnO and ZnO-PVP (**Table** 1).

This is because, at high calcination temperatures, aggregation is more likely to occur. Agglomeration can affect the absorption shift and the resulting band gap. The resistivity of the material can cause the band gap value to increase with increasing calcination temperature, the primary material for making particles, and the quality of the resulting coating [18]. This is caused by electrons that are free to move in the ZnO conduction band and accelerate the conductivity of ZnO.

The band gap produced by the 800 °C calcined ZnO is 3.37 eV (**Table** 1). This is following Gungor and Gungor [19] who state that ZnO is a semiconductor with a vast band gap energy of 3.37 eV, so 800 °C calcined ZnO is used for further characterization. Based on the results obtained, the synthesized ZnO and ZnO-PVP include insulator-type materials because the band gap value produced is more than 3 eV, while the band gap value of semiconductor materials ranges from 1–3 eV [20].

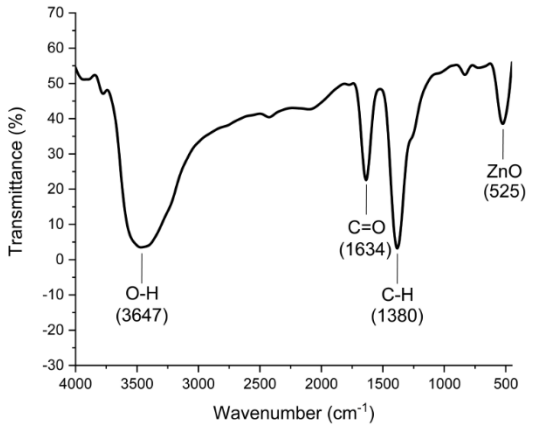

Figure 2. FTIR results of ZnO calcination 800 °C

FTIR Analysis Results

ZnO calcination at 800 °C showed major peaks at wave numbers 3647 cm^{-1}, 1634 cm^{-1}, 1380 cm^{-1}, and 525 cm^{-1} (**Figure** 2). The peaks at wave numbers 3647 cm^{-1} are related to the O-H stretch from the presence of water absorbed on the ZnO surface or can indicate the formation of hydrogen bonds [21]. The peak at the wave number 1634 cm^{-1} indicates the symmetrical stretching of C=O [22]. The peak at wave number 1380 cm^{-1} indicates C-H buckling of alkane groups [12]. Metal oxides generally give absorption bands in the fingerprint region below 1000 cm^{-1} arising from interatomic vibrations [23]. The peak at wave number 525 cm^{-1} is associated with ZnO phonon vibrations [12].

XRD Analysis Results

Figure 3 is the XRD diffraction pattern of ZnO and ZnO-PVP at a calcination temperature of 800 °C. All samples' analysis results with XRD instruments have almost the same peaks at angular values (2θ). The difference in XRD peak intensity affects the (FWHM) value and crystal size. Peak ZnO calcination temperature 800 °C is at an angle of 2θ with Miller's index as follows: 31.65° (100); 34.51° (002); 36.35° (101); 47.59° (102); 56.62° (110); 62.80° (103); 66.39° (200); 68.04° (112); and 69.14° (201). The peak of ZnO-PVP calcination temperature of 800 °C is at angles 2θ with Miller indices 31.65° (100); 34.31° (002); 36.26° (101); 47.59° (102); 56.80° (110); 62.88° (103); 66.35° (200); 67.86° (112); and 68.9° (201). The peak positions in **Figure** 3 indicate the formation of a hexagonal wurtzite crystal structure with three orientations (100), (002), and (101), which is in good agreement with the JCPDS standard (No. 36-1451) with crystal lattice values $a = b = 3.249$ and $c = 5.206$ (**Table** 2) [24]. The diffraction peaks obtained are solid and narrow, indicating that the synthesized ZnO has good crystallinity [12].

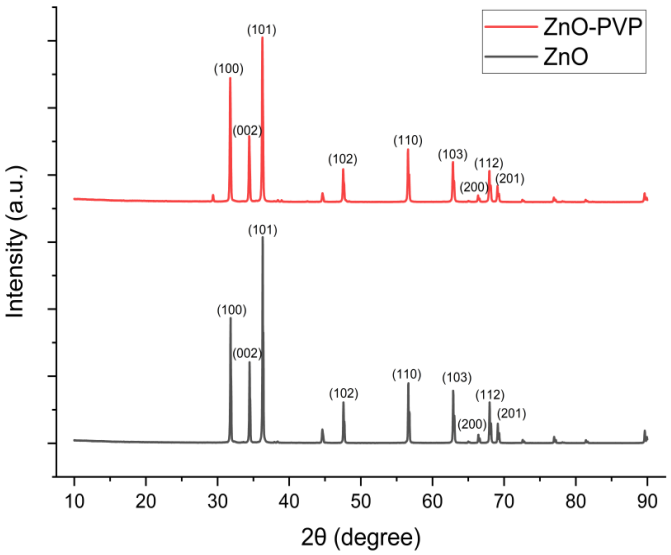

Figure 3. ZnO diffractogram of calcination temperature 800 °C

Interpretation of X-ray diffraction data in the form of FWHM can be used to determine the crystal size of a sample using the Debye-Scherrer equation.

$$D = \frac{K\lambda}{\beta \cos\theta} \quad (7)$$

where D is the crystal size (nm), k is the crystal form factor (0.9−1), λ is the X-ray wavelength (0.15406 nm), β is the value of Full Width at Half Maximum (FWHM) (rad), and θ is the diffraction angle (°) [25] .

The %Crystallinity of ZnO can be found using the following equation:

$$\text{Crystallinity (\%)} = \frac{\text{Crystalline peak fraction}}{(\text{Crystal peak fraction}+\text{Amorphous peak fraction})} \times 100\% \quad (8)$$

Crystal or apparent crystal size (ACS) is sought using equation (7). The crystal size results are in **Table** 3. The resulting crystal sizes of ZnO and ZnO-PVP calcination temperatures of 800 °C 45.64 nm and 40.37 nm, respectively. The addition of PVP into the synthesis of ZnO decreased the crystal size. This can occur because PVP can form a shell that surrounds the particles to prevent particles from becoming large [26]. This indicates that PVP controls the growth of ZnO during synthesis. The %crystallinity was found using equation (8). The %crystallinity results obtained for ZnO and ZnO-PVP were 59.74% and 61.34%, respectively. The degree of crystallinity indicates that the arrangement of atoms in crystalline particles has a regular and repetitive shape in 3-dimensional space.

Table 3 Crystal size and crystallinity degree of ZnO calcination temperature 800 °C

Sample	Crystal size (nm)	Crystallinity (%)
ZnO	45.64	59.74
ZnO-PVP	40.37	61.34

Table 2 Comparison of Miller Index of ZnO with JCPDS data no. 36-1451

Miller Index (*h k l*)	Angle 2θ according to JCPDS no. 36-1451	Angle 2θ ZnO 800 °C	Angle 2θ ZnO-PVP 800 °C
1 0 0	31.770	31.65	31.65
0 0 2	34.422	34.51	34.31
1 0 1	36.253	36.35	36.26
1 0 2	47.539	47.59	47.59
1 1 0	56.603	56.62	56.80
1 0 3	62.380	62.80	62.88
2 0 0	66.380	66.39	66.35
1 1 2	67.963	68.04	67.86
2 0 1	69.100	69.14	68.90

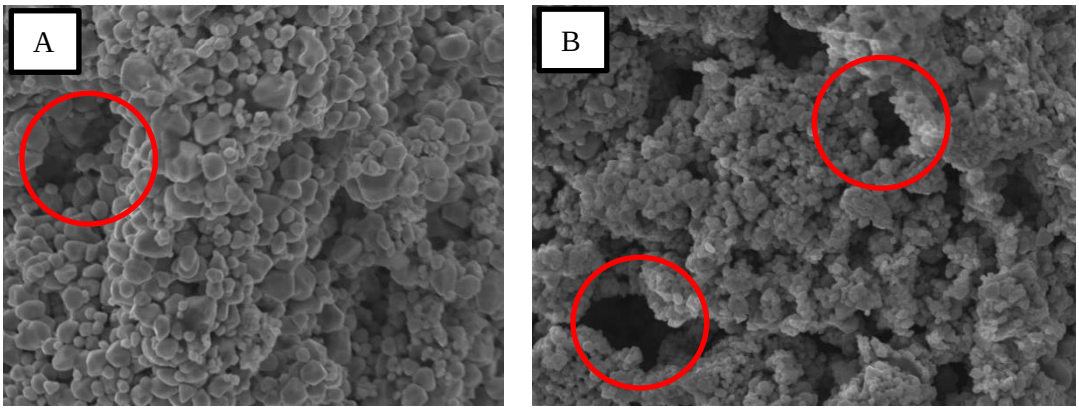

Figure 4. Morphology of ZnO calcination temperature 800 °C (A) ZnO with magnification 5000×, (B) ZnO-PVP with magnification 10,000×)

SEM Analysis Results

Figure 4 shows SEM photographs for samples A (ZnO) and B (ZnO-PVP). The gap between sample A is not visible due to aggregation. Agglomeration between ZnO particles occurs due to the influence of polarity, electrostatic power of ZnO, and significant energy on the surface of the sample [27]. The gap between the particles of sample B looks clearer due to the addition of PVP. This indicates that adding PVP to the synthesis of ZnO reduces the occurrence of agglomeration.

Antibacterial Activity Analysis Results

The antibacterial activity of the synthesized ZnO colloidal solution was tested using Gram-positive bacteria *S. aureus*. The test was carried out by

evaluating the width of the clear zone formed on the bacterial media after surface contact with a paper disk that had previously been moistened with a 30% colloidal ZnO solution with DMSO solvent. The antibacterial activity test uses DMSO solvent because the solvent does not affect the results of the ZnO antibacterial activity test [28]. The concentration of the solution also affects the ability to inhibit bacterial growth: the more significant the concentration used, the greater the ability of the particles to inhibit bacterial growth [29]. The colloidal ZnO solution showed inhibitory activity against *S. aureus* bacteria by forming a clear zone on agar media containing the bacteria. The clear zone indicates the strength of inhibition of the test sample. The wider the clear zone formed, the stronger the inhibition of the compound against bacterial growth [6]. With a concentration of 30%, ZnO produces an average clear zone width of 7.84 mm, so the inhibition strength is categorized as moderate.

CONCLUSION

ZnO was successfully synthesized using the coprecipitation method. The calcination temperature of 800 °C is the best temperature to synthesize by the coprecipitation method. The addition of PVP resulted in a smaller crystal size than pure ZnO. The best level of crystallinity is produced on ZnO added with PVP. PVP has also been shown to prevent the agglomeration of ZnO. The synthesized ZnO still provides moderate antibacterial activity with the formation of inhibition zones on *S. aureus* bacteria.

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