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# SYNTHESIS OF ZINC OXIDE NANOPARTICLES USING *Aloe vera* LEAF AQUEOUS EXTRACTS AND ITS CHARACTERIZATION

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## Abstract

The aim of this research was to synthesize zinc oxide nanoparticles via green routes using *Aloe vera* leaf aqueous extracts as well as via chemical method and to study its characteristics. In biosynthesis of zinc oxide nanoparticles, zinc acetate was used as a precursor and *A. vera* leaf hot and cold aqueous extracts used as reducing and stabilizing agents. On the other hand, zinc oxide nanoparticles were synthesized chemically from zinc acetate in the presence of sodium hydroxide. The synthesized zinc oxide nanoparticles were characterized by X-ray diffraction (XRD), FT IR, scanning electron microscopy (SEM) and thermogravimetric /differential thermal analysis (TG-DTA) techniques. The amount of yield percents of the synthesized ZnO NPs obtained by biosynthesis method were higher than those of the chemical method at the same temperature. The zinc oxide nanoparticles were observed as particles agglomeration studied by the morphology using SEM images and had hexagonal wurtzite structure with the lattice constants of a = b = 3.2511 Å and c = 5.2076 Å in average crystallite size about 18 ~ 19 nm, according to XRD analysis. FT IR spectrum showed the Zn-O absorption bands in range between 600 ~ 400 $cm^{-1}$. TG-DTA analysis resulted the weight loss about 4 ~5 % of the synthesized zinc oxide nanoparticles at nearly 250 °C. Small weight losses of zinc oxide samples indicated the thermal stability of the synthesized zinc oxide nanoparticles.

**Keywords***:* Biosynthesis, ZnO nanoparticles, *Aloe vera* leaf aqueous extracts, wurtzite structure, SEM, XRD, FT IR, TG-DTA

## Introduction

The term “nano” is derived from the Greek word “nanos” which means small and it is used as the prefix for one billionth part ($10^{-9}$). "Nano" is now a popular label for much of modern science, and many “nano” words have recently appeared in dictionaries, including: nanometer, nanoscale, nanoscience, nanotechnology, nanotube, nanowire, and nanorobot (Sovan,

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2011). Nanochemistry is a branch of nanoscience, deals with the chemical applications of nanomaterials in nanotechnology. Nanochemistry involves the study of the synthesis and characterization of materials of nanoscale size. Nanotechnology is emerging as a rapidly growing field with its application in science and technology for the purpose of manufacturing new materials at the nano scale level (Rana *et al.,* 2010).

Metal oxides play a very important role in many areas of chemistry, physics, and materials science. The increases surface area and smaller size of these particles make them an ideal antibacterial agent. The zinc oxide nanoparticles are of significant interest as they provide many practical applications in worldwide (Mayekar *et al.*, 2013).

Particles are further classified according to diameter. Coarse particles cover a range between 2,500 and 10,000 nanometers. Fine particles are sized between 100 and 2,500 nanometers. Ultrafine particles, or nanoparticles are between 1 and 100 nanometers in size.

## Synthesis Approaches and Techniques

To prepare these nanomaterials, novel synthesis procedures have been developed that can be described as physical and chemical methods. In general, there are two approaches to the synthesis of nanomaterials and the fabrication of nanostructures: top-down and bottom-up. Top-down approach refers to slicing or successive cutting of a bulk material to get nanosized particles. Bottom-up approach refers to the build-up of a material from the bottom. Both approaches play very important roles in nanotechnology (Zheng**,** 2009).

Currently, a large number of physical, chemical and biological methods are available to synthesize different types of nanoparticles. The chemical synthesis involves toxic solvents, high pressure, energy and high temperature conversion and microbe involved synthesis is not feasible industrially due to its lab maintenance (Ayodeji and Olabisi, 2013). Organic chemical solvents are toxic and require extreme conditions during nanoparticle synthesis (Parthasarathy *et al.,* 2016). The development of safe eco-friendly methods for biosynthesis production is now of more interest due to simplicity of the procedures and versatility (Vishwakarma, 2013). Plant extracts function as stabilizing, capping or hydrolytic agents. These plant extracts also allow a controlled synthesis (Mohammed Osman and Mustafa, 2015).

## Physical and Chemical Properties of Zinc Oxide

Zinc oxide is an inorganic compound with the formula ZnO. It usually appears as a white powder known as zinc white. It is usually crystallizes in three different forms: hexagonal wurtzite (Figure 1), cubic zinc blende and cubic rocksalt (rarely). Among them, the hexagonal wurtzite is most stable at ambient conditions. In zinc oxide, zinc and oxide centers are tetrahedral, except rocksalt (Behera, 2013).

In nature, zinc oxide is usually orange or red in colour due to manganese impurity and other elements. Crystalline zinc oxide is thermochromic, which changes from white to yellow colour when heated and reverting to white colour on cooling. This change in colour is caused by a very small loss of oxygen at high temperatures. Zinc oxide is amphoteric oxide. It is nearly insoluble in water and alcohol, but it is soluble in (degraded by) most acids, that is it reacts with both acids and alkalis (Behera, 2013).

**Type of Compound - Inorganic Compound**

| | |
|---|---|
| Molecular Formula | - ZnO |
| Molar Mass | - 81.408 g/mol |
| Appearance | - White Solid |
| Odour | - Odourless |
| Relative Density | - 5.607 |
| Melting Point | - 1975 °C |
| Refractive Index | - 2.004 |

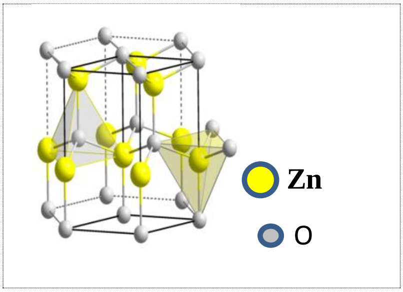


**Figure 1:** Hexagonal wurtzite structure model of ZnO

## Advantages of Nanoparticles Synthesis

ZnO nanoparticles can be prepared on a large scale at low cost by simple solution methods, such as alkali precipitation, thermal decomposition, hydrothermal synthesis, sol-gel methods, spray pyrolysis and other routes.

Among all these different methods, the precipitation is one of the most important methods to prepare nanopowder, due to its excellent advantages such as low cost, low temperature, non-toxic operation and environmental friendliness. It is suitable for industrial, technical and medical applications due to its diverse properties which have been found to strongly depend on their morphology. So, it is necessary to confirm by techniques employ for nanoparticles characterization.

So, the biosynthesis method is the best option for the synthesis of nanoparticles by using *A. vera* leaf aqueous extracts. In this present work, *A. vera* leaf extracts were used as reducing agent for synthesis of zinc oxide nanoparticles and the characteristics of the prepared zinc oxide nanoparticles were studied by XRD, SEM, FT IR and TG-DTA analyses.

## Materials and Methods

The experiments were carried out at Department of Chemistry, University of Yangon. All of the chemicals used in the present work were analytical reagent grade. Fresh *Aloe vera* leaves were purchased from the local market in Hledan, Kamayut Township, Yangon. The samples were identified by authorized botanist of Department of Botany, University of Yangon.

### Preparation of *Aloe vera* Leaf Aqueous (Hot and Cold) Extracts

The fresh *A. vera* leaf (100 g) were thoroughly washed with distilled water, dried and then boiled in 150 mL of deionized water (solution changed from watery to light green) for half an hour. The resulting solution was cooled and filtered to get the *A. vera* aqueous hot extracts (AHE).

On the other method, *A. vera* leaf (100 g) were thoroughly washed with distilled water, dried and then percolated in 150 mL of deionized water for 24 h. The resulting solution was filtered to get the *A. vera* aqueous cold extracts (ACE) (Parthasarathy *et al.,* 2016).

### Preliminary Phytochemicals Screening of *A. vera* Leaf Aqueous Extracts

Preliminary phytochemical tests were performed to know the different types of compounds present in *A. vera* leaf aqueous extracts. Phytochemicals screening for alkaloids, $\alpha$-amino acid, carbohydrates, coumarins, cyanogenic glycosides, flavonoids, glycosides, phenolic compound, quinones, saponins, steroids, tannins and terpenoids were carried out by the reported methods.

**Biosynthesis and Chemical Synthesis of ZnO Nanoparticles**

In biosynthesis method, 2.00 g, 2 %, w/v of zinc acetate was dissolved in 100 mL of distilled water. Each of (10 mL) *A. vera* leaf aqueous extracts in the ratio of 1:10, v/v was added drop-wise and the resulting mixtures were stirred for 10 min by using a magnetic stirrer at temperature 120 °C. In order to adjust the pH-12 of the solution with sodium hydroxide (2 M) was added drop-wise while stirring. A white crystalline precipitate of zinc hydroxide was obtained, which was washed repeatedly with distilled water and ethanol, filtered and then dried on an oven at 120 °C for 3 h to obtain the zinc oxide nanoparticles (Elizabeth and Mary, 2015).

In chemical method, 2.00 g, 2 %, w/v of zinc acetate was also dissolved in 100 mL of distilled water. 2 M Sodium hydroxide in the ratio of 1:10, v/v was added drop-wise into the 2 % zinc acetate solution with stirring for 10 min by using a magnetic stirrer at temperature 120 °C. A white crystalline precipitate of zinc hydroxide was obtained, which was washed repeatedly with distilled water and ethanol, filtered and then dried on an oven at 120 °C for 3 h to obtain the ZnO nanoparticles (Mayekar *et al.*, 2013).

**Characterization of the Prepared Zinc Oxide Nanoparticles**

**XRD analysis**

Zinc oxide nanoparticles were examined by X-ray diffractometer (Rigaku D/max, Japan) for $2\theta$ values ranging from 10° to 70° using (Cu / K-$\alpha$) radiation at $\lambda$ = 1.5406 Å. The average crystallites size (D) of the synthesized zinc oxide nanoparticles were calculated by using the well-known Scherrer formula.

**SEM analysis**

The surface morphological features of synthesized zinc oxide nanoparticles were studied by scanning electron microscope (EVO - 18, Brand

ZEISS, Germany). SEM is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons.

**FT IR analysis**

The prepared zinc oxide nanoparticles were characterized by fourier transform infrared spectroscopy. FT IR infrared radiation refer to the part of electromagnetic spectrum between the visible and microwave regions. The FT IR spectrum recorded in the range 4000–400 $cm^{-1}$ by using FT IR spectrometer (8400-SHIMADZU, Japan) at universities research centre (URC).

**TG-DTA analysis**

TG-DTA measures both heat flow and weight changes in a material as a function of temperature or time in a controlled atmosphere (Coats, 1963). The stability of the synthesized zinc oxide nanoparticles was studied with the TG-DTA thermogram measured by using Perkin-Elmer thermogravimetric-differential analyzer.

## Results and Discussion

### Phytochemicals Present in *Aloe vera* Leaf Aqueous Extracts

Preliminary phytochemical screening revealed that alkaloids, α-amino acids, coumarins, flavonoids, glycosides, tannins and terpenoids were found to be present in *A. vera* leaf aqueous extracts. All of these secondary metabolite phytoconstituents could support to reduce zinc acetate to zinc oxide nanoparticles.

### Biosynthesis and Chemical Synthesis of Zinc Oxide Nanoparticles

In the biosynthesis, zinc oxide nanoparticles were prepared from zinc acetate in the presence of *A. vera* leaf aqueous hot extracts (AHE) and in *A. vera* leaf aqueous cold extracts (ACE) at 120 °C. The pH of the reaction mixture was controlled at pH-12 by using 2 M sodium hydroxide solution. The ZnO nanoparticles were respectively obtained as a light brown crystalline powder (ZnO-AHE) and as a pale yellow crystalline powder (ZnO-ACE) in 41.17 % and 41.01 % of yields by using *A. vera* leaf aqueous hot extracts and *A. vera* leaf aqueous cold extracts as reducing agent (Figures 2 (a) and 2 (b)).

Zinc oxide nanoparticles were also chemically synthesized from zinc acetate by reducing agent with 2 M soidum hydroxide solution (1:10, v/v) at 120 °C (Jeeva Lakshmi, 2012). The zinc oxide nanoparticles (ZnO-CM) were obtained as white crystalline powder in 38.21 % of yield (Figure 2 (c)).

The yield percents of zinc oxide nanoparticles synthesized by green route and chemical synthesis are listed in Table 1. The yield percents obtained in biosynthesis were slightly higher than that obtained in chemical synthesis (38.21 %). The yield percents of zinc oxide by using *A. vera* leaf aqueous hot extracts (41.17 %) was found to be similar that obtained by using *A. vera* leaf aqueous cold extracts (41.01 %).


**Figure 2:** Photographs of the synthesized zinc oxide powders by using (a) *A. vera* aqueous hot extracts, ZnO-AHE (b) *A. vera* aqueous cold extracts, ZnO-ACE and (c) chemical method, ZnO-CM

**Table 1: Yield Percents of the Synthesized Zinc Oxide Nanoparticles**

| No. | Methods used | ZnO nanoparticles | Weight of ZnO nanoparticles (g) | Yield of ZnO nanoparticles (%) |
|---|---|---|---|---|
| 1. | Biosynthesis | ZnO-AHE | 0.82 | 41.17 |
| 2. | Biosynthesis | ZnO-ACE | 0.83 | 41.01 |
| 3. | Chemical synthesis | ZnO-CM | 0.76 | 38.21 |

Zinc acetate = 2.0 g
ZnO-AHE = ZnO nanoparticles prepared by using *A. vera* leaf aqueous hot extracts
ZnO-ACE = ZnO nanoparticles prepared by using *A. vera* leaf aqueous cold extracts
ZnO-CM = ZnO nanoparticles prepared by using chemical method

## Characteristics of Zinc Oxide Nanoparticles

### XRD analysis

The x-ray diffraction patterns of ZnO nanoparticles prepared by biosynthesis and chemical synthesis are shown in Figures 3, 4 and 5 for the results of ZnO-AHE, ZnO-ACE and ZnO-CM, respectively. The purity and crystallite size were characterized by studying the x-ray diffraction patterns. It is evident from the diffractograms that, the 7 peaks observed in all of the diffractograms are similar and their peak positions coincide with that of reference values. The 7 peaks noticed are in accordance with ZnO phase. The x-ray diffractograms of ZnO samples obtained at 120 $^{o}$C showed well-defined peaks with Miller indices (100), (002), (101), (102), (110), (103) and (112) corresponding to Bragg angles, 31.57$^{o}$, 34.22$^{o}$, 36.10$^{o}$, 47.30$^{o}$, 56.39$^{o}$, 62.70$^{o}$ and 67.89$^{o}$ in AHE, 31.66$^{o}$, 34.30$^{o}$, 36.18$^{o}$, 47.46$^{o}$, 56.47$^{o}$, 62.76$^{o}$ and 67.84$^{o}$ in ACE and 31.80$^{o}$, 34.40$^{o}$, 36.26$^{o}$, 47.54$^{o}$, 56.67$^{o}$, 62.89$^{o}$ and 67.96$^{o}$ in CM, respectively. All diffraction peaks can be readily indexed to hexagonal wurtzite zinc oxide structure with the average crystallite size about 18 ~ 19 nm. X-ray diffraction patterns indicated that the obtained ultrafine nanoparticles are in good crystallinity which exhibited in the range of nano scale (< 100 nm).

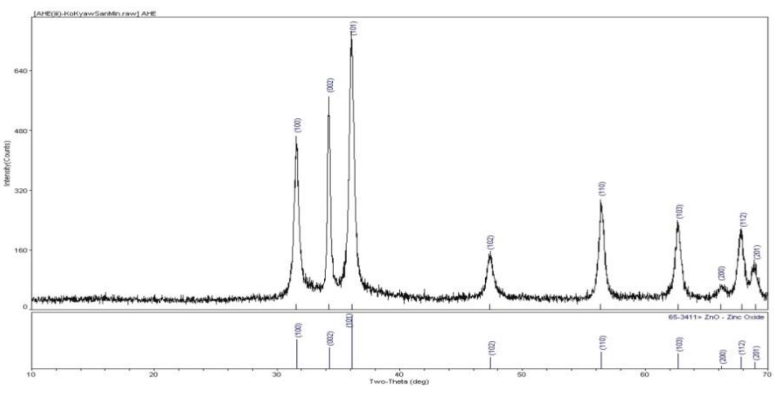


**Figure 3:** XRD diffractogram of zinc oxide nanoparticles prepared by using *Aloe vera* leaf aqueous hot extracts (10 mL) at 120 $^{o}$C

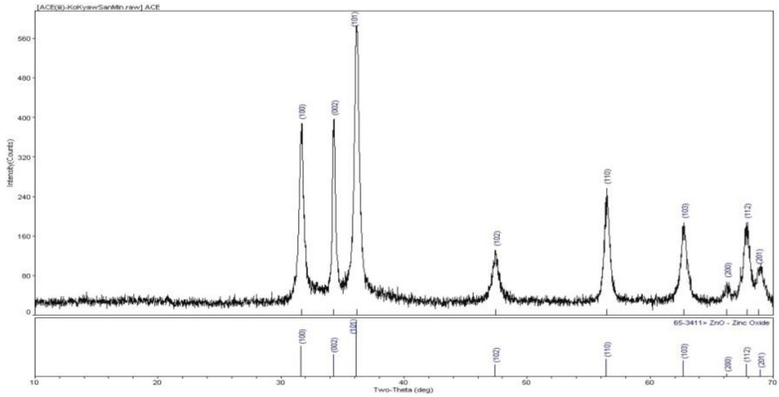


**Figure 4:** XRD diffractogram of zinc oxide nanoparticles prepared by using *Aloe vera* leaf aqueous cold extracts (10 mL) at 120 °C

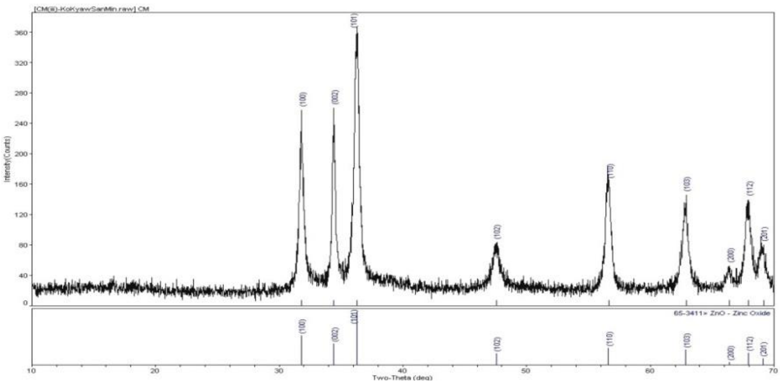


**Figure 5:** XRD diffractogram of zinc oxide nanoparticles prepared by using sodium hydroxide (10 mL) at 120 °C

**SEM analysis**

The SEM analysis was used to identify the size and shape of the zinc oxide nanoparticles. According to the SEM analysis, the morphology of the synthesized all ZnO nanocrystals (Figures 6, 7 and 8) were found to be observed as agglomerated particles at temperature 120 °C in both of biosynthesis and chemical synthesis.

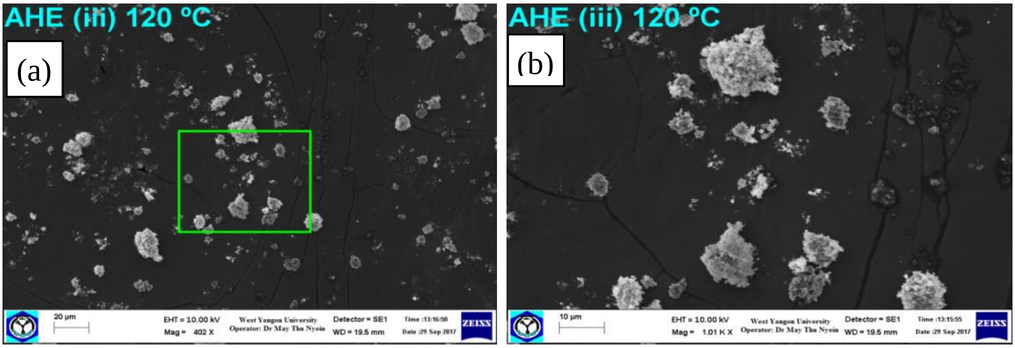


**Figure 6:** SEM images of the synthesized ZnO nanoparticles (ZnO-AHE) prepared at 120 $^{o}$C (a) 402 X magnification and (b) 1000 X magnification

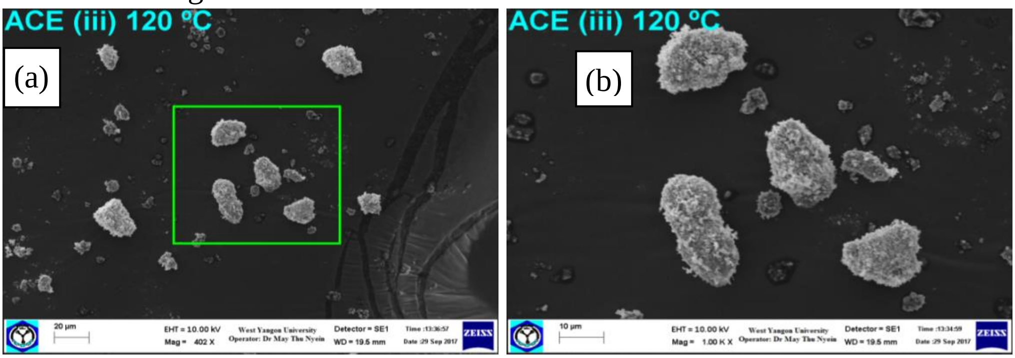


**Figure 7**: SEM images of the synthesized ZnO nanoparticles (ZnO-ACE) prepared at 120 $^{o}$C (a) 402 X magnification and (b) 1000 X magnification

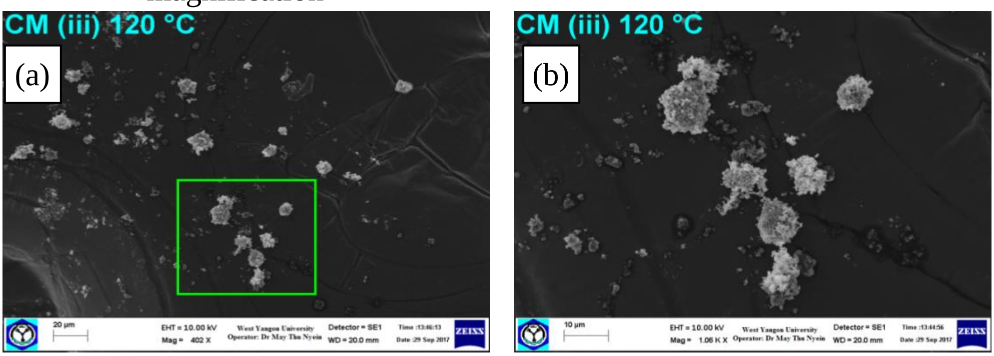


**Figure 8:** SEM images of the synthesized ZnO nanoparticles (ZnO-CM) prepared at 120 $^{o}$C (a) 402 X magnification and (b) 1000 X magnification

**FT IR analysis**

Fourier Transform Infrared Spectroscopy was used to identify the presence of biomolecules which plays an important role in the biosynthesis of nanoparticles. In all of the synthesized ZnO-AHE, ZnO-ACE and ZnO-CM at temperature 120 °C, the absorption bands observed at 3455, 3431 and 3472 $cm^{-1}$ are due to the O-H stretching vibration of water (Figure 9). The peaks at 1414 and 1383 $cm^{-1}$ which correspond to the O-H bending vibration of water for ZnO-ACE and ZnO-CM (Willard *et al.*, 1965). Similarly, the absorption peaks at 916, 903 and 897 $cm^{-1}$ are also due to the formation of tetrahedral coordination of Zn in all samples. The main absorption peaks at 426, 421 and 419 $cm^{-1}$ which indicated the formation of (Zn-O linkage) zinc oxide nanoparticles (Elizabeth and Mary, 2015).

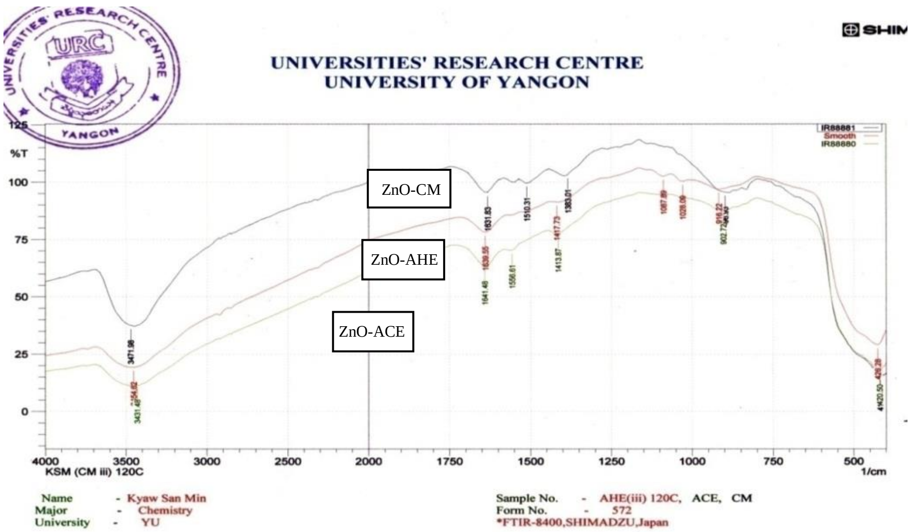


**Figure 9:** FT IR spectra of zinc oxide nanoparticles [ZnO-AHE, ZnO-ACE and ZnO-CM] prepared at 120 °C

**TG-DTA analysis**

The thermogravimetric analysis of the synthesized zinc oxide nanoparticles was performed on Perkin-Elmer thermogravimetric-differential analyzer. It showed that the small endothermic peaks (Figures 10, 11 and 12) for loss of weight (5.10 % in ZnO-AHE, 3.23 % in ZnO-ACE and 2.85 % in ZnO-CM) at nearly 200 °C due to the loss of water. In TG-DTA thermogram of ZnO-ACE nanoparticles (Figure 11), a small exothermic peak was continuously observed at nearly 370 $^{o}$C, indicating the loss of weight in 1.94 % due to the loss of other organic residues components in the plant extracts. On the other hand, the weight loss (1.19 %) in ZnO-CM nanoparticles, a small exothermic peak (Figure 12) occurred at 370 $^{o}$C might be due to the decomposition of precursor leads to the formation of zinc oxide (Zhaol *et al.*, 1998).

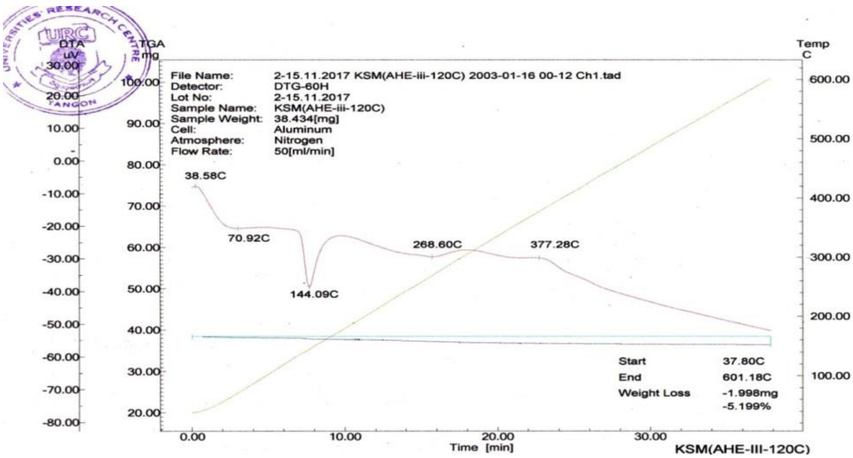


**Figure 10:** TG-DTA thermogram of the synthesized zinc oxide nanoparticles (ZnO-AHE) prepared at 120 $^{o}$C

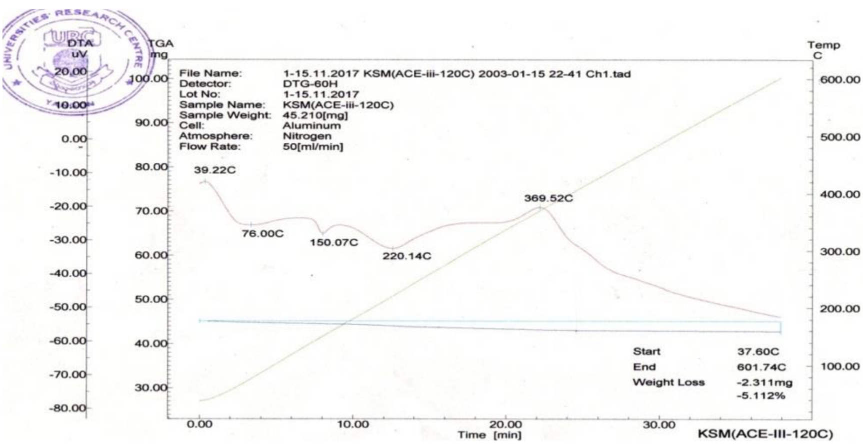


**Figure 11:** TG-DTA thermogram of the synthesized zinc oxide nanoparticles (ZnO-ACE) prepared at 120 $^{o}$C

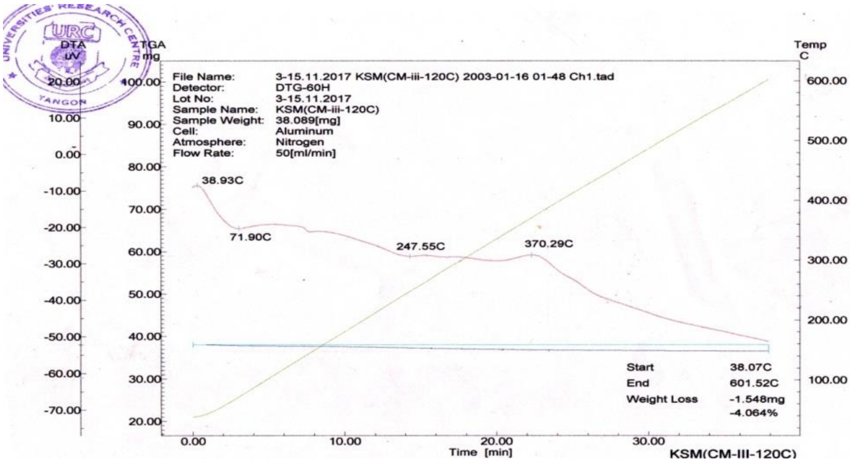


**Figure 12:** TG-DTA thermogram of the synthesized zinc oxide nanoparticles (ZnO-CM) prepared at 120 $^{o}$C

## Conclusion

The zinc oxide nanoparticles were synthesized by using zinc acetate as metal ion precursor materials and *Aloe vera* leaf aqueous extracts as reducing and stabilizing agents. Zinc oxide nanoparticles have been successfully synthesized by using *Aloe vera* leaf extracts (hot and cold) and also by chemical method. Preliminary phytochemical constituents in *Aloe vera* leaf aqueous extracts were investigated. In this study, alkaloids, α-amino acid, coumarins, flavonoids, glycosides, tannins and terpenoids were found to be observed while the other compounds (carbohydrates, cyanogenic glycosides, phenolic compound, quinones, saponins and steroids) were not present in *Aloe vera* leaf aqueous extracts. The zinc oxide nanoparticles were respectively obtained as a light brown crystalline powder (ZnO-AHE) and as a pale yellow crystalline powder (ZnO-ACE) while obtained as white crystalline powder (ZnO-CM). The synthesized zinc oxide nanoparticles were characterized by using several techniques such as XRD, SEM, FT IR and TG-DTA. The synthesized zinc oxide nanoparticles with average crystallite size about 18 ~ 19 nm had hexagonal wurtzite structure with the lattice constants of a = b = 3.2511 Å and c = 5.2076 Å, according to XRD analysis. The SEM analysis of ZnO exhibited the morphology of the samples to be networked with some agglomeration. The FT IR studies exhibited a characteristic absorption peaks of the synthesized ZnO NPs nearly at 450 $cm^{-1}$ (Zn-O linkage) which indicated the formation of zinc oxide nanoparticles.

In this research, according to the XRD and SEM analyses of the synthesized zinc oxide nanoparticles are not different between the two methods used : biosynthesis and chemical methods. However, the amount of yield percents of the synthesized ZnO NPs obtained by biosynthesis method was higher than those of the chemical method at the same temperature. Since, from the results of XRD and FT IR analyses, there was no significant impurity present in all samples synthesized by these two methods at temperature 120 $^{o}$C. From the results of TG-DTA analysis, the weight loss of all samples were in the range of 4.064 % to 5.199 % and it was also observed no significantly changed by the temperature affect, except a little amount of weight loss due to removal of water absorbed. The zinc oxide nanoparticles obtained from chemical method were slightly more stable than those of the biosynthesis method. Small weight losses of zinc oxide samples indicated the thermal stability of the synthesized zinc oxide nanoparticles.

Therefore, these two methods used in this study have advantages to prepare zinc oxide nanoparticles that might be due to the lack of toxicity, less expensive, easy and faster process.

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