SYNTHESIS OF ZINC OXIDE NANOPARTICLES (ZnO NPs) WITH BANANA PEELS EXTRACT (BPE) FROM JACKFRUIT BANANA: EFFECT OF pH AND REACTION TIME.

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Abstract*—This present study is about synthesis ZnO NPs with Banana Peels Extract (BPE) from Jackfruit Banana by varying pH and reaction time. This method on producing ZnO NPs by using eco-friendly method especially biological method which are less harmful and BPE is choose as reducing agent and act as stabilizer. The samples were then characterized to identify the optimum condition found at pH 12 and 60 mins . The results Fourier Transform Infra Red (FTIR) Spectroscopy shown ZnO bonds allocate at range of 500-571 $cm^{-1}$for all sample, while for UV-Visible (UV-Vis) the band gap energy was found in between 3.34eV and 3.43eV. Furthermore, by using the X-ray Diffraction (XRD) determine the crystalline size within the range of 12.11nm to 16. 32 nm for pH. The average crystallite size at pH 12 was 14.16 nm while at 60 min was 14.16nm smallest particle size for reaction time. Brunaner-Emmet-Teller (BET) analysis shown all the samples were nanoparticles because less than 100 nm size of particle. Lastly, for Zeta- Potential analysis, the sample size is larger than 100nm because sample were highly agglomerates.

INTRODUCTION

Nanotechnology is the innovation technology in the 21st century. The research development of nanotechnology field is rising rapidly in the world. It is also playing important roles in few fields such as pharmacology, agriculture. The continues of research about nanotechnology leading to nanoparticle base. The varies of potential technology applications of metal oxide NPs like solar cells[1], photodetector[2], photocatalysis[3], laser diodes[4] made nanoparticles getting more attention among researchers. [5]. Additionally , its applied in including sensors, energy storage, optics, coatings and biotechnology. [6].

Zinc oxide nanoparticles (ZnO NPs) is known as important class metal oxide materials because of their extraordinary physical, chemical , electrical, biomedical, optical properties. [6]. Its also exhibit interesting characteristics like good adsorption of dye in waste water treatment and high catalytic efficiency[7]. Todays, most studied application of ZnO is in biosensing because have good criteria like fast electron transfer kinetics, biocompatibility and high isoelectric point (9.5). [8]. Furthermore, the important role of Zn as a micronutrient in the human health over maintenance of DNA structures, proper function in immune system, its antioxidant activity has reported by previous study by the both in vivo and in vitro studies. [9]. This nanoparticles revelation is new and better-quality properties with larger particles of the bulk material and properties are derivative due to the difference in specific characteristics such as distribution, size and morphology of the particles. Also, its comes out with higher surface area to volume ratio with reduction of distribution , size and morphology of the particles.[10] Other than that it can increase the conductivity .[11]

Moreover, various method in synthesis of ZnO NPs using physical, Biological and chemical processes. Examples of physical method such as Laser Abletion, ultrasonication, photoirradiation, radiolysis, vaporization and many else. While for chemical such as Sol-gel, Solvothermal, Co-precipitation, chemical reduction and reverse micelles. Biological method divided to two techniques which are Biosynthesis (Bacteria, Fungi, Alga, Plant extract, Amino Acids Peptides) and Biomimetic (DNA/RNA, Proteins, Viruses, Pollen). Physical and chemical processes are commonly used technique to produce ZnO NPs. According to the Aziz & Karim (2019), the difference between these two processes are physical needs high energy and high vacuum leads to high cost while for chemical is cost effective and suitable for huge production line.

Therefore, this situation leading needs to find another technology that greener. In this favor using green methods in the synthesis of ZnO NPs has become topic of attention because the chemical process needs chemical compounds/ organics solvents as reducing agent. [10]. Thus, a classical example of biosynthesis raw material abundantly available is Banana. On the general available literature, we dedicated the banana peels is rich in polymers such as lignin, hemicellulose and pectin's. [12]. Due to the composition in banana peels, it was decided to used banana peels extract to produce ZnO NPs.

Various factors such as pH, reaction time, temperature and concentration can affect the synthesized of ZnO NPs. Morphology such as shape and size nano particles are controlled by controlling pH of the solutions. The H+ or OH- ions plays important role when this ratio of ions affects polymerization of the metal oxygen during synthesis , hydrolysis and condensation of the solutions. [13].

Here in this paper, synthesis and characterization of ZnO nanoparticles using bio synthesis process plant extraction method from Banana peel waste has been reported. The effect of varying pH and reaction time on the particle size of the synthesized ZnO NPs were studied. In addition, the optimum pH and reaction time to produce ZnO NPs will be determined.

I. METHODOLOGY

A. Material and Chemicals

Zinc acetate dehydrates ($C_4H_6Zn_2H_2O$) from R&M Chemical was used as the precursor. Ultrapure water was used as the synthesis medium. While, Jackfruit banana peels was collected from stall vendor at section 7, Shah Alam, Selangor.

B. Preparation of peels extract

Banana peels were washed several times with tap water to remove any particulate matter or dust and were cut into small pieces. Then, the banana peels and ultrapure water were mixed at ratio 1:10 for extraction process. Next, the mixture is covered with aluminum coil and heated at 70°C and 1000 rpm for 30 minutes. After a light brown were appeared, the solution was cooled at room temperature. Then the banana peels extract (BPE)was filtered using filter paper and stored in bottle. After that, put in the regenerator at 4°C for further used.

C. Synthesis of ZnO NPs

The synthesis of ZnO NPs were experimented by added 0.1M Zinc Acetate dehydrate ($C_4H_6Zn\ 2H_2O$) into BPE at ratio of 9:1. Then, the solution was prepared at different pH values which namely 7, 9, 11, 12 and 13 by adding 2M Sodium Hydroxide (NaOH). The solution was heated at 70℃ for 1hr by double boiled method under continues stirring at 1000 rpm. For reaction time the solution was prepared at pH 12 by adding 2M Sodium Hydroxide. Then heated the solution at 70℃ for 30, 60, 90, 120, and 150min of reaction time also used double boiled method under continues stirring at 1000 rpm. Make sure in double boiled process, the temperature of solution was maintained along the time. Cool down the temperature of solution before filtered. Then, the solution was filtered with filter paper. White precipitate was obtained and dried it at 40℃ for overnight. The white powder of ZnO NPs was ready to analyze.

D. Characterization of Synthesized ZnO NPs

The structural properties of the sample were characterized using X-ray diffractometer (XRD: 20-80 pattern) at 40kV and 40mA using X'Pert PRO diffraction to determine the phase identification of crystal and determine the dimension of the crystal present in the sample. While for the Fourier Transform Infra-Red Spectroscopy FTIR (300-4000 cm^{-1} spectra range) by PerkinELmer (model: Spectrum one) to analyses the functional group and element presence in the sample. The UV-vis spectrometer by PerkinElmer (model: Lambda 750) used for determining the adsorption capacity carry 60 Agilent within of a wavelength range of 500-800 nm. The Zeta-potential by Malvern (model: ZEN 3600) to measure the nanoparticle size. The specific surface area and pore size distribution each sample were investigated by Brunauer-Emmet-Teller (BET) model micromeritics.

II. RESULTS AND DISCUSSION

A. Effect of pH and reaction time on the Zinc Oxide Nanoparticles (ZnO NPs)

This biosynthesis process will form precipitate as solid phase reaction. According to the Wahab et.al (2009), when the reaction of aqueous solution zinc ($C_4H_6Zn.2H_2O$) with salt (NaOH), forming compound that has low solubility white precipitate from the solutions. The chemical reactions between ($C_4H_6Zn.2H_2O$) as precursor and NaOH was used to control the aqueous pH were showed in the following reactions.

$$Zn(CH_3COO)_2.2H_2O + 2H_2O + 2NaOH \rightarrow Zn(OH)_2 + 2CH_3COONa + 2H_2O$$

$$Zn(OH)_2 + 2H_2O \rightarrow Zn(OH)_4^{2+} + 2H^+$$

$$Zn(OH)4^{2+} \rightleftharpoons ZnO + H_2O + 2OH^-$$

Therefore, in this studied the size was manipulated by controlling the pH and reaction time. The polymerization of the metal oxygen bond gets affected by solution of H^+ and OH^- ratio during the synthesis. Basically, the pH of precursor solution affects condensations and hydrolysis of the solutions [13]. The pH also can altered the number of growth and nuclei units [14]. For various in reaction time, when the reaction time is increase the particle crystalize will agglomerate.[15]

B. Fourier transform infrared spectrometer (FTIR) analysis

Fig. 1 shows the FTIR pattern of Zinc oxide nanostructures analysis as increasing pH (7,9.11,12,13) was investigated at room temperatures in range of 500-4000 cm^{-1} and the strong stretching mode of vibration of C=O was observed between 1450-1550 cm^{-1} .There were some different bands for each different pH were detected, but still has the same functional group.[14]. Furthermore, at pH 13 the bands at 3200-3500 cm^{-1} more narrower because additional amount of OH^- from NaOH reacts with ($C_4H_6Zn_2H_2O$) as correspond to the O-H mode of vibration. [14]. While for different reaction time (30,90.120,150) min were investigated were shown in Fig. 2. As we can see the strong stretching mode of vibration was observed between 1000- 1500 cm^{-1} was the bond of C-O-C stretching modes while for 1500-1800 cm^{-1} was -C-O stretching mode. The absorption peaks around 1400-1500 cm^{-1} represents the carboxylate group (COO^-).[6] According to the researchers, in the infra-red region, the characteristics Zn-O stretching mode was found between 350 – 390 cm^{-1} for pH while for different reaction time were found at 418-571 cm^{-1}. [6] [16]. Thus in this study the peak ZnO was shown at 1550-1600 cm^{-1} almost similar to other peak of pH and reaction time . The range of wavenumber in this study need to reduce to 200-4000 $cm^{-1\ s}$ scale to analyze the stretching mode of ZnO.

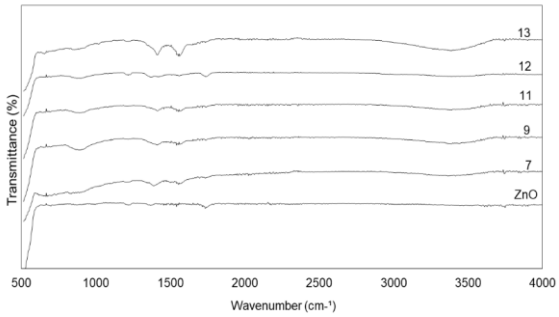

Fig.1: FTIR graph for different pH

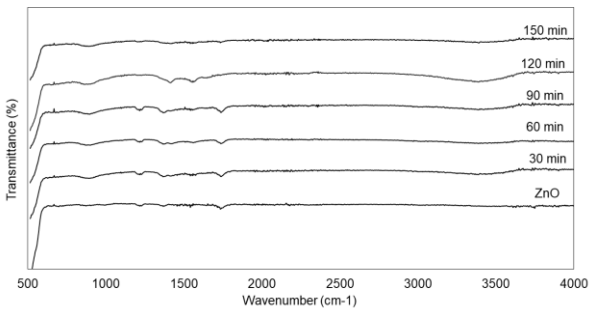

Fig.2: FTIR graph for different reaction time

C. X-ray diffraction (XRD) Analysis

Fig.3 and Fig.4 shows the X-ray diffraction pattern of zinc oxide nanoparticle of Pisang Nangka pH (7,9,11,12,13) and different reaction time (30,60,90120,150) min accordingly. X-ray Diffraction (XRD) was used in determination the crystallinity and crystal phase by Cu Kα radiation (λ= 1.5406) at the bragg angle range from 20º to 80º. All the diffraction peaks from the graph was matched with the Joint Committee on Powder Diffraction Standards for ZnO bulk (JCPDS 36-1451) with lattice constant *a*=3.249 and c= 5.206Å. The diffraction peaks were identified at lattice planes (101) (1 0 1), (0 0 2), (1 0 1), (1 0 2), (1 1 0), (1 0 3), (2 0 0), (1 1 2),(2 0 1), (0 0 4) and (2 0 2). These specifics peak indicated to the hexagonal wurtrize structures ZnO NPs. Moreover, the narrower and higher intensity of width ZnO crests indicated that the powder has virtuous crystallinity and fully-fledged along c-axis. As shown in Fig. 3, we have slightly different peaks at 40-50 (2θ Deg) and 60-70(2θ Deg) while at Fig.4 at 40-50 (2θ Deg), 60-70(2θ Deg) and 70-80 (2θ Deg) because of some noise disturbance. The diffraction peaks were affected by increasing the pH value and decreasing the intensities of diffraction line and affecting crystalline size of ZnO NPs. [5].

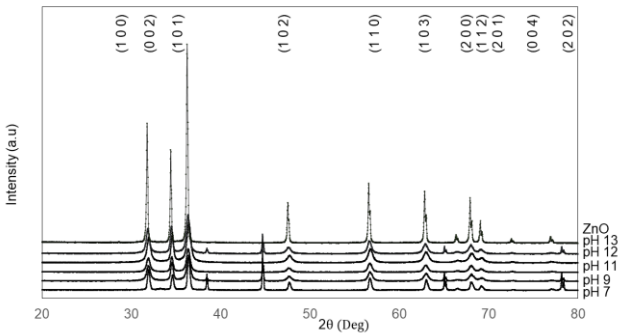

Fig.3: XRD analysis graph at different pH value

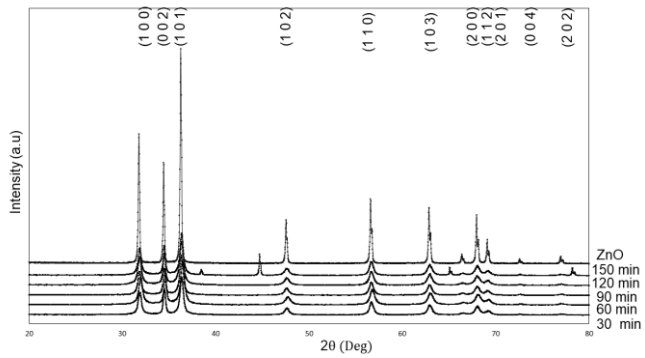

Fig.4: XRD analysis graph at different reaction time

The crystalline size, D was calculated using the Debye-Scherrer equation:

$$D = \frac{0.89\lambda}{\beta COS\theta} \qquad \text{Eq. (1)}$$

where the λ is the wavelength of X-ray used is 0.1506 nm, β is the angular peak width at half maximum (FWHM) in radians and θ is the Bragg's angle.

The crystalline size at different pH and different reaction time were reported in Table 1. As observed the crystallite size of ZnO NPs were increased at pH 7 until pH 11 and decreased at pH 12 and pH 13. This is because increasing pH value, also increases of basicity solution will smaller the tensile strain within the ZnO NPs. Also, the amount of OH- dissolved was higher, the reaction with ZnO caused dissolution and made the crystallites size and particle size decrease and agglomerated.[14] For reaction time analysis the average crystallized slightly increases as the reaction time increases. According to the researchers, this due to the increase of nucleation and growth of nanoparticles.[6].

Table 1: Crystalline size D (nm) of Pisang Nangka at Different pH and different reaction time

pH	Crystalline size, D (nm)	Reaction time (min)	Crystalline size, D (nm)
7	12.11	30	14.46
9	15.36	60	14.16
11	16.32	90	15.09
12	14.16	120	15.09
13	14.11	150	14.48

D. UV- visible spectrophotometer analysis (UV-VIS)

The UV-Vis absorption spectrum was carried out to find the potential of optical characteristics to determine the electronic structure of the synthesized ZnO NPs. Fig. 5 and Fig 6 shows theUV-Vis absorption spectra with different pH.value and reaction time at normal ranging between 200 until 800nm. From Fig. 5 all the pH value have an absorption peak at range of 350-400nm indicated that the size of ZnO NPs was in nano dimension. As can be seen at pH 11 was higher absorbance and sharpness compared to other pH value due to the intrinsic band gap of ZnO that related with electron transitions from the valence band to conduction band. The peak also indicated monodispersed nature of the nanoparticle distribution. [5]. From the Fig. 6 all the different reaction time have an absorption peak at range of 350-400nm indicated that the size of ZnO NPs was in nano dimension.

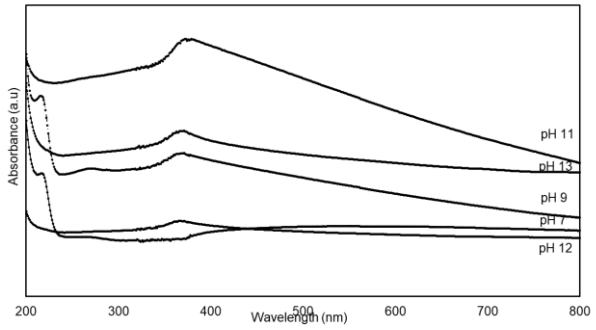

Fig.5: UV-VIS spectrometer analysis of synthesized ZnO NPs with different pH

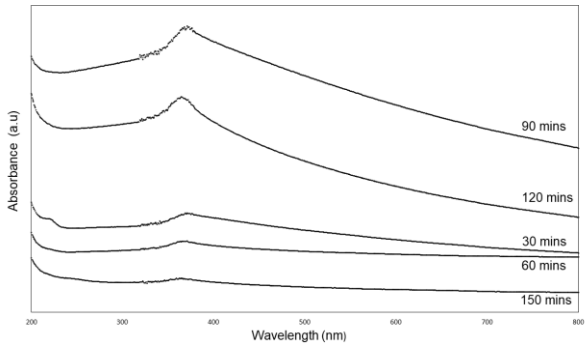

Fig.6: UV-VIS spectrometer analysis of synthesized ZnO NPs with different reaction time

According to the Dharma and Pisal (2009), there is simple equation to calculate the band gap energy from value of ZnO NPs in powder form using UV-Vis spectrometer.

$$E = hc/\lambda \qquad \text{Eq. (2)}$$

where h is 6.626×10^{-34} Joule Sec(planks constant), c is 3.0×10^{8} m sec $^{-1}$ (speed of light) and λ is 416.57×10^{-9} m (cut off wavelength) while 1 eV is equal to 1.6x10-^{19} Joule. The spectrum showed qualified absorption peak of ZnO NPs for different reaction time as shown in Table 2.

Table 2: UV-Vis absorption intensity and band gap energy of ZnO NPs at different reaction time

Parameter	Peak (nm)	Band Gap (eV)
pH 7	560.63	2.21
pH 9	370.95	3.34
pH 11	372.49	3.33
pH 12	370.96	3.34
pH 13	363.05	3.42
30 mins	371.06	3.34
60 mins	370.96	3.34
90 mins	371.07	3.34
120 mins	364.22	3.40
150 mins	361.13	3.43

According to the extensive researchers, the crystallites size affecting the band gap energy and automatically electrical performance effected because band gap is important criteria of physical characteristics any substances and the band gap energy will decrease when the pH value increases thus it means the particle size will increase[17]. But noticed in our case, the band gap is directly proportional to pH, decrease the particle size because the band gap energy value is rise. This indicated that lower contaminants and produce high purity of ZnO because the contaminants will act as dopants made the band gap energy lower, appear as small peaks or drop in the UV-vis spectrum [18]. For different reaction time the values of band gap energy are in the range (3.34-3.43eV). These obtained values are slightly different with bulk ZnO (3.37eV) because of the quantum internment effects in the prepared samples. [6] Moreover, as in the Fig 5 at pH 7 and 9 the sharp decreasing around 200 nm because of the band edge absorption or related with confinement effect of minor size ZnO. [14].

D. Brunauer-Emmet-Teller (BET) Analysis

In determined the surface area of all porous structures adsorbed in the small gas molecules, the diameter of particle size for synthesized ZnO NPs were calculated from BET surface area by using the following equation in nanometer (nm) size as below:

$$Diameter\ of\ particle\ size = \frac{6000}{BET\ surface\ area\ x\ density} \qquad \text{Eq. (3)}$$

Where the Diameter of Particle size (D_{BET}) is the average particle size(nm), ρ is the density of ZnO NPs, 5.606 g/cm^3 and the BET surface area in m^2/g. Thus, the BET surface area and calculated diameter particles size of synthesized ZnO NPs with different pH value and different reaction time were tabulated in the table 5. From the table, we observed as the surface area increases the diameter particles size of ZnO NPs were decreases. it can be concluded the BET surface area value is inversely proportional with diameter size of particles.

Table 3: BET surface area and the average particle size of synthesized ZnO NPs with different pH value and reaction time

pH	BET Surface area (m^2/g)	Average particle size (nm)	Reaction time (min)	BET Surface area (m^2/g)	Average particle size (nm)
7	15.82	14.78	30	25.06	23.41
9	32.23	30.12	60	29.34	27.41
11	24.03	22.45	90	28.96	27.06
12	29.30	27.41	120	43.64	40.78
13	40.52	37.86	150	31.32	29.27

E. Zeta potential size particle distribution analysis

The zeta potential size particle was measured by analyzing 0.01g of sample dilute by 10ml ethanol(dispersant). Then, the solutions were sonicated for 30 min and 100ppm of solutions were prepared which is 1ml of stock solution dilute with 9ml of ethanol. The particle size can get affected by dispersant, sonification and adding a stabilizer, thus ethanol was selected as dispersant because rate of coagulation is lower so can decrease size of particle than water [19]. Table 6 below shown the size of ZnO NPs result.

Table 4: Zeta potential size particle distribution e of synthesized ZnO NPs with different pH value and reaction time

pH	particle size (d nm)	Reaction time (min)	particle size (d nm)
7	901.8	30	231.8
9	791.6	60	435.1
11	432.7	90	728.9
12	435.1	120	250.5
13	404.2	150	131.3

As you can see the pattern of size decreasing as the pH value increase but dissimilar with reaction time, the size of particle is fluctuated even the reaction time increase. The smallest size depends on parameter is at pH 13 and 150min. But all the readings were more than 100nm and should be lesser 100nm because ZnO NPs were nanoparticles. The factors were the samples are not stable because of the poly dispersion value (pdi) mostly higher than 0.600. Other than that, when a solid active gradient is dispersed in a liquid, the agglomeration, flocculation (increments of size particle) and sedimentation (tendency the nanoparticles forming

aggregates or agglomerate due Vander Walls Forces or other attractive forces because of high surface area) were highly occur. [20]. Sometimes analyses presence of nanoparticles in dispersions having various of particle size can cause problem measurement technique due to aggregates of the smaller particle can cover the presence of nanoparticle.[21]

CONCLUSION

ZnO NPs have been successfully synthesized by the bio synthesis of banana peels extract at different pH value and reaction time. All the samples have good structural and optical characteristics. The ZnO NPs produced at pH 12 and 60 min of reaction time were best optical properties. The effect of pH is when the pH increases the particle size decreases. while for reaction time, The XRD showed formation of hexagonal structure of the ZnO NPs within the range of 12.11nm to 16. 32 nm for pH. The average crystallite size at pH 12 was 14.16 nm while at 60 min was 14.16nm smallest particle size for reaction time. The band gap energy was found at the range 3.34eV to 3.43eV. FTIR report all samples get show Zn-O bonding. BET analysis showed all the average size particle is less than 100nm and it was success the samples were nanoparticle. Thus, at pH 12 BET surface area was 29.34(m^2/g), While at 60 min the BET surface area also 29.34 (m^2/g) with average particle size 27.41 nm like pH 12. while for Zeta potential size particle, the analysis result of all samples were more 100nm because of the sample is highly agglomerates.

For further study, synthesis ZnO NPs for other parameters like concentration and temperature also affect the synthesis of ZnO NPs. Other than that, suggestion to increase the stability of sample, the sample need through the calcine process to reduce the energy and to prevent the sample to agglomerates. The characterization of morphology ZnO NPs need to study more details using FESEM.

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References

[1] A. Nicolaev, T. L. Mitran, S. Iftimie, and G. A. Nemnes, “Solar Energy Materials & Solar Cells Optimization of halide perovskite solar cells based on nanocolumnar ZnO,” *Sol. Energy Mater. Sol. Cells*, vol. 158, pp. 202–208, 2016.

[2] C. Park, J. Lee, H.-M. So, and W. S. Chang, “An Ultrafast Response Grating Structural ZnO Photodetector with Back-to-Back Schottky Barriers Produced by Hydrothermal Growth,” *J. Mater. Chem.*, pp. 2737–2743, 2015.

[3] A. Di, M. Cantarella, G. Nicotra, and V. Privitera, “Applied Catalysis B : Environmental Low temperature atomic layer deposition of ZnO : Applications in photocatalysis,” *"Applied Catal. B, Environ.*, vol. 196, pp. 68–76, 2016.

[4] Y. Fujita, S. Yanase, H. Nishikori, Y. Hiragino, and Y. Furubayashi, “Near ultraviolet light emitting diodes using ZnMgO : N / ZnO hetero-junction grown by MOVPE,” *J. Cryst. Growth*, vol. 464, no. November 2016, pp. 226–230, 2017.

[5] A. Aziz and A. Karim, “Precursor Concentration Effect on Physicochemical Properties of Zinc Oxide Nanoparticle Synthesized with Banana Peel Extract,” vol. 797, pp. 262–270, 2019.

[6] H. S. W. M. S. Abd, E. Sadek, and M. Henini, “Influence of reaction time and synthesis temperature on the physical properties of ZnO nanoparticles synthesized by the hydrothermal method,” *Appl. Phys. A*, vol. 0, no. 0, p. 0, 2018.

[7] Q. Tian *et al.*, “Zinc Oxide Coating Effect for the Dye Removal and Photocatalytic Mechanisms of Flower-Like MoS2Nanoparticles,” *Nanoscale Res. Lett.*, vol. 12, no. 1, 2017.

[8] R. Wahab, S. G. Ansari, Y. S. Kim, M. Song, and H. Shin, “Applied Surface Science The role of pH variation on the growth of zinc oxide nanostructures,” vol. 255, pp. 4891–4896, 2009.

[9] Z. Salari *et al.*, “Journal of Trace Elements in Medicine and Biology Microwave-assisted biosynthesis of zinc nanoparticles and their cytotoxic and antioxidant activity,” vol. 39, pp. 116–123, 2017.

[10] C. Vidya, S. Hiremath, M. N. Chandraprabha, M. A. L. Antonyraj, and I. Venu, “Green synthesis of ZnO nanoparticles by Calotropis Gigantea,” no. Ncwse, pp. 2012–2014, 2013.

[11] N. Nesakumar, J. Bosco, and B. Rayappan, “Influence of pH on Structural Morphology of ZnO Nanoparticle,” no. December, 2012.

[12] A. Bankar, B. Joshi, A. Ravi, and S. Zinjarde, “Colloids and Surfaces A : Physicochemical and Engineering Aspects Banana peel extract mediated novel route for the synthesis of silver nanoparticles,” *Colloids Surfaces A Physicochem. Eng. Asp.*, vol. 368, no. 1–3, pp. 58–63, 2010.

[13] R. Ashraf, S. Riaz, S. S. Hussain, and S. Naseem, *Effect of pH on properties of ZnO nanoparticles*, vol. 2, no. 10. Elsevier Ltd., 2015.

[14] S. S. Alias, A. B. Ismail, and A. A. Mohamad, “Effect of pH on ZnO nanoparticle properties synthesized by sol-gel centrifugation Author ’ s personal copy,” no. December 2018, pp. 1–8, 2010.

[15] C. Pholnak *et al.*, “Effects of Precursor Concentration and Reaction Time on Sonochemically Synthesized ZnO Nanoparticles 2 . Experimental Procedure 3 . Results and Discussion,” vol. 17, no. 2, pp. 405–411, 2014.

[16] A. Aziz and A. Karim, “The Effect of pH on Zinc Oxide Nanoparticles Characteristics Synthesized from Banana Peel Extract,” vol. 797, pp. 271–279, 2019.

[17] U. Airlangga, “THE INFLUENCE OF PH VALUES ON THE CRYSTALLITE SIZE OF ZnO,” vol. 2, no. 2, pp. 118–122, 2017.

[18] M. J. Chithra and M. S. K. Pushpanathan, “Effect of pH on Crystal Size and Photoluminescence Property of ZnO Nanoparticles Prepared by Chemical Precipitation Method,” 2015.

[19] R. Marsalek, “Particle size and Zeta Potential of ZnO,” *Procedia - Soc. Behav. Sci.*, vol. 9, pp. 13–17, 2014.

[20] M. Chabni, H. Bougherra, H. Lounici, T. Ahmed-zaı, and J. Canselier, “Open Archive Toulouse Archive Ouverte (OATAO) Evaluation of the Physical Stability of Zinc Oxide Suspensions Containing Sodium Poly- (acrylate) and Sodium Dodecylsulfate,” vol. 32, pp. 1786–1798, 2011.

[21] A. S. Dukhin, P. J. Goetz, X. Fang, and P. Somasundaran, “Journal of Colloid and Interface Science Monitoring nanoparticles in the presence of larger particles in liquids using acoustics and electron microscopy,” *J. Colloid Interface Sci.*, vol. 342, no. 1, pp. 18–25, 2010.