Synthesis of Zinc Oxide Nanoparticles with Banana Peel Extract (BPE) from Musa Corniculata (*Pisang Tanduk*): Effects of Precursor Concentration and Reaction Temperature

Nor Azmah Kamaruddin, Rabiatul Adawiyah Abdol Aziz

Faculty of Chemical Engineering, Universiti Teknologi MARA

***Abstract*— Synthesis of zinc oxide nanoparticles (ZnO NPs) has become intention to the researchers as advanced technological materials that have unique structure and also their electrical and optical characteristics give many beneficial applications to the industry. However, the used of conventional synthesis methods often required usage of reducing and stabilizing agent that are non-benign and highly toxic to the environment. Thus, green approach method is used in this study that focusing on using plant extract which is banana peel extract (BPE) from Musa Corniculata. Zinc acetate dehydrate is used as the precursor to synthesis the ZnO NPs with different concentration and reaction temperature. There were five analysis equipment used to characterize the ZnO NPs which are X-ray Diffractometer (XRD), UV-visible Spectrometer (UV-Vis), Fourier Transform Infrared Spectroscopy (FTIR), Brunauer-Emmett-Teller (BET) and Zeta Potential Nanosizer. From XRD result, it shown that the crystallite size of NPs is in between 13 to 16 nm while BET indicated that the surface area of particle plays an important role because some of the particle were identified as NPs but they had small surface area that contribute to the larger value of average particle size. Beside, from Zeta Potential Nanosizer analysis, there were samples that meet the criteria as monodisperse while other samples may not suitable for this measurement which indicate as polydisperse The band gap calculated from absorbance wavelength of UV-Vis is ranging from 3.28 eV - 3.41 eV. FTIR analysis revealed the stretching vibrational bond of Zn-O which allocated between 400 and 600 cm^{-1}. As conclusion, the green synthesis method use is successfully conducted by using banana peels extracts as it can produce high yield of zinc oxide in a short period of time, thus it can help industry to achieve higher profit with the low time consume in the production line.**

Keywords*— Green synthesis, banana peel extract (BPE) zinc acetate dehydrate, metal oxide nanoparticles. biological synthesis.*

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I. INTRODUCTION

In the past decades, nanotechnology is widely used in the field of material science. Recently, due to the desirable properties and applications of nano-sized particles in different areas such as catalysts, highly functional and effective devices made those the semiconductor materials to the one's interest [1]. ZnO NPs can be obtained via either top-down or bottom-up processes. Top-down process also called physical methods involved the breaking down of bulk into small using mechanical energy such as lithography whereas bottom-up process also called chemical methods involved the joining atom by atom forming nano-sized particles [2]. The use of non-benign stabilizing agent (restrict nanoparticles from agglomeration) and reducing agent (reduction of metal ion) may harmful and dangerous to human being and environment [3]. Besides, this methods are also costly, high energy consumption and high possibility for the formation of toxic by-product [4]. Therefore, many researchers come out with the greener method that give good impact towards our environment and health. Meanwhile, it is a very good practice in the sense of fully utilize the waste generate and reduces their impacts to the environment that highly fulfilled the green chemistry aspect.

In this modernization era, the green approach method become more popular since it can be done in a simplest way, inexpensive and also use the environmental friendly materials which do not give harm to the living things. This methods is considered as biological method because it do not involve any chemicals that will produce toxic by-products. Literally, the plant extracts have been reported to be more advantageous over the microbes as an inexpensive in cost due to isolation, cultivation and maintenance. In addition, by using plant extracts, it give more advantages over the chemical and physical method since it is low in price which can be obtained from natural sources and can produce ZnO in short duration of time [3].

There are several research on the biosynthesis of ZnO NPs by using plant and fruit extracts and also agricultural waste, for examples, *Phyllanthus Embilica Stem* [5], *Sesbania Grandiflora Leaf* [6], *Green Tea Leaf* [7] and *Garcinia Mangostana Fruit Pericarp* [8]. Moreover, the researchers believed that extraction from plants and fruits contains higher organic compounds such as polyphenols that can act as reductants while the stabilization particles will be help by amines and carboxyl groups in order to convert metal compounds into specific NPs [9]. Hence, in continuation of study, researchers have been attracted to the advantageous of banana peel extracts over other plants and fruits extract [10]. This is because, BPE has been reported that it can serve as the reducing agent and macromolecules such as pectin, lignin and also hemicellulose with stabilizing properties since it contained a huge amount of dopamine and L-DOPA which specifically classify in the catechol functional group that contained high antioxidant activity as well as other bio-components [11].Thus in this research of study, Musa Acuminata is used in order to study the synthesis of zinc oxide by using green method in

various parameters which are reaction temperature and precursor concentration consideration. This is because the most important factor that affects the reading of the data is concentration of additives and synthesized temperature [12].

II. METHODOLOGY

A. Chemicals and Materials

Zinc acetate dehydrate ($Zn(CH_3COO)_2.2H_2O$) in powder form with a molecular weight of 219.51 g/mol and sodium hydroxide (NaOH) pellets with a molecular weight of 40.0 g/mol were purchased from R&M Chemicals while the banana peels were obtained from the local food stall vendors in front of Giant Seksyen 7 Shah Alam, Selangor, Malaysia. All the chemicals used in this study were analytical grade.

B. Preparation of Banana Peel Extract

The banana peels were washed with tap water in several times to remove any particulate matter and also other impurities. Then, the peels were dried for a night before cutting it into pieces. About 50 g of the peels were weighed before adding into 600 mL beaker containing 500 mL of ultrapure water. Then the mixture was heated at 70ºC with 1000 rpm for 30 minutes until the water turned into brownish colour. The extract then was cooled at room temperature, filtered and stored for further experimental analysis.

C. Green Synthesis of Zinc Oxide Nanoparticles (ZnO NPs)

The synthesized of ZnO NPs was obtained by pouring 20 mL of BPE with 180 mL of zinc acetate dehydrate solution that first been prepared with various concentration. The measurement used was considered the ratio 1:9 of banana peel extract to zinc acetate dehydrate solution in 400 mL beaker. Next, 1 M of NaOH solution was added dropwise into the mixture until reached pH 12 before heating up for an hour at 70ºC with 1000 rpm by using double boiled technique. After that, the precipitate obtained was filtered and dried in the dryer at 40ºC until the moisture left while the white precipitate formed. Table 1 shown the summarization for each parameter that kept as constant and manipulated due to the different precursor concentrations and also different reaction temperatures used.

Table 1: Parameter used in synthesizing ZnO NPs

Constant Variable	Manipulated Variable
pH = 12 Reaction Time = 1 hour Temperature = 70ºC	Precursor Concentration (M) 0.2, 0.1, 0.05, 0.025, 0.1
pH = 12 Reaction Time = 1 hour Precursor concentration = 0.1M	Reaction Temperature (ºC) : 50, 60, 70, 80, 90

D. Characterization of Zinc Oxide Nanoparticles

The characterization of the synthesized nanoparticles from different concentration of the precursor was characterized by using six analysis equipment. The functional group present in the synthesized ZnO NPs were analyzed by using powder sample and were recorded by FTIR spectrometer with detector at region of 4500-500 cm^{-1} with 4 cm^{-1} resolution. Next is ZnO NPs were characterized by Shimadzu XRD 6000 Diffractometer equipped with a Cukα (K=1.54 Å) radiation [13]. This analysis can be accomplished by maintaining applied voltage of 40 kV while current at 30 mA, wavelength of X-Ray beam (λ) with speed of 2.0min^{-1} and between 20º to 80º angles of the scanning range [1]. The crystalline domain diameter (D) was obtained from XRD peaks using Debye-Scherrer's equation.

$$D = \frac{K\lambda}{\beta cos\theta} \quad \text{Eq. (1)}$$

Where,
- K = Sherrer constant (typical value is 0.89)
- λ = Wavelength of the incident X-Ray beam, Cukα (1.54 Å)
- θ = Bragg's diffraction angle
- β = Full width at half maximum (FWHM) of diffraction peak (in radian)
- D = Crystalline size diameter

According to Dobrucka et al., (2016) the optical properties of ZnO NPs were studied and recorded via Perkin Elmer Lambda 35 UV-Visible Spectrophotometer scanning in the wavelength range of 200 nm to 800 nm by using deionized water as a blank [14]. The band gap energy, Eg is important to determine electrical conductivity of ZnO NPs. This value is calculated by using Eq. (2) [2].

$$E_g = \frac{hc}{\lambda} \quad \text{Eq. (2)}$$

Where,
- Eg = Band gap energy, (eV)
- h = Planck's constant, (6.626 x 10^{-34} Js)
- c = Light velocity, (3.0 x 10^8 m/s)
- λ = Absorption wavelength, (nm)

Besides, the value of surface area and particle size of synthesized ZnO NPs were characterized by using BET analysis and be calculated by using Eq. (3) [15].

$$D_{BET} = \frac{6000}{Sp \times \rho} \quad \text{Eq. (3)}$$

Where,
- D_{BET} = Average particle size (nm)
- ρ = Density of ZnO particle, (5.606 g/cm^3)
- Sp = Surface area of particle (BET surface area) in m^2/g.

Last but not least, Zeta potential nanosizer was used to identify the stability of the particles which it measure the effective electric charge on the nanoparticle surface [16]. Generally, sonification and adding a stabilizer are the two ways suggested to influence the particle size distribution [17].

In this research, 0.01 g of samples were diluted in 10 ml of additives solutions and sonicated for 15 to 30 minutes [18]. After that second dilution was done with consideration of 9:1 of ethanol and samples before being analyzed by zeta potential nanosizer. The aim of sample preparation is to break up flocs and disperse the particles since dispersant also plays very important role to determine the particle size distribution [16].

III. RESULTS AND DISCUSSION

A. Functional Groups of Synthesized ZnO NPs

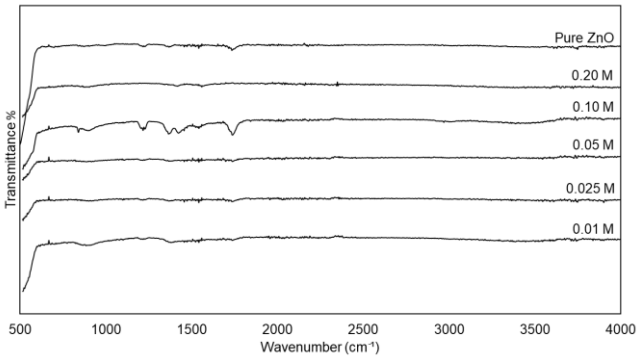

Figure 1(a)

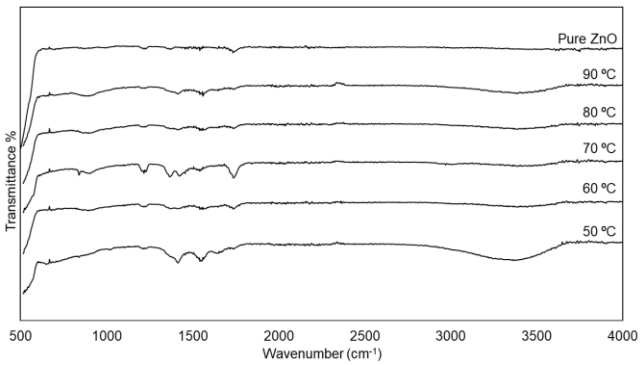

Figure 1(b)

Figure 1: (a) and (b) are the Fourier Transform Infrared Spectroscopy (FTIR) spectra of synthesized ZnO NPs with BPE from Musa Acuminata

The FTIR transmission spectra of synthesized ZnO NPs in different precursor concentrations and various reaction temperature used were shown in Figure 1 (a) and 1 (b). The absorption bands which also known as vibrational bands and the chemical compound in the sample were the two readings that being interpreted by the reading of the IR spectrum [14]. This is the important role in studying the reduction and stabilization process of green synthesis nanoparticles. The FTIR spectrum was recorded in the range of 500 – 4000 cm^{-1} [14]. Bands that shown in between 3500 cm^{-1} to 3200 cm^{-1} are belongs to O-H stretch from alcohol, phenol or maybe some excess water remain on the NPs, 1750 cm^{-1} – 1735 cm^{-1} correspond to the esters functional group of C=O stretch while C-H in-plane bend that refer to alkenes group is at 1430 cm^{-1} - 1290 cm^{-1} and carboxylic acid is appeared at stretching band of 1320 cm^{-1} – 1210 cm^{-1} which refer to C-O stretch vibrational stretching band [1]. In addition, based on the several research had been done, the significant band appeared at 511 cm^{-1} that referred to presence of zinc oxide [1]. Furthermore, this can be confirmed as Zn-O bond since Yuvakkumar et al., 2015 also stated that the peak for Zn-O also be allocated between 400 and 600 cm^{-1}. As concluded by Dhanemozhi et al., (2017), the reduction process is occur when presence of phenolic group of molecules while the amino acids and amide linkages in protein are responsible for the stabilization of the ZnO nanoparticles [7].

B. Crystalline Characteristics of Synthesized ZnO NPs

In order to further confirm the structure of the ZnO NPs, X-ray Diffractometer (XRD) analysis was taken into consideration. The pattern of ZnO NPs is shown in Figure 2 and peak spectra (1 0 0), (0 0 2), (1 0 1), (1 0 2), (1 1 0), (1 0 3), (2 0 0), (1 1 2), (2 0 1), (2 0 2) and (0 0 4) consistently followed the standard of JCPDS Card at references code (00-036-1451). Debye-Scherrer's equation at Eq. (1) was used to obtain the size of ZnO NPs and the calculated data obtained is tabulated in Table 2(a) and Table 2(b).

Table 2(a): Average crystallite structure size of ZnO NPs for different precursor concentration

Precursor Concentrations (M)	Average crystallite structure size (nm)
0.010	14.99
0.025	14.43
0.050	14.74
0.100	14.74
0.200	15.34

Table 2(b): Average crystallite structure size of ZnO NPs for different reaction temperature

Reaction Temperatures (°C)	Average crystallite structure size (nm)
50	13.52
60	15.21
70	14.74
80	15.16
90	15.77

From the calculated data, the average crstallinity of particles to be in the range of 13 nm – 16 nm. Moreover, due to the observation values, it greatly showed that the structure for the ZnO NPs is belong in the hexagonal wurtzite structure. As mentioned by Vishwakarma (2013), XRD spectrum which indexed (102) of the wurtzite crystal structure of ZnO had showed at diffraction peaks around 37° [20]. This structure indicates that the synthesized ZnO NPs have good crystallinity structure and were clean from the impurity peak [19]. However, Wong (2016) stated that as the minor peak in between 60° to 80° presents indicate the existence of water soluble and also water insoluble impurities [1] during preparation of samples that present on the surface of synthesized nanoparticles [20] while the extra peak present in between 30º to 50º because of incomplete reaction of the produced samples [21].

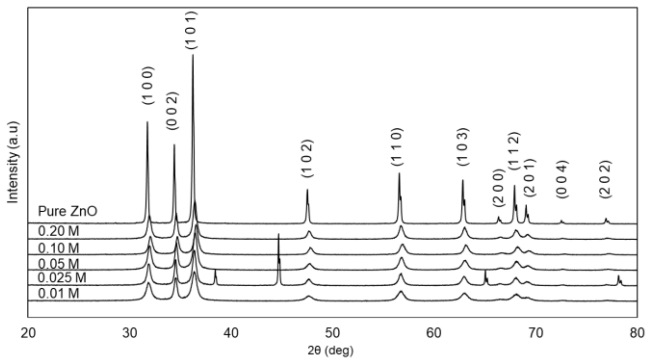

Figure 2(a)

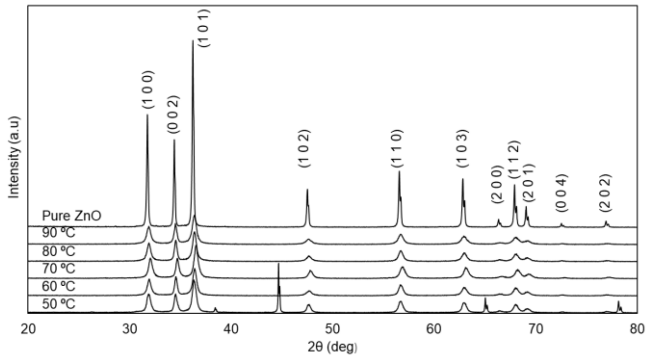

Figure 2(b)

Fig. 2: XRD diffractogram of Synthesized ZnO NPs of 2θ in between 20° to 80° : (a) ZnO NPs crystallinity due to different precursor concentration, (b) ZnO NPs crystallinity size based on different reaction temperature

Generally, the broadening of the peaks in the Figure 2 (a) and (b) of XRD pattern can be attributed to the small particle size of the synthesized ZnO Furthermore, the sharpness of the peaks shows good crystal growth of the oxide particles [22] since broader peaks reflect the effects due to experimental conditions on the nucleation and growth of the crystal nuclei. Thus, XRD results also suggested that the crystallization of the bio-organic phase occurs on the surface of the ZnO NPs or vice versa [21].

C. Band Gap Energy of Synthesized ZnO NPs

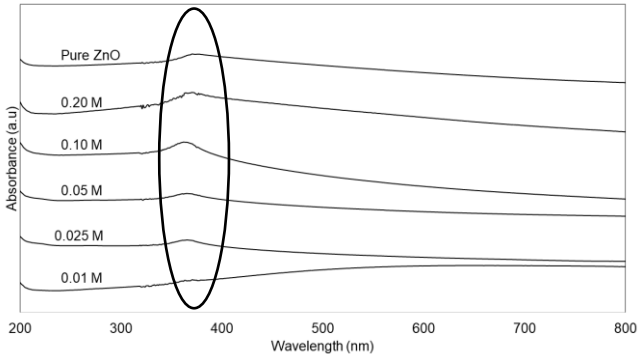

Figure 3(a)

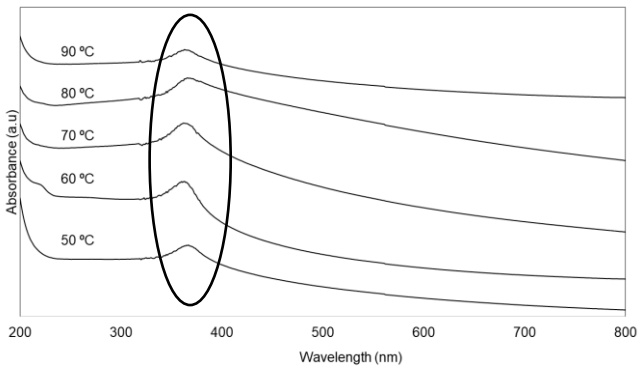

Figure 3(b)

Figure 3: UV-Visible (UV-Vis) spectrum of synthesized ZnO NPs,(a) Effect on Precursor concentration, (b) Effect on reaction temperature of solution

UV-Vis is the most known method for structural characterization of nanoparticles in order to confirm that ZnO NPs exist in the prepared samples. Figure 3 (a) and (b) shown an intense absorption peak observed in UV-Vis spectrum under ultraviolet (UV) region of 200 nm to 800 nm. The absorption value at the chosen wavelength can be related to the band gap energy (E_{bg}) between valence band (VB) and conduction band (CB) of the synthesized semiconductor. From Table 3, it can be observed that the nearest and similar value calculated to the theoretical value of band gap for ZnO NPs (3.37 eV) [1] are at 0.01 M and 0.05 M while for the reaction temperature, the value of 3.37 eV are at 50°C and 80°C. No other peak was observed in the spectrum, confirming that the synthesized product was ZnO only. ZnO NPs have been reported to exhibit a characteristic broad absorption peak between 330-460 nm [23]. Thus it can be deduced from Figure 3(a) and (b) that the biomolecules present in the plant extract induce the reduction of Zinc ions into ZnO NPs. This process of reduction is extracellular, fast and thus can be developed into an easy method for nanoparticle synthesis.

Table 3: Band Gap calculated data using Eq. (2) for each parameter

Precursor Concentration (M)	**Peak (nm)**	**Band Gap (eV)**
No BPE	377	3.28
0.010	368	3.37
0.025	367	3.38
0.050	368	3.37
0.100	365	3.40
0.200	373	3.32
Reaction Temperature (°C)	**Peak (nm)**	**Band Gap (eV)**
50	368	3.37
60	364	3.41
70	365	3.40
80	368	3.37
90	366	3.39

D. Particle Size of Synthesized ZnO NPs

Brunauer-Emmett-Teller (BET) was used to measure the specific surface area of ZnO NPs by nitrogen adsorption temperature at 77 K. This also include the pore size distribution at different precursor's concentration and also difference reaction temperature parameter. The calculated values were tabulated in the Table 4(a) and Table 4(b) respectively with the surfaces area resulted from the analysis. The particles size were calculated by using Eq. (3).

Table 4(a): Calculated value on average particle size for different precursor concentration

Precursor Concentrations (M)	0.01	0.025	0.05	0.1	0.2
Average Particle size (nm)	139.97	158.89	49.17	50.48	71.03

Table 4(b): Calculated value on average particle size for different reaction temperature

Reaction Temperature (ºC)	50	60	70	80	90
Average Particle size (nm)	55.65	64.56	50.48	55.73	69

From the result, it shown that the reaction temperature and also concentration of precursors were two important factors caused the different surface area value of ZnO NPs. The surface area rate were

fluctuated due to the heating temperature. As the temperature increased, the surface area became decreased when the comparison made between 50ºC and 90ºC. According to Wurster 1995, there were some possible causes for the decreases in surface area which are changes of crystal structure and particles' pores might be collapsed. On the other hand, the mean particle size of ZnO NPs calculate from BET result is higher than the crystallite size obtained from XRD analysis, however it still in the range of nano-size which is less than 100nm [24].

In addition, from the data tabulated in Table 5 which being analyzed by using Zeta Potential Nanosizer, ZnO shown great differences in the particle size distribution varying the concentration of precursor and also reaction temperature. The value of Z-average which also known as particle diameter were fluctuated in both result together with polydispersity index (PdI). The size recorded at 70ºC was 925.5 nm while at 0.2 M of zinc acetate dehydrate was 879.3 nm were the minimum particle diameter that can be achieved through the raw correlation data obtained. This system is classify as monodisperse because the mean effective diameter of the particles can be determined [18]. On the other hand, some of the result tabulated have higher Z-average value and this indicate that the sample is polydisperse and may not suitable for dynamic light scattering (DLS) measurements [21]. The sample also may contains large particle and dust or the aggregation was occurred. As discussed by Marsalek (2014) if particles aggregate, there will be a larger population of particles with a larger radius. Therefore, selecting and setting the methodology used with the proper type of dispersant is very important [18] to prevent agglomerated of synthesized ZnO NPs occurred.

Table 5: Data tabulated from Zeta Potential analysis on particle distribution intensity and average size on the effects of varied concentration of the precursor and reaction temperatures.

Reaction Temperature (°C)	Z-Average (nm)	Particle Distribution Intensity (PdI)
50	1876	0.272
60	4971	0.072
70	925.5	0.241
80	1404	0.488
90	3635	0.456
Precursor Concentration (M)	Z-Average (nm)	Particle Distribution Intensity (PdI)
0.01	2429	1.000
0.025	1019	0.395
0.05	1137	0.349
0.1	1937	1.000
0.2	879.3	0.330

IV. CONCLUSION

In this study, ZnO NPs has been successfully synthesized through a green synthetic pathway with the aid of Musa Acuminata BP extract as reducing agent as well as stabilizing agent. The synthesized ZnO NPs was characterized using XRD, FTIR, UV-Vis spectroscopy, BET and Zeta Potential Nanosizer. The crystalline size calculated from Debye-Scherrer's Equation according to the highest diffraction peaks in the XRD diffractogram is in the range of 14 to 16 nm while from BET surface area result, it revealed that some of the nano size particles at XRD is still has small surface area that contributed to the larger particle size. Generally, by UV-Vis spectroscopy, it can be deduced that the biomolecules present in the plant extract induce the reduction of Zinc ions into ZnO NPs at which the nearest and similar band calculated from theoretical band gap are at 3.37 eV at 0.01 M and 0.05 M while 3.37 eV also at 50°C and 80°C. Beside, Zeta potential nanosizer analysis result on particle distribution intensity and Z- average size make the justification strongly on the existence of nanosize particles due to the BET calculated result of average particle size (nm). Moreover, FTIR identified some of the vibrational bond at defined wavelength that referred to the possible biomolecules responsible for the reduction of ZnO and capping agent of bio-reduced ZnO NPs.

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