



Influence Variation Concentration $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ Against Group Function, Size Particles, and Morphology Particle Nanoparticles ZnO with Method Green Synthesis Using Extract Leaf Moringa

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Article Information

Article history:

Received June 27, 2023

Received in revised form
July 25, 2023

Accepted August 31, 2023

Keywords: Moringa leaves,
ZnO nanoparticles, green
synthesis, $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$

Abstract

This research was conducted to determine the effect of varying concentrations of $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (zinc nitrate hexahydrate) on functional groups, particle size, and particle morphology of ZnO nanoparticles using the green synthesis method using Moringa leaves. The materials used in this study were moringa leaf extract, $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ with various concentrations of 0.2, 0.4, 0.6, and 0.8 M, and NaOH of 0.1 M. Based on the results of characterization using FTIR, it shows that the functional groups in the content of Moringa leaves play a role in the formation of ZnO nanoparticles. Meanwhile, the results of the XRD characterization showed a diffractogram difference with standard ZnO at concentrations of 0.6 M and 0.8 M $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ with the appearance of the NaNO_3 phase. SEM characterization results show that the morphology of ZnO nanoparticles using the green synthesis method is irregular in shape.

Informasi Artikel

Proses artikel:

Diterima 27 Juni 2023

Diterima dan direvisi dari
25 Juli 2023

Accepted 31 Agustus 2023

Kata kunci: Daun kelor,
nanopartikel ZnO, sintesis
hijau, $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$

Abstrak

Penelitian ini dilakukan untuk mengetahui pengaruh variasi konsentrasi $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (seng nitrat heksahidrat) terhadap gugus fungsi, ukuran partikel, dan morfologi partikel nanopartikel ZnO dengan metode sintesis hijau menggunakan daun kelor. Bahan-bahan yang digunakan dalam penelitian ini adalah ekstrak daun kelor, $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ dengan variasi konsentrasi 0,2; 0,4; 0,6; dan 0,8 M dan NaOH sebesar 0,1 M. Berdasarkan hasil karakterisasi menggunakan FTIR diperoleh gugus fungsi dalam kandungan daun kelor berperan dalam pembentukan nanopartikel ZnO. Sedangkan hasil karakterisasi XRD menunjukkan perbedaan difraktogram dengan standar ZnO pada konsentrasi $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ 0,6 M dan 0,8 M dengan munculnya fasa NaNO_3 . Hasil karakterisasi SEM menunjukkan bahwa morfologi nanopartikel ZnO dengan metode sintesis hijau menunjukkan bentuk partikel tidak merata dan adanya gumpalan pada sampel.

1. Introduction

Nanotechnology is a branch of science that studies material that has a size on the nanoscale of 1-100 nm or $1-100 \times 10^{-9}$ m (Akbari *et al.*, 2011). In this dimension, the material shows physical, chemical, and mechanical properties superior to existing materials, so it is hoped that this nanomaterial will continue to provide breakthroughs for technology in the future (Ganguly *et al.*, 2015).

Nanoparticles can be synthesized using physical methods and chemical methods. The process of synthesis of the physical method is the breakdown of large materials into nanometer-sized materials or the combination of microscopic materials into nanometer-sized particles without changing the properties of materials (Kumar *et al.*, 2016), while chemical synthesis processes involve reaction chemistry from a number precursor so that other nanometer-sized materials are produced (Salah *et al.*, 2011).

Several methods for synthesizing ZnO nanoparticles have disadvantages, such as poisonous material that cannot decompose, large and expensive equipment, low product yield, and relatively long reaction time. To overcome

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limitations, the green synthesis method, also called *synthesis green*, is one alternative for synthesizing nanoparticles. ZnO uses materials which do not poisonous and can decompose (Verma *et al.*, 2021). Method This synthesis uses proposed plants and microorganisms technology in a more alternative environment than physics and chemistry methods.

Types of plants contain compounds metabolites, including proteins, enzymes, phenols, amines, alkaloids, flavonoids, terpenoids, and pigments, which can play roles as agent reducers and stabilizers (Rachmawati & Suriawati, 2019). Agent reducer plays a role in secondary metabolite compounds in biological organisms that will reduce Zn^{2+} ions to ZnO. Protein can catch the existing metal ions on the surface and convert them into ZnO molecules. These ZnO molecules will form ZnO nanoparticles (Marslin *et al.*, 2018). Meanwhile, the stabilizers play a role in secondary metabolite compounds by wrapping (*capping*) the formed ZnO nanoparticles so that aggregation or clumping does not occur and the resulting nanoparticles are stable (Jayachandran *et al.*, 2021).

Leaves (*Moringa oleifera*) are tropical and can be found in countries on the continents of Asia, India, and Africa. Leaf Moringa is rated Enough as safe, effective, cheap, and easy to find. The active compounds in Moringa are found in many parts of the leaves. Compound active contained in leaf Moringa, namely flavonoids, saponins, alkaloids, and phenols, role as agent reducers and stabilizers in the formation of nanoparticles ZnO (Rachmawati & Suriawati, 2019). The use of Moringa leaves is still limited primarily to the household scale.

Research on synthesizing ZnO nanoparticles using plant extracts has yet to be widely reported in Indonesia. This is because, so far, the physicochemical parameters still have to be researched. One of these parameters is variation concentration precursor. Kaningini *et al.* (2022) have studied the synthesis of ZnO nanoparticles using variation $Zn(NO_3)_2 \cdot 6H_2O$ precursor concentration that is 0.05, 0.10, 0.5, and 1 gr. According to Nurbayasari *et al.* (2017), precursor concentration varied from 0.05, 0.1, and 0.15 M and gained a good concentration of 0.15 M. In the research of Samat and Nor (2013), the synthesis of ZnO nanoparticles with different precursors used a concentration of 0.05, 0.10, 0.15, and 0.20 M and got a concentration good precursor of 0.2 M. In synthesis nanoparticles ZnO What more Good use variation concentration precursor as significant as 0, 2; 0.4, 0.6, and 0.8 M for get estimation optimal concentration.

According to Kaningini *et al.* (2022), a decline in concentration precursor can cause a decrease in n sites peak on the cluster function. He reduced the intensity peak in his research because the concentration precursor was too low (0.05 M and 0.10 gr). According to Nurbayasari *et al.* (2017), improvement in concentration can cause the size of particle ZnO to increase. In his research, the value of *Full Width at Half Maximum* (FWHM) is the lowest, that is, nanoparticles ZnO with concentration precursor 0.15 M. According to Samat and Nor (2013), variation concentration will show morphology surface particles that do not uniform. The higher the concentration, the better structure the nanoparticles have. ZnO nanoparticles obtained part big shaped round. In his research, 0.15 M and 0.20 M were seen as structures uniform with a size of around 100 nm.

This research synthesized ZnO nanoparticles from Moringa leaf extract using the precursor $Zn(NO_3)_2 \cdot 6H_2O$ and NaOH solvent. Moringa leaf extract 10 ml was reacted with varying concentrations of the $Zn(NO_3)_2 \cdot 6H_2O$ by 0.2, 0.4, 0.6, and 0.8 M and added NaOH solvent until the solution pH was 10. The characterization of ZnO nanoparticles from Moringa leaf extract was carried out as follows: *Fourier Transform Infra-Red* (FTIR) to determine the functional groups, *X-ray diffraction* (XRD) to determine the particle size, and *Scanning Electron Microscopy* (SEM) to determine the morphology of the ZnO particles.

2. Methods

This research uses extract leaf moringa, zinc nitrate hexahydrate ($Zn(NO_3)_2 \cdot 6H_2O$) 99% (Sigma Aldrich), sodium hydroxide (NaOH) 97% (Merck), distilled water, and ethanol (C_2H_5OH) (Merck). To get nanoparticles, ZnO must do several stages, including synthesis of ZnO, drying, and characterization tests using FTIR, XRD, and SEM.

The process carried out before synthesizing nanoparticles ZnO is the preparation of extract leaf Moringa. Leaf Moringa is washed using running water followed by distilled water, later taken as much as 500 gr and dried in a temperature room for ten days. Samples that have dry mashed with a blender to facilitate the extraction process use the maceration method for 124 × hours until the filtrate is obtained. The filtrate obtained was evaporated using a vacuum *rotary* evaporator to produce a dense extract.

The process carried out afterward is a synthesis process of nanoparticles ZnO using 10 ml of extract leaf moringa reacted with 30 ml of zinc nitrate hexahydrate with variation concentration by 0.2, 0.4, 0.6, and 0.8 M. Mixture stirred and heated using water bath at a temperature of 60 -70 °C and *magnetic stirrer* at 700 rpm for 10 minutes. 0.1 M NaOH solution was added with variations in the pH of the mixture of 10 accompanied by stirring continuously without hot for 1 hour. Solution left overnight so precipitate can form.

Table 1. Table composition sample.

Sample	Extract Leaf Moringa (ml)	$Zn(NO_3)_2 \cdot 6H_2O$ (gr)	NaOH (gr)
A	10	1.24	0.4
B	10	2.48	0.4
C	10	4.96	0.4
D	10	9.93	0.4

The solution was disinfected at 1000 rpm for 5 minutes three times with ethanol and distilled water to remove impurities. The precipitate obtained was collected from a Cup of ceramic and left dry in the oven at 100 °C for about one hour. The dried precipitate is ground to a fine powder.

Sample characterized by FTIR using Shimadzu brand machine type Iraprestige 21, XRD uses machine PanAnalytical type expertpro Where long wave used, i.e. 1.54059 Å operated with voltage 40 kV and strong current of 30 mA as well range peak angle 2θ is used 15° to 90° with size step of 0.020° and scan time of 10°/ min., and SEM using SEM-EDS Quattro S engine.

3. Results and Discussion

3.1. Analysis Fourier Transformation Infrared (FTIR)

Figure 1 show FTIR results for all sample. Peak different absorption detected in the range of $4000\text{--}400\text{ cm}^{-1}$. Peak uptake The observed area is 3429 cm^{-1} , and 3425 cm^{-1} is vibration stretch group OH function in polyphenols caused by water absorption by ZnO (Mydeen *et al.*, 2020). Absorption peaks between 1595 and 1583 cm^{-1} indicate vibrations stretch group function C=C of alkenes (Yedurkar *et al.*, 2016). There is a peak sharp at wave numbers 1384 and 1382 cm^{-1} allegedly because of the presence of the NO functional group derived from precursor ZnNO_3 or the existing vibration stretch C=O from sour carboxylate (Nurbayasari *et al.*, 2017). At the top, 1049 cm^{-1} and 1047 cm^{-1} indicate vibration stretch group CN aliphatic amines (Chemingui *et al.*, 2019). Peak 835 cm^{-1} indicates that the group Zn-OH function exists (Kaniningini *et al.*, 2018). At a peak of 459.06 , 418 , 433 , and 420 cm^{-1} , ZnO nanoparticles have been formed (Hassan *et al.*, 2018). ZnO is one group oxide metal, where the peak can be identified in the range $400\text{--}500\text{ cm}^{-1}$ (Kaniningini *et al.*, 2018).

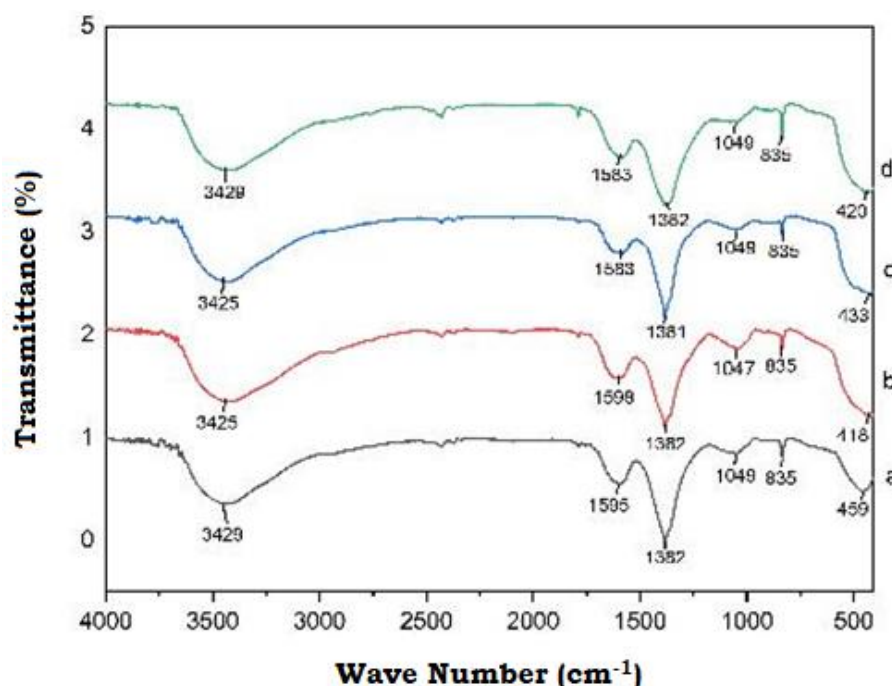


Figure 1. Sample FTIR graph variation concentration $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (a) 0.2 (b) 0.4 (c) 0.6 (d) 0.8 M.

The peak of about $600\text{--}1400\text{ cm}^{-1}$ refers to the C=O-carboxyl group or the CN group, an extension of bond amides in proteins, which signifies its high protein content in leaves plants (Mohammed & Hawar, 2022). Peletiri *et al.* (2012) report that if the protein is involved in the Zn^{2+} reduction process, it becomes Zn^0 . Proteins can trap metal ions on their surface and convert them into Zn^0 atoms; these Zn^0 atoms will form ZnO nanoparticles.

3.2. Analysis X-Ray Diffraction (XRD)

Figure 2 shows the synthesis of XRD diffractogram nanoparticles ZnO. It has been seen that the XRD pattern was obtained by the structure hexagonal crystal ZnO. Where phase dominant emerges, namely zincite at concentrations of 0.2 M and 0.4 M with group room namely P63MC according to diffractogram ICSD standard 082029 PDF Card No. 01-089-0511 and nitratine at concentrations of 0.6 M and 0.8 M with group room namely R-3C according to diffractogram ICSD standard 078802 PDF Card. 01-089-0311 (Sawada *et al.*, 1996; Gonschorek, W., 1995). **Figure 2** (a) is a sample with $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ 0.2 M, **Figure 2** (b) represents a sample with $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ 0.4 M, **Figure 2** (c) represents a sample with $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ 0.6 M and **Figure 2** (d) represents sample $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ 0.8 M.

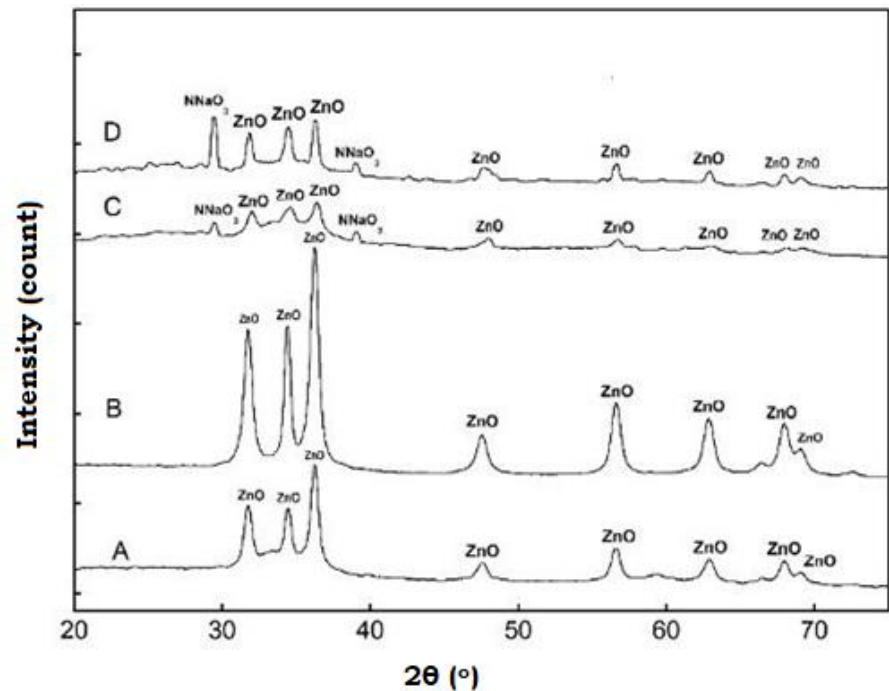


Figure 2. XRD diffractogram of nanoparticles ZnO

Corner highest 2θ diffraction results diffractogram along with wide full at half maximum or *Full Width at Half Maximum* (FWHM) and results calculation size something particle the served in **Table 1**. Use this database because it has the most suitable peak between the results study and the standard. To see the magnitude size of some particles on XRD results requires calculations based on the Scherrer equation as follows:

$$L = \frac{k \lambda}{B \cos \theta} \tag{1}$$

with L as the size particle of something crystal, B as the broad peak on FWHM, and k as a constant value of 0.94. In the Scherrer formula, the unit is radians. If the data is still diffracted between angle 2θ and intensity, then the unit is FWHM is degrees ($^\circ$), and units this degree should be changed into radians where 1° is 0.01745.

Table 2. Peak highest angle 2θ and measure particle.

Sample	$2\theta(^\circ)$	(FWHM) ($^\circ$)	Size Particle (nm)
A	36.24	0.59	14
B	36.26	0.52	16
C	36.26	0.28	30
D	36.26	0.24	35

Kindly general variation concentration precursor No make peak diffraction shift, everyone peaks consistent at a relative angle The same by ICSD standard (Fauza *et al.*, 2021). **Table 2** shows that with the existing variation concentration $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, which increases, angle 2θ at the highest peak also shifts. According to Nurbayasari (2017), increasing Zn^{2+} concentration peak FWHM value tends to decrease. It is suspected that enhancement concentration can cause crystallinity ZnO to increase.

The exciting thing from this research is that the addition of $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ occurs in the emergence of a new phase, and this research proves that the addition of $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ gives rise to a new phase with the method synthesis this green. This is expected Because, during the study, there was not enough washing with a centrifuge.

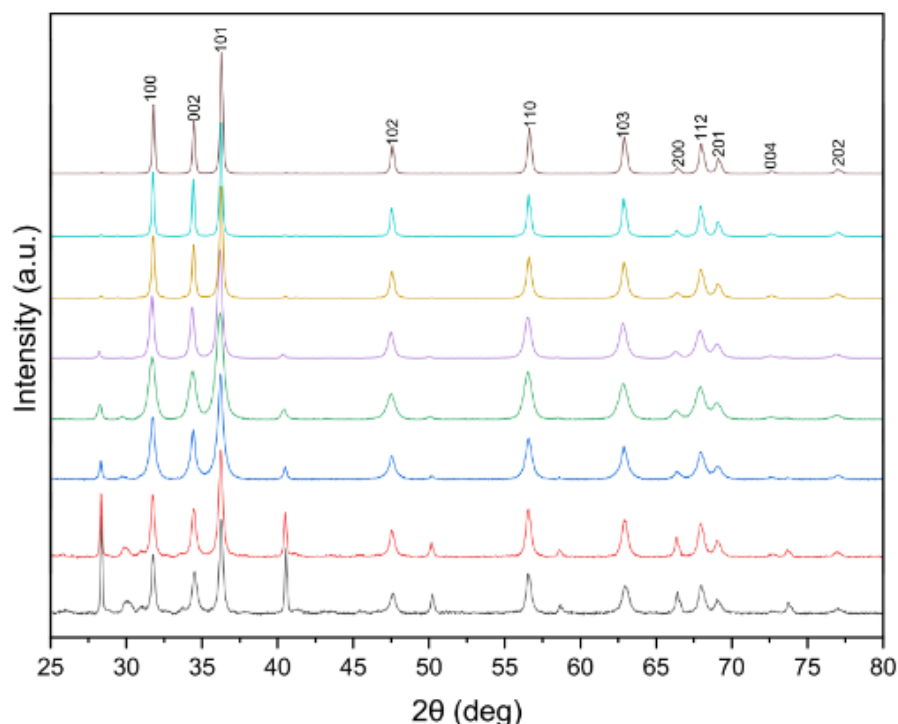


Figure 3. XRD diffractogram of nanoparticles ZnO use extract leaf *Athrixia phylicoides*.

Figure 3 shows the XRD pattern of synthesized powder using concentration of different precursors by Kaningini *et al.* (2022). All samples show peaks at 32, 34, 36, 47, 56, 63, 66, 68, 69, 73, and 77° are corresponding with the Wurtzite structure of nanoparticles ZnO (ICDD#897102). Peak other identified at 28, 31, 41, and 51° disappeared along with enhancement concentration precursor and associated with existing KCl, K_2SO_4 , and KZnO_2 in powder as results of the K, S, and Cl ions present in composition experience *athrixia phylicoides*. Using the Scherrer equation, the obtained crystals range between 24.53 and 63.02 nm.

Furthermore, the analysis is quantitative, with the refinement of XRD data using the Rietveld method and device soft Rietica. Crystal model results refinement uses the Rietveld method with phase parameters used, namely parameters according to research by Sawada *et al.* (1996) where mark $a = b \neq c$ equal to 3.2523 and 5.2096 Å, likewise with $\alpha = \beta \neq \gamma$ of 90° and 120°, results refinement served in **Figure 4**. Refinement involves 15 parameters.

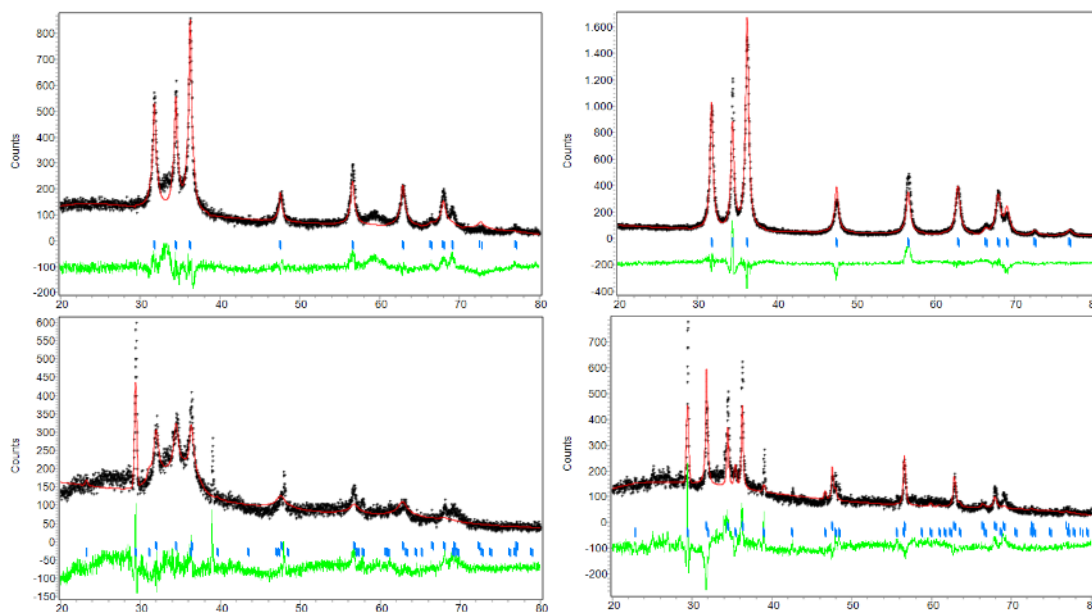


Figure 4. Refinement results Rietveld method (a) $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ 0.2 M (b) $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ 0.4 M (c) $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ 0.6 M (d) $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ 0.8 M.

Table 3. Nanoparticle XRD data refinement parameters ZnO

Sample	R_p	R_{wp}	R_{exp}	GOF	R_B
A	10.79	14.95	9,28	2.597	5.86
B	11.63	15.26	9.31	2.689	6.96
C	12.21	15.45	9,23	2.802	4.02
D	14.27	17.71	9.47	3.495	11.23

After refinement with the Rietveld method, which produces output files in the form of parameters R_p , R_{wp} , R_{exp} not enough than 20% and GOF less than 4% so this value satisfies standard the Rietveld method, as shown in **Table 3** parameter refinement results. One of the refinement outputs is the obtained parameter cells; the following is a table between parameter cells $a = b$ and c , which are presented in **Table 3**.

Table 4. Nanoparticle Parameter Cell ZnO

Sample	Grid Parameters		
	a	b	c
0.2 M	3,249	3,249	5,207
0.4 M	3,253	3,253	5,208
0.6 M	3,253	3,253	5,237
0.8 M	3,253	3,253	5,211

Based on **Table 4** shows that parameter cell on phase *Zincite* and *Nitratine* because grid parameter cells $a = b \neq c$. The lattice parameter values used are from COD 901162, where cell parameter $a = 3.249 \text{ \AA}$ and parameter $c = 5.207 \text{ \AA}$. Seen that **Table 4** parameter cell $a = b$ experiences enhancement along addition variation concentration of $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ 0.2 M to various concentration of 0.8 M and on the c axis is visible experience enhancement from addition variation concentration of $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ 0.2 M to various concentration of 0.6 M which decreased with variation concentration of 0.8 M.

This research results have parameters similarity grid parameter values with RRUFF ID R050394 occurs in the 0.2 M sample, namely lattice parameters $a = 3.249 \text{ \AA}$ and $c = 5.207 \text{ \AA}$, the more variation $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ increases so parameter cells a and c experience increase.

3.3 Analysis Scanning Electron Microscopy (SEM)

Figure 5 shows the results of picture morphology from the sample with a concentration of 0.4 M. When analyzing, SEM is required to process the data from the SEM results, and the device software used is ImageJ with the 64-bit version of Java 1.8.0_172.

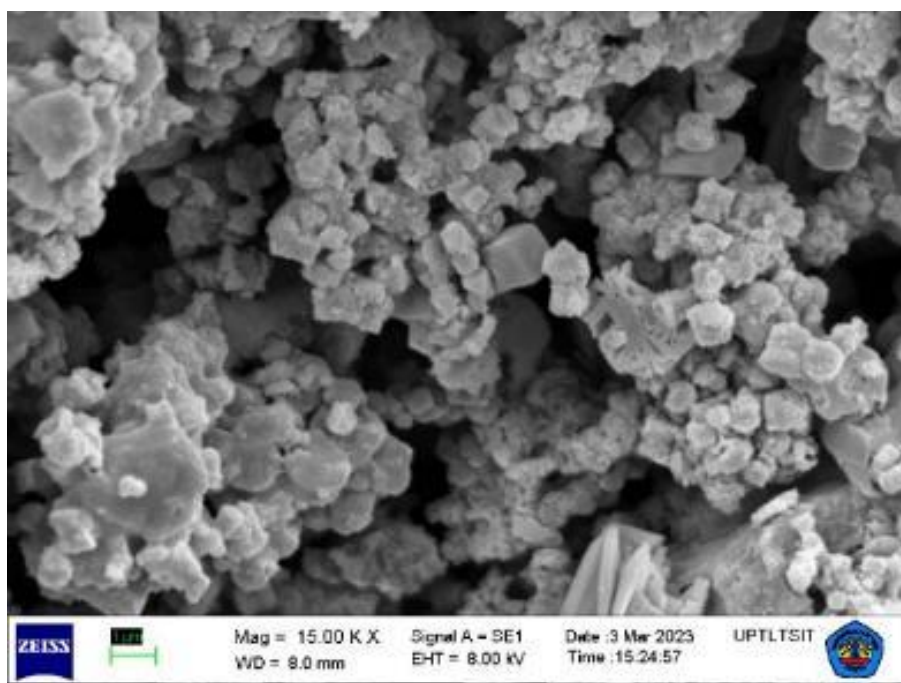
**Figure 5.** Morphology sample concentration $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ 0.4 M.

Figure 5 shows particles that do not even present lumps in the sample. An interesting thing from this picture is that morphology particles show little grains; some items are shaped round, and some inclined items form a cube. There is a distribution of particles that do not equal because they exist, which causes the size of the particle to increase. According to the study by Puspitasari (2018), agglomeration in the morphology surface particle is caused by a concentration extract and no calcination process, so there is still a coating extract particle.

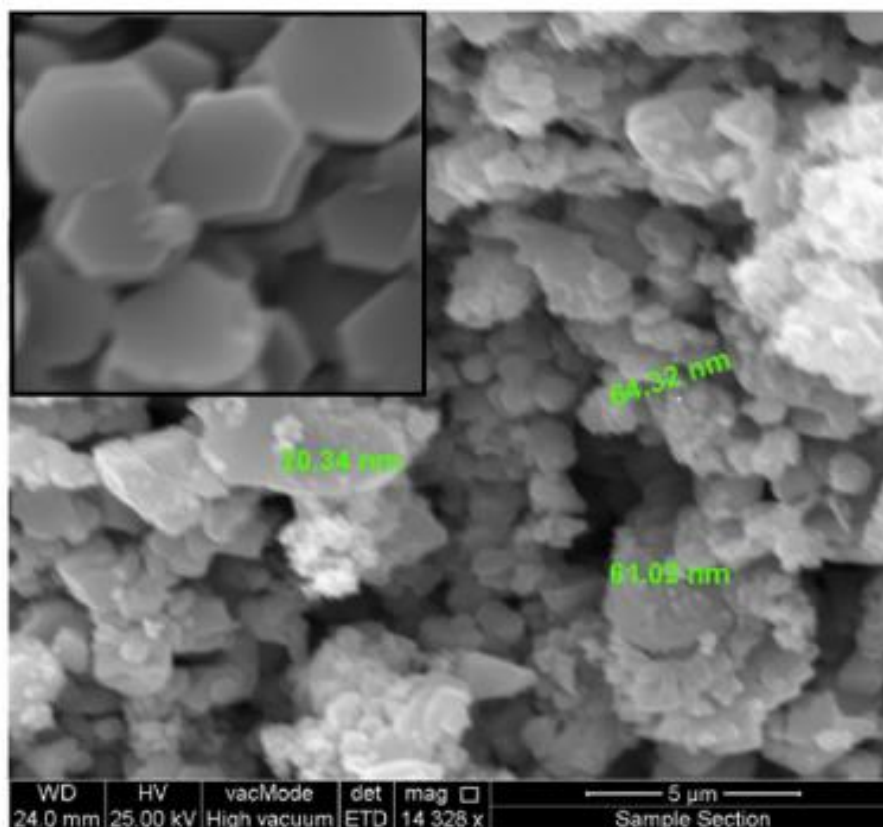


Figure 6. Morphology nanoparticles ZnO use extract leaf *Eucalyptus globules*.

Morphology in research Barzinjy and Azeez (2020) use extract leaf *Eucalyptus globulus* shown in **Figure 6** that part big nanoparticles ZnO is in nanometer scale and shape hexagonal with an average diameter of 35 nm. In addition, nanoparticles ZnO will experience A little agglomeration using method synthesis green. This is due to nanoparticle synthesis of green's broad, more surface and long-lasting affinity that causes aggregation or agglomeration. Factors are ecologically very influential in the stability of nanoparticles and agglomeration. Thus, during the forming process nanoparticles, nanoparticles stick to One another and collectively form an asymmetrical group.

4. Conclusions

Based on the results of research that has been obtained, it can be concluded that the additional concentration of $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ does not influence group function sample nanoparticles ZnO, cluster functions contained in the sample, namely OH, C=C, NO, C=O, CN, Zn-OH, and ZnO, decreasing intensity peak in the cluster hydroxyl (OH), increase size particle sample nanoparticles ZnO and its emergence new phase NaNO_3 at concentrations of 0.6 M and 0.8 M. In SEM with variation $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ 0.4 M method synthesis green shows form particle No even and present lumps in the sample.

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