

CHARACTERIZATION OF ALOE VERA PLANT AND SYNTHESIS OF ZINC OXIDE FROM ALOE VERA LEAF USING GREEN SOL-GEL METHOD

Nur Liyana Binti Johari, Rabiatal Adawiyah Abdol Aziz

Faculty of Chemical Engineering, Universiti Teknologi Mara

Abstract— Zinc oxide is a white powder with a chemical formula of Zn. Zinc oxide have a wide uses such as being used as colorant, skin protectant and also as bulking agent. The main objective of this study is to identify the functional group in the Aloe Vera plant and to synthesis zinc oxide from the Aloe Vera extract by using sol-gel method. Other than that, to study the effect of different concentration of precursor (zinc acetate dehydrate) on the size of nanoparticle produce. In order to identify the functional group in the aloe vera leaf, the Fourier Transform Infrared Spectroscopy (FTIR) analysis is being done. The zinc oxide is synthesis by using sol-gel method. The zinc oxide is synthesize with varies precursor concentration (zinc acetate dehydrate) concentration of 1.0, 0.8, 0.6, 0.5, 0.4, 0.3 and 0.2 M. The synthesize nanoparticle are then characterize by using X-ray powder diffraction (XRD) FTIR analysis. The result shows that all of the powder produce is prove to be zinc oxide particle. While the size of the nanoparticle increased as the concentration of the precursor increase.

I. INTRODUCTION

Zinc oxide commonly exist physically as a white powder with a chemical formula of ZnO. It is classed as an inorganic oxide. Zinc oxide have a wide uses such as being used as colorant, skin protectant and also as bulking agent. Zinc oxide also one of the most used nanoparticle in the world. Zinc oxide nanoparticle have undergoes various researches.

The synthesis of zinc oxide can be done by using various method such as sol-gel method, green sol-gel method, green synthesis, gas condensation, pyrolysis, chemical precipitation attrition and hydrothermal synthesis. From all the available method, green sol-gel is well known as it is the simplest and easier way to prepare nanoparticle. The different between a sol-gel and green sol-gel method is the used of the solvent. In sol-gel method, the solvent used is alcohol, while in green sol-gel, the solvent used is from plant extract.

Zinc oxide is known worldwide as it used in medical and have a huge amount of import and export value at 1.27 billion US dollar. (OEC, 2014) Thus this prove that the production of zinc oxide are huge industries and used a lot of solvent in order to produce it. The problem industries faces is the solvent that they have being using is toxic chemical which is not environmentally friendly. Other than that, the cost of alcohol is also relatively high.

extract as the replacement of alcohol. But the used of aloe vera extract with precursor of different concentration is not yet being investigated.

Thus in this study, its aims to give an alternative solvent that can replace the toxic chemical used in producing ZnO. This study will specifically producing the ZnO by using the aloe vera extract as solvent. The leaf are choose due to the fact that the skin of the aloe vera are most likely to be dumped after the food or cosmetic manufacturer have extract the fillet that consist of the gel. Thus the leaf of the aloe vera that are dumped by the industries, can be collected and used to produce the ZnO.

The main objective of this study is to identify the functional group and chemical content in the Aloe Vera plant and to synthesis zinc oxide from the Aloe Vera extract by using green sol-gel method. In order to identify the mineral element in the aloe vera leaf, Fourier Transform Infrared Spectroscopy (FTIR) is being used. The zinc oxide is synthesize with varies precursor concentration which is the zinc acetate dehydrate concentration of 1.0, 0.8, 0.6, 0.5, 0.4, 0.3 and 0.2 M. The synthesized nanoparticle are then characterize by using (XRD) FTIR analysis. The different in concentration is being studied to observe the effect of precursor concentration on the size of nanoparticle.

II. METHODOLOGY

A. Aloe Vera Plant Standard Preparation

The aloe vera plant is not be removed from the pot until it is required to be used in the experiment. The aloe vera leaves length is in between 30 to 50 cm, were remove from the entire plant by cutting it closer to the roots. After the leaves were remove from the plant, the spike along the leaf is discard. The aloe vera were then separated into two parts which is the skin and the fillet. Both of the parts were washed by using distilled water to remove any dirt. In order to keep the sample fresh, the sample taken from the pot will not be stored more than 24 hours and stored in the refrigerator in the lab. The leave is then dried in the oven for 6 hours at 100°C. After the leave is completely dried, the leaves is then grinded until it is in forms of fine powder. The aloe vera leave powder were then being used as raw standard throughout the experiment. While the gel is centrifuge in the centrifuger in order to obtain its cell wall (solid content).

There are several studies done that suggest the used of plant

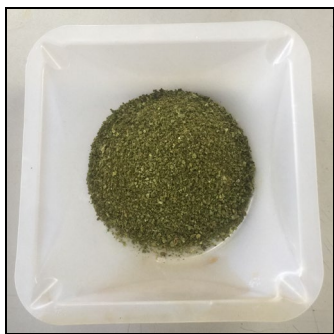


Figure 1: Grinded Aloe Vera Leaf

B. Extract Preparation

Extract is prepared from the dried skin of the aloe vera. The aloe vera leaf is dried and grind. 20 gram of the grinded aloe vera skin is added to the 500ml beaker. 400ml of distilled water is added to the beaker. The beaker is then place over a hot plate. The hot plate is set at 120°C and at 160rpm. The beaker is left for 1 hours. After 1 hours, the extract is filtered by using vacuum filter in order to discard the solid substance from the extract. The extract is then kept in the beaker as standard throughout the experiment. The extract is being used within 48 hours.

C. Synthesis of Zinc Oxide by using Sol-gel Method

In a beaker, 50 ml of the extract is added to the beaker with 50 ml of zinc acetate dehydrate concentration of 0.2M. These step is then repeat for zinc acetate dehydrate concentration of 0.3M, 0.4M, 0.5M, 0.6M, 0.8M and 1.0M in different beaker. Initial pH of each beaker is then recorded. The mixture is place over a hot plate where the temperature is setup at 120°C while continuously stirred at 360 rpm. The mixture is let to stir for 5 minutes before NaOH is added to the mixture until the final pH of the mixture reach 12. The pH is set to be at 12 as the particle will be in its best morphology state. White precipitate will formed slowly. The stirring is continued for 1 hours. After 1 hours, the stirring is stop and the beaker is removed from the hot plate. The white precipitate is then recovered by using vacuum filter. The recovered precipitate is then weight. The precipitate is dry at 100°C for 6 hours and the dried precipitate is then mashed in mortar and pestle before it being weight again.

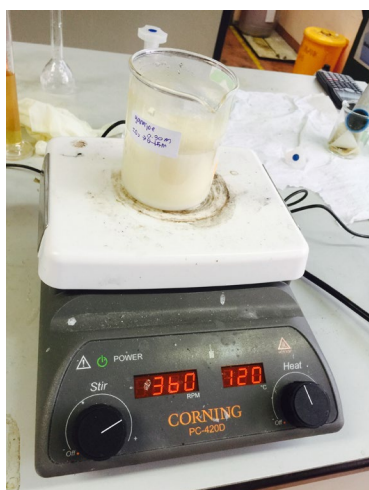


Figure 2: Zinc oxide synthesise on the hot plate

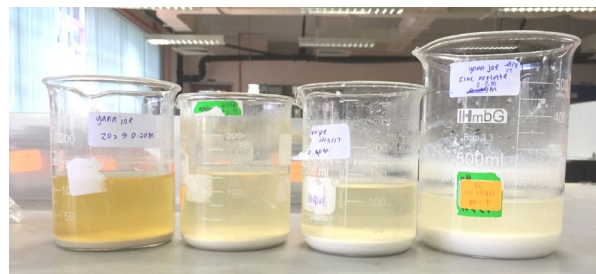


Figure 3: Zinc oxide synthesise settling at the bottom of beaker

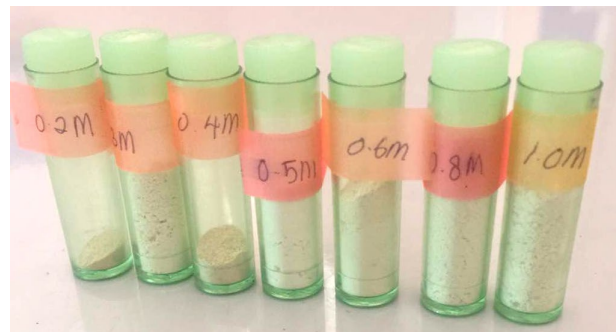


Figure 4: Zinc oxide synthesized

D. Characterization of Aloe Vera Leaf and Gel

FTIR is used for characterization in order to determine the functional group.² (Reusch, 2013) FTIR analysis used is the FTIR Perkin Elmer spectrum one. Sample used in the FTIR is aloe vera leaf and gel powder. The sample hole of the FTIR were clean by using acetone. Suitable amount of the aloe vera leaf powder prepared earlier in the standard preparation were place in sample hole. The handle is placed on top of the filled hole. The handle is then level to press toward the sample in the hole. The pressure are then adjusted to be between level 6 to 7. The spectrum were then run. The spectrum of the sample were then obtained from the system. These steps is repeated for aloe vera gel powder.

E. Characterization of Zinc Oxide Nanoparticle

XRD and FTIR is used in characterization of zinc oxide nanoparticle. XRD is used to identify the size of the nanoparticle while FTIR is used to identify the functional group in the zinc oxide.

For XRD analysis the zinc oxide powder with concentration of zinc acetate dehydrate of 1.0M is fill in the sample cup and packed tightly so that the powder is stay in place. The voltage is setup to be 30 kV, ampere 15mA, start angle at 10°, the end angle at 80° and the speed angle is at 3° per min. The sample is obtained and place into the sample holder. Sample is place in the holder in the sample positioner. The doors is closed slowly. The green ready light should be the only LED displayed. The commander program is run. Title of the sample is set based on the concentration of the sample. The scan is start. The scan is done within 30 minutes. The data is then recorded. These steps is repeated for other sample concentration.

Sample used in the FTIR is zinc oxide powder with concentration of zinc acetate dehydrate of 1.0M. The sample hole of the FTIR were clean by using acetone. Suitable amount of the

zinc oxide powder prepared earlier in the standard preparation were place in sample hole. The handle is placed on top of the filled hole. The handle is then level to press toward the sample in the hole. The pressure are then adjusted to be between level 6 to 7. The spectrum were then run. The spectrum of the sample were then obtained from the system. These steps is repeated for other concentration.

III. RESULTS AND DISCUSSION

A. The Functional Group in Aloe Vera Plant

After the aloe vera leaf and the gel is characterize by using FTIR analysis, the FTIR spectrum is obtained.

Figure 5 shows the FTIR spectra of aloe vera leaf. For peaks observed at 3299.41 cm^{-1} , the functional group is correspond to water OH stretch. As for peak 2916.96 cm^{-1} is due to C-H stretch. While 1737.63 cm^{-1} peaks is correspond to C=O aldehyde. The 1598.35 cm^{-1} peaks is correspond to the C=C. The 1228.52 cm^{-1} is due to C-O-C stretch. Last but not least, peak 1014.74 cm^{-1} is correspond to the C-OH stretch.

Figure 6 shows the FTIR spectra of aloe vera gel. For peaks observed at 3313.13 cm^{-1} , the functional group is correspond to water OH stretch. As for peak 2170.82 cm^{-1} is due to C=C stretch. While 1637.82 cm^{-1} peaks is correspond to C=C alkene.

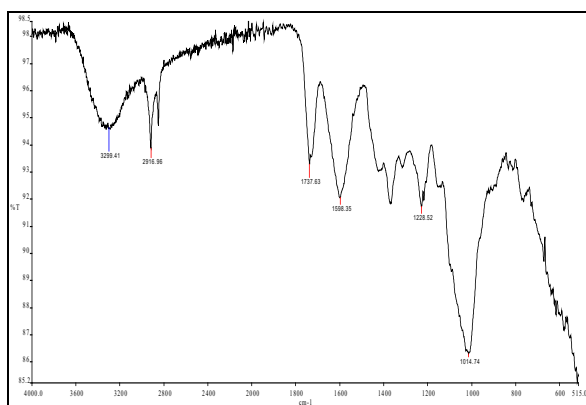


Figure 5: FTIR Result for Aloe Vera Leaf

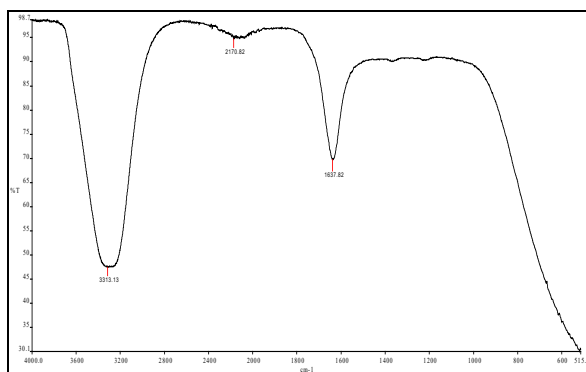


Figure 6: FTIR Result for Aloe Vera Gel

Table 1: Aloe Vera functional group and its FTIR peaks.

Functional group	Aloe vera leaf Peaks (cm^{-1})	Aloe vera gel Peaks (cm^{-1})
OH stretch	3299.41	3313.13

C=C stretch	-	2170.82
C=C alkene	-	1637.82
C-H stretch	2916.96	-
C=O aldehyde	1737.63	-
C=C aromatic	1598.35	-
C-O-C stretch	1228.52	-
C-OH stretch	1014.74	-

B. The Weight of Zinc Oxide synthesize

The synthesized zinc oxide is filtered by using vacuum filter and the weight of the zinc oxide is record before the zinc oxide is being dried in the oven. The weight of each zinc oxide is shown as in table 2.

Where;

Zinc oxide weight = final weight – apparatus weight

Final weight = dried zinc oxide + foil + petri dish + filter paper

Apparatus weight= foil + petri dish + filter paper

Table 2: Weight of Zinc Oxide Synthesize

Zinc acetate dehydrate concentration	Zinc oxide weight (g)
1.0 M	3.93
0.8M	3.43
0.6M	3.09
0.5M	2.84
0.4M	1.84
0.3M	1.79
0.2M	1.46

From table 2, the weight of the zinc oxide increase as the concentration of the zinc acetate dehydrate used in the sol gel method increases. The weight of zinc oxide is lowest (1.46 g) as the concentration of the zinc acetate dehydrate used is also lowest which is 0.2M. While zinc oxide weight with zinc acetate dehydrate concentration of 1.0 M is the highest which is 3.93 g.

Thus the higher the concentration of zinc acetate dehydrate use, the higher the amount of zinc oxide produces. This is due to more zinc content in the precursor to be bond with oxygen ion in order to forms zinc oxide molecule.

C. The Functional Group in Zinc Oxide synthesize

Each of the FTIR spectrum of each sample is analyzed. The summary of all spectrum and functional group found in the zinc oxide powder synthesize is tabulated in table 3.

Table 3: Summary of Functional Group in the Zinc Oxide Nanoparticle Powder.

concentra tion (M)	OH (cm^{-1})	C-H bend (cm^{-1})	Alkoxy C-O (cm^{-1})	C=C (cm^{-1})	Zn-O (cm^{-1})
1.0	3272.28	1407.73	1019.19	1637.45 1550.22	647.81
0.8	3220.41	1406.45	1085.79 1032.09	1542.11	841.66 712.61 646.10
0.6	3271.17	1407.62	1019.34	1638.29 1550.10	643.23
0.5	3371.61	1412.02	-	1555.06	651.45
0.4	3387.11	1415.58	-	1561.77	891.23

0.3	3387.01	1411.58	-	1555.03	833.75
0.2	3379.80	1410.10	1024.49	-	871.12

As shown in table 3, all of the zinc oxide have functional group of OH stretch and C-H bend. While zinc oxide with concentration of precursor of 1.0, 0.8, 0.6, and 0.2 M c alkoxy C-O stretch. Other than that, the C=C alkene functional group is observed in all sample except sample 0.2 M. Last but not least, all sample consist of metal oxide (zinc oxide) stretch. Thus all of the sample is prove to be zinc oxide powder synthesized. The compilation of all FTIR result of the zinc oxide is as in figure 7.

D. X-ray Diffraction Analysis on Zinc Oxide

X-ray Diffraction (XRD) instrument were used in order to identify the size of the zinc oxide nanoparticle size. The size of the nanoparticle is obtain by using the Scherrer's formula. The formula required the value of λ which is the wavelength of the X rays used, β which is the width of half maximum (FWHM) and the θ , which is the angle of diffraction. All of the value can be obtained from the XRD result. While the k value, which the constant is obtained to be 0.9 and the λ is known which is 1.5406 Å. The summary of the size is tabulated as in table 4. The Scherrer's formula is shows as follow:

$$D = \frac{k\lambda}{\beta \cos \theta}$$

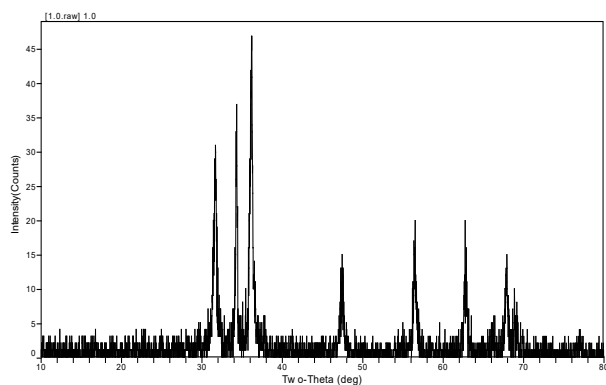


Figure 8: XRD Result for concentration of 1.0 M

Above figure 8 shows the XRD result for Zinc Oxide (Concentration of Zinc Acetate Dehydrate of 1.0M). The average size of the particle is calculated. The average size is calculated to be 456.24 angstrom which is equivalent to 4.5624 x 10¹³m as in table 4.

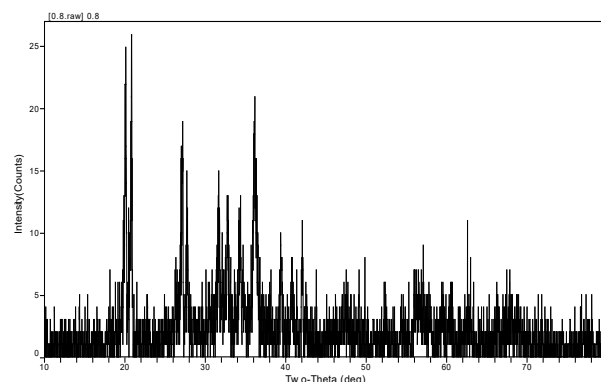


Figure 9: XRD Result for concentration of 0.8 M

Above figure 9 shows the XRD result for Zinc Oxide (Concentration of Zinc Acetate Dehydrate of 0.8M). The average size of the particle is calculated. The average size is calculated to be 446.34 angstrom which is equivalent to 4.4634 x 10¹³m.

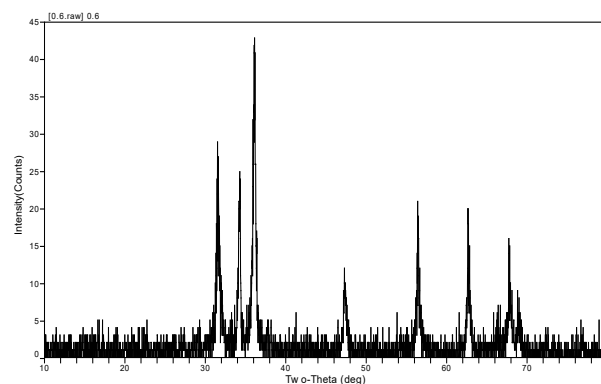


Figure 10: XRD Result for concentration of 0.6 M

Above figure 10 shows the XRD result for Zinc Oxide (Concentration of Zinc Acetate Dehydrate of 0.6M). The average size of the particle is calculated. The average size is calculated to be 325.375 angstrom which is equivalent to 3.25375x10¹³m.

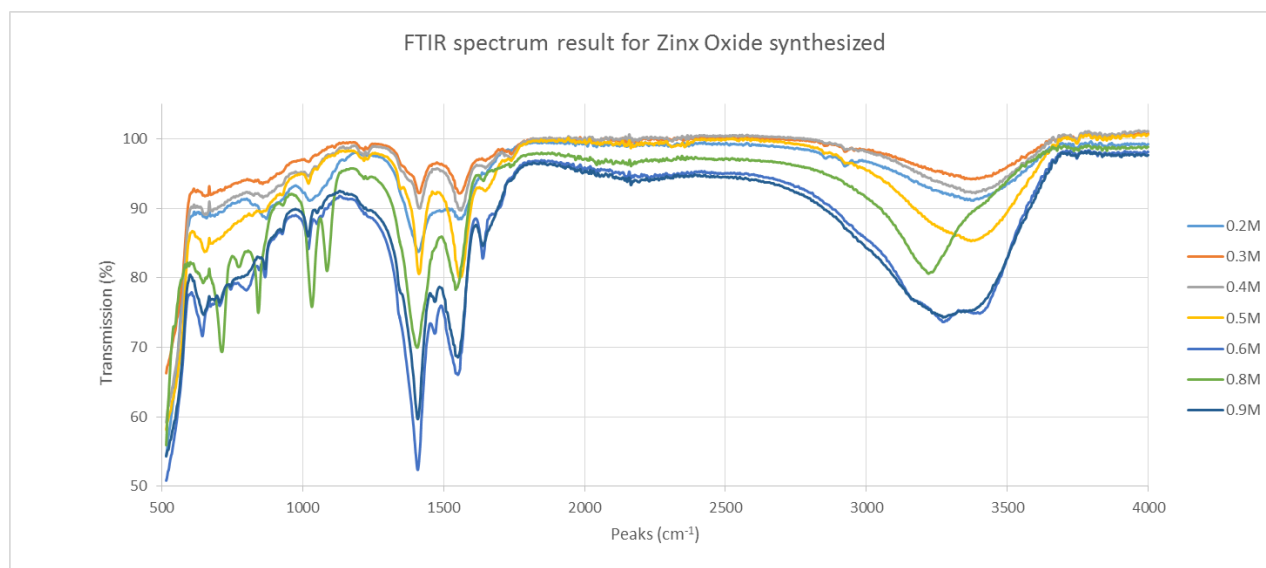


Figure 7: Compilation of FTIR Graph

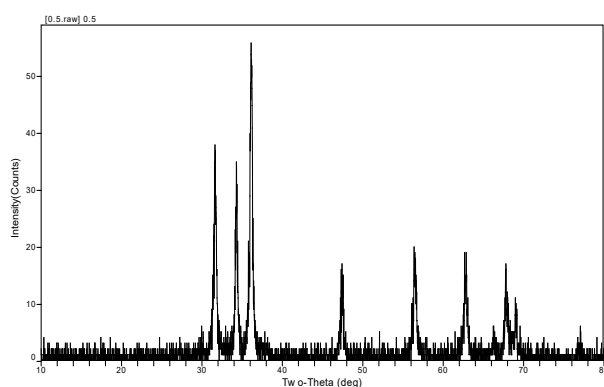


Figure 11: XRD Result for concentration of 0.5 M

Above figure 11 shows the XRD result for Zinc Oxide (Concentration of Zinc Acetate Dehydrate of 0.5M). The average size of the particle is calculated. The average size is calculated to be 316.80 angstrom which is equivalent to $3.1680 \times 10^{-13} \text{m}$.

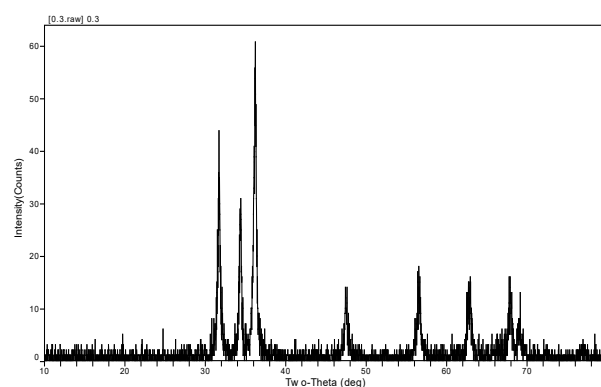


Figure 13: XRD Result for concentration of 0.3 M

Above figure 13 shows the XRD result for Zinc Oxide (Concentration of Zinc Acetate Dehydrate of 0.3M). The average size of the particle is calculated. The average size is calculated to be 251.485 angstrom which is equivalent to $2.51485 \times 10^{-13} \text{m}$.

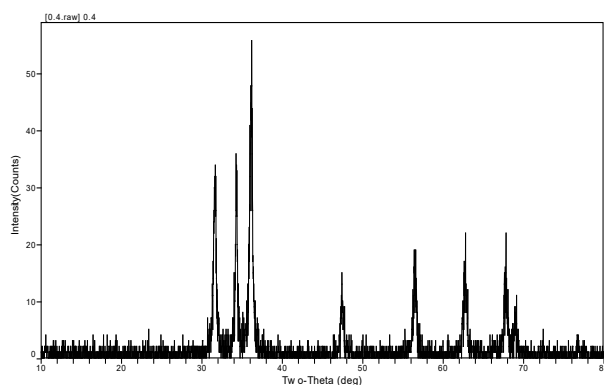


Figure 12: XRD Result for concentration of 0.4 M

Above figure 12 shows the XRD result for Zinc Oxide (Concentration of Zinc Acetate Dehydrate of 0.4M). The average size of the particle is calculated. The average size is calculated to be 304.65 angstrom which is equivalent to $3.0465 \times 10^{-13} \text{m}$.

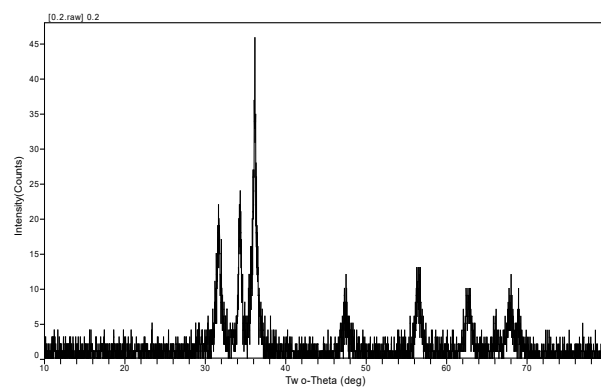


Figure 14: XRD Result for concentration of 0.2 M

Above figure 14 shows the XRD result for Zinc Oxide (Concentration of Zinc Acetate Dehydrate of 0.2M). The average size of the particle is calculated. The average size is calculated to be 172.61 angstrom which is equivalent to $1.7261 \times 10^{-13} \text{m}$.

Table 4: Summary of XRD Analysis of Zinc Oxide Nanoparticle Size

Zinc oxide with zinc acetate dehydrate concentration (M)	2θ (degree)	FWHM (degree)	Size (ang)	Size (nm)
1.0	36.16	0.3451	456.24	45.624
0.8	36.16	0.5002	446.34	44.634
0.6	36.10	0.3583	325.38	32.538
0.5	36.12	0.2801	316.80	31.680
0.4	36.12	0.3967	304.65	30.465
0.3	36.24	0.3674	251.49	25.149
0.2	36.16	0.4537	172.30	17.230

As observed in the table 4, the size of the nanoparticle is less than 100nm. The size of the zinc oxide particle is increasing as the concentration of the precursor increasing. The size of the nanoparticle is increasing due to the increasing metal ions concentration. The higher amount of metal ion allows the nucleation and the aggregation processes to occur more to forms the nanoparticle. Thus increasing the size of the nanoparticle synthesized.

For any future research being done based on this journal, some recommendation should be considered. First, the plant should be from one pot or one plant only. The characterization can be more accurate for the specific plant. It is also recommended that the plant extract is filtered from one standard aloe vera powder. The extraction process should be done within 24 hours after the aloe vera powder is obtained to ensure the freshness of the extract. The extracted liquid should be kept only for 48 hours and not more. The zinc oxide that have been synthesize should be remove from the filter paper before its being dried in the oven to prevent the paper to contaminate the sample.

IV. CONCLUSION

Functional group that observed in the aloe vera leaf is Water OH stretch, C-H stretch, C=O aldehyde, C=C aromatic, C-O-C stretch, and C-OH stretch functional group. While aloe vera gel consist of Water OH stretch, C=C stretch, and C=C alkene. Based on summary of weight of zinc oxide synthesize table, the higher the concentration of precursor used, the higher the amount of zinc oxide synthesize. This is due to more zinc oxide ions can be obtain from the higher amount of zinc acetate dihydrate concentration. All of the powder synthesize is zinc oxide particle as all of the powder consist of functional group for metal oxide which is at range of 1000 cm⁻¹ and below. Based on the XRD analysis of zinc oxide, the size of zinc oxide particle is decreasing as the concentration of zinc acetate used as precursor is decreasing.

ACKNOWLEDGMENT

First and foremost, I would like to thank to God for all that he have given to me which is the opportunities, the strength and the will to complete my report. Secondly, I would like to thank my supervisor Madam Rabiatal Adawiyah Abdol Aziz for all of her effort and time spend for guiding me to complete my report. My gratitude also goes to lab assistant that have been helping me locating the instrument to be used and give me technical support regarding the instrument. I would also thank to my fellow friends that have giving me guide and support to complete my report especially Umi Atikha, Tunku Farah and Muhammad Nazri. Last but not least, thanks to my parents that have been giving me a nonstop mental support and financially support.

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