

Asian Journal of Phytomedicine and Clinical Research

Journal home page: www.ajpcrjournal.com



PREPARATION AND CHARACTERIZATION OF ZnO NANOPARTICLES USING *MORINGA OLEIFERA* EXTRACT BY GREEN SYNTHESIS METHOD

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ABSTRACT

In this communication, preparation of Zinc Oxide nanoparticles using *Moringa Oleifera* aqueous extract from leaf, flower and bark by green synthesis sol-gel method is done. Zinc Oxide Nanoparticles are analyzed by X-ray diffraction (XRD), Scanning Electron Microscope (SEM), UV-Visible and Fourier Transform Infrared spectroscopy (FTIR). From UV-Visible spectroscopy, higher band gap energy of 4.3eV is obtained in the near visible region at the wavelength of 286.5 nm. Among these extract prepared from leaf, flower and bark of *Moringa Oleifera*, ZnO nanoparticles using bark of the plant is shown sharp peaks in XRD confirming the crystallinity of the particles.

KEYWORDS

Zinc Oxide Nanoparticles, *Moringa Oleifera* and Green Synthesis.

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INTRODUCTION

Nowadays researchers attempt extensive research for the synthesis of nanoparticles using the extract obtained from different plants and succeeded in the synthesis of Zinc Oxide nanoparticles, silver nanoparticles, TiO₂ nanoparticles etc., in the field of biotechnology and pharmaceutical science. The ZnO nanoparticles is capable of removing sulphur and arsenic from the water. ZnO nanoparticles is prepared using the leaf extract of *Moringa Oleifera* by chemical duration method and by Elumalai *et al.*

(2015) and various characterization studies were done. From XRD spectra, it is observed that ZnO nanoparticles has hexagonal wurtzite structure and antibacterial activity is shown maximum inhibition zone for gram (+ve) bacteria and followed by Gram (-ve) bacteria for the concentration of 200 µg/ml¹. Elumalai and Velmurugan (2015) synthesized ZnO nanoparticles using aqueous leaf extract of *Azadirachta indica* (L) by sol-gel green synthesis method. The antimicrobial activity of ZnO nanoparticles increased for the increase in concentration of 50, 100 and 200 µg/ml. Also concentration of ZnO nanoparticles increased with increase of H₂O₂ concentration from the surface of ZnO². Anbuvaran *et al.* (2015) prepared ZnO nanoparticles using Zinc nitrate and *Phyllanthus niruri* leaf extract by biological method. ZnO nanoparticles are confirmed with the estimated band gap energy of 3.51 eV and Photoluminescence (PL) intensity observed at 402 nm in the visible region and the intensity of the peak and band gap energy depends on the size of the nanoparticles and geometrical shape³.

The mediated bio reduction of ZnO nanoparticles using leaf extract of *Solanum nigrum* by green biosynthesis method was prepared and characterized by Ramesh *et al.* (2014). The SEM results of the study reveal that the diameter of the ZnO nanoparticles are in the range of 20-30 nm⁴. Suresh *et al.* (2015) synthesized multifunctional ZnO nanoparticles from Zinc nitrate hexahydrate and aqueous leaf extract of *Arto carpus gomezianus* by solution combustion method. The morphology of the ZnO nanoparticles are highly porous and the photocatalytic activity of ZnO nanoparticles is shown the degradation of methylene blue dye exposed to sun light and UV light⁵. Thirunavukkarasu santhoshkumar *et al.* (2014) prepared and tested TiO₂ nanoparticles using *Psidium guajava* aqueous extract. The mediated biosynthesis of titanium dioxide nanoparticles is shown prominent peaks at 3410 cm⁻¹, 1578 cm⁻¹, 1451 cm⁻¹ and 1123 cm⁻¹ respectively. Presence of alkynes, alkanes and C-O is confirmed by FT-IR

spectra and results of FESEM show spherical shape with average particle size of 32.58 nm⁶.

Biosynthesis of Zinc oxide nanoparticles was carried out with the different concentration of Zinc acetate and aqueous extract of *Citrus aurantifolia* by Nurul Ain Samat and Roslan Md Nor (2013). The XRD result mentioned the orientation of reflection plane at (100) with hexagonal structure⁷. Senthilkumar and Sivakumar (2014) synthesized ZnO nanoparticles using the leaf extract of green tea (*Camellia sinensis*) and characterized by various techniques. The results of the UV-Visible spectrum give the absorption peaks at 325 nm and by XRD result, the average size of ZnO nanoparticles is found to be 16 nm. The antibacterial activity and antifungal activities are carried out using Agar well-diffusion method on pathogenic species⁸. Niranjana Bala *et al.* (2015) prepared ZnO nanoparticles using *Hibiscus subdariffa* leaf extract and found that the growth of the ZnO nanoparticles depend on the annealing temperature. It is confirmed that the increase of temperature decreases the size of the particle. Antibacterial activity of the particles is analyzed using gram (+ve) positive and gram negative bacteria⁹. Sagar *et al.* (2015) investigated various parameters of ZnO nanoparticles such as physical properties; antibacterial property, high piezoelectricity and binding energy. Larger potential of Zinc Oxide nanoparticles in pharmaceutical, textile, paint and rubber industries is highlighted¹⁰.

Sagar Raut *et al.* (2015) explained green biosynthesis of zinc oxide nanoparticles using aqueous *Ocimum tenuiflorum* plant extract. The utilized plant is a reducing agent in the direct chemical synthesis of ZnO nanoparticles. From the XRD result, the average particle size is found to be 13.86 nm¹¹. Green synthesis of zinc oxide nanoparticles using aqueous *Cassia fistula* plant extract was done by Suresh *et al.* (2015) and the leaf extract has reducing components such as polyphenols (11%) and flavonoids (12.5%). Absorption peak is recorded at 370 nm in the UV-Vis spectrum and TEM results give the average crystalline size of 5-15 nm¹². Ramesh *et al.* (2014)

prepared zinc oxide nanoparticles using *Citrus aurantifolia* extract by biosynthesis method. Various characterization result reveals that the ZnO nanoparticles are moderately stable, roughly spherical and maximum particle has the size in the range from 9 to 10 nm in diameter¹³.

Manokari *et al.* (2016) prepared ZnO nanoparticles from zinc nitrate hexahydrate and aqueous extract of a medicinal plant (*Micrococca mercurialis*) by sol-gel biosynthesis method. The *M. mercurialis* plant is found to be high in primary and secondary metabolites such as proteins, steroids and alkaloids. The UV-Visible result shows that the prepared ZnO nanoparticles from various parts extract such as leaves, stems, roots and fruits corresponding to the absorbance peaks at 305 nm, 299 nm, 311 nm and 302 nm respectively¹⁴. Rajesh kumar *et al.* (2015) prepared zinc oxide nanoparticles using *Camellia sinensis* leaf extract by sol-gel heating method. The antimicrobial activity of ZnO nanoparticles has the maximum zone inhibition for *Pseudomonas aeruginosa* (32 ± 0.050) and minimum for *Staphylococcus aureus* (25 ± 0.100)¹⁵. Ramesh *et al.* (2014) investigated ZnO nanoparticles prepared from 1 mM of zinc acetate using *Cassia auriculata* flower extract by sol-gel method. Characterization results reveal the presence of Zinc oxide nanoparticles¹⁶. Bhumi and Savithamma (2014) studied ZnO nanoparticles prepared from zinc acetate and sodium hydroxide using bio components of *Catharanthus roseus* leaves extract by biological synthesis method. ZnO Nanoparticles are found to be spherical in shape and the average size of the particles in the range of 23-57 nm¹⁷.

In the present study, an attempt is made to synthesize ZnO nanoparticles using leaf, bark and flower extract of *Moringa Oleifera* as the plant has traditional and medicinal values since 200 years by ancient Tamil Siddhas' and used for various diseases. Moreover, the plant grows in all climatic conditions and specific medicinal value of boosting libido.

Green synthesis of zinc oxide nanoparticles using leaf, bark and flower extract of *Moringa Oleifera*

and Figure No.1. Show the photograph of the drumstick tree.

MATERIAL AND METHODS

Materials

Zinc nitrate hexahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), Zinc acetate dehydrates ($(\text{CH}_3\text{COO})_2 \text{Zn} \cdot 2\text{H}_2\text{O}$), distilled water and leaf, bark and flower extracts of *Moringa Oleifera* is used for the preparation ZnO nanoparticles.

Preparations of Aqueous Extract from *Moringa Oleifera* Leaf

Green leaves of *Moringa Oleifera* are collected and washed with distilled water many times for the removal of dust particles. The leaves are then grinded using mortar and pestle until the leaves are grinded finely. Whatman Filter paper is used to filter the grinded leaves to collect the leaf extract. The extract is collected and kept at the room temperature.

Preparation of Bark Extract

The barks of the tree are collected and cut into small pieces for the convenience. Distilled water is used to wash the barks for many times and dried in a shadow region for 5 days. The 150 grams of dried bark is taken in a 250 mL beaker containing alcohol solution and kept for one day at the room temperature. The color of the solution turned into yellow and the solution is filtered using Whatman filter paper to get pure extract of the bark.

Preparation of Flower Extract

The flowers of the tree are collected and washed for many time using distilled water. Washed flowers are dried in a shadow region for 3 days. The flower becomes dried and 100 grams of the flower is taken in a 250 mL beaker containing 150 mL distilled water and boiled for 20 minutes at 90°C. The flower extract turns yellow kept inside the refrigerator for 3h. Finally the extract is filtered for many times using Whatman filter paper.

Green Synthesis of ZnO Nanoparticles Using *Moringa Oleifera* Leaf Extract

Zinc nitrate of 0.2M with the prepared leaf extract of the plant is taken in a beaker and using magnetic stirred, the solution is continuously stirred at 80°C.

The stirring is continued until, a colloid of the solution is obtained. The colloidal solution is dried in hot air oven in the range of 120-130°C for 1h and annealed at 300°C for 1h in Muffle furnace. The yellow color particles obtained is crushed in ceramic mortar pestle to get ZnO nanoparticles.

Similarly, ZnO nanoparticles are prepared using the leaf and flower extract of *Moringa Oleifera*. Various characterization studies are carried out for the structural, morphological and physical properties of the prepared ZnO particles. Figure No.2 shows the graphical flow-chart for synthesis of ZnO nanoparticle using *Moringa Oleifera* extract.

RESULTS AND DISCUSSION

UV-Visible spectra analysis

ZnO nanoparticles prepared using the extract obtained from the leaf, flower and bark of *Moringa Oleifera* is subjected to record UV-Vis spectroscopy. Figure No.3 (a), 3 (b) and 3 (c) shows the photograph of the UV-Vis spectra of ZnO nanoparticles prepared from the extracts of *Moringa Oleifera*. The absorption peaks is obtained at the wavelength 286.5, 285.89 and 289.93nm for ZnO particles prepared from leaf, bark and flower of *Moringa Oleifera*. From the UV-Vis graphs, energy band gap is obtained as 4.3, 4.3 and 4.2eV using the formula

$$E_g = 1240 / \lambda_{max} eV \quad (1)$$

FTIR spectra analysis

To identify the functional groups involved in the ZnO particles prepared from the leaf, bark and flower extract of *Moringa Oleifera*, FTIR- spectra is recorded in the wavelength from 400cm⁻¹ to 4000 cm⁻¹.

The Figure No.4 shows FTIR analysis for ZnO nanoparticles using *Moringa Oleifera* aqueous extract and measured absorption band is carried out in the wave number range from 400 cm⁻¹ to 4000 cm⁻¹. Figure No.4 (a), 4 (b) and 4 (c) shows the photograph of the FTIR spectroscopy recorded for the prepared samples. From the Figure No.4 (a), it is observed that, Energy band at 3517.99, 1652.93, 1010.77 and 828.85 cm⁻¹ corresponds to the O-H,

O-amino, C-O stretching and C-Cl stretching vibrations respectively. Table No.1 represents the energy band and its corresponding functional group identified using FTIR spectra.

XRD Analysis

XRD spectrums of ZnO nanoparticles using the leaf, bark and flower extract of *Moringa oleifera* are shown in the Table No.2, Figure No.5 (a), 5 (b) and 5 (c). The diffraction peaks are obtained for 2θ values viz., 31.85, 34.32, 36.47, 47.61 and 56.62 correspond to the lattice planes (100), (002), (101), (102) and (110). The XRD spectrum reflects hexagonal wurtzite structure and it is in agreement with JCPDS data No: 89-7102 and also the diffraction peaks indicate the crystallinity of the particles. The crystallite average size is calculated using Scherrer equation

$$D = K\lambda / \beta \cos\theta \quad (2)$$

Where D is the mean size of crystallites (nm), K is crystallite shape factor a good approximation is 0.9, λ is x-ray wavelength (1.540Å), B is full width at half the maximum (FWHM) in radians of the X-ray diffraction peak and θ is the Bragg angle. The lattice parameters can be calculated by using the following relation.

$$1/d^2 = 4/3 [h^2+hk+l^2/a^2] + l^2/c^2 \quad (3)$$

SEM Analysis

SEM photograph is taken for ZnO nanoparticles in different resolutions and depicted in the Figure No.6-8. Figure No.6 shows the SEM result of ZnO nanoparticles obtained from the leaf extract of *Moringa Oleifera*. It is observed that the particles show flakes like structure and there are no pores. Figure No.7 represents the SEM image of ZnO nanoparticles synthesized using bark extract and it is observed that the flakes formed are clinged together and it reveals that the particle size decreases with increase in annealing temperature. Similarly, SEM photographs of ZnO nanoparticles from the flower extract are shown in the Figure No.8. From the figure, it is clear that the particles are granular nanosized and size of the particle ranges from 14-21nm.

Table No.1: FTIR results of ZnO nanoparticles (Leaves Extract)

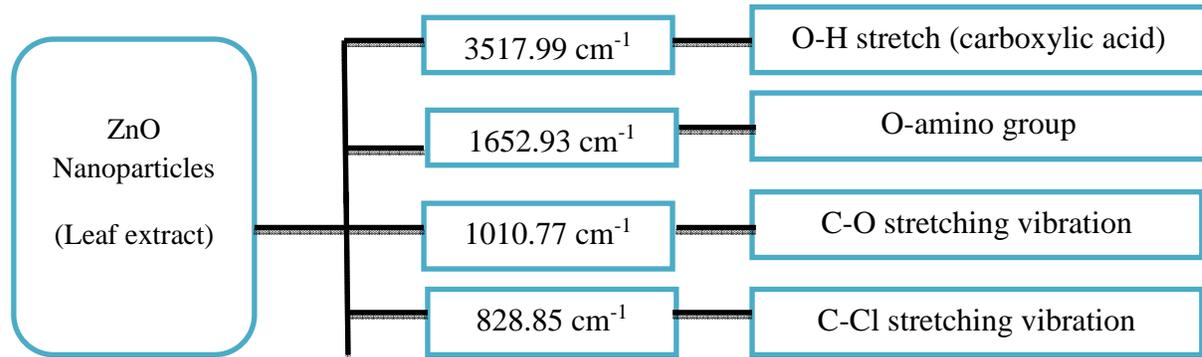


Table No.2: Shows XRD results grain size and lattice parameter of ZnO nanoparticles

| S.No | Sample (<i>Moringa Oleifera</i>) | Grain Size(nm) | Lattice Parameter Å | |
|------|---------------------------------------|----------------|---------------------|--------|
| | | | a | c |
| 1 | ZnO NPs (leaf extract) | 16.63 | 3.2870 | 3.4534 |
| 2 | ZnO NPs (bark extract) | 11.10 | 3.2461 | 5.2121 |
| 3 | ZnO NPs (flower extract) | 11.73 | 3.2313 | 3.3162 |



Figure No.1: Drumstick tree (*Moringa Oleifera*)

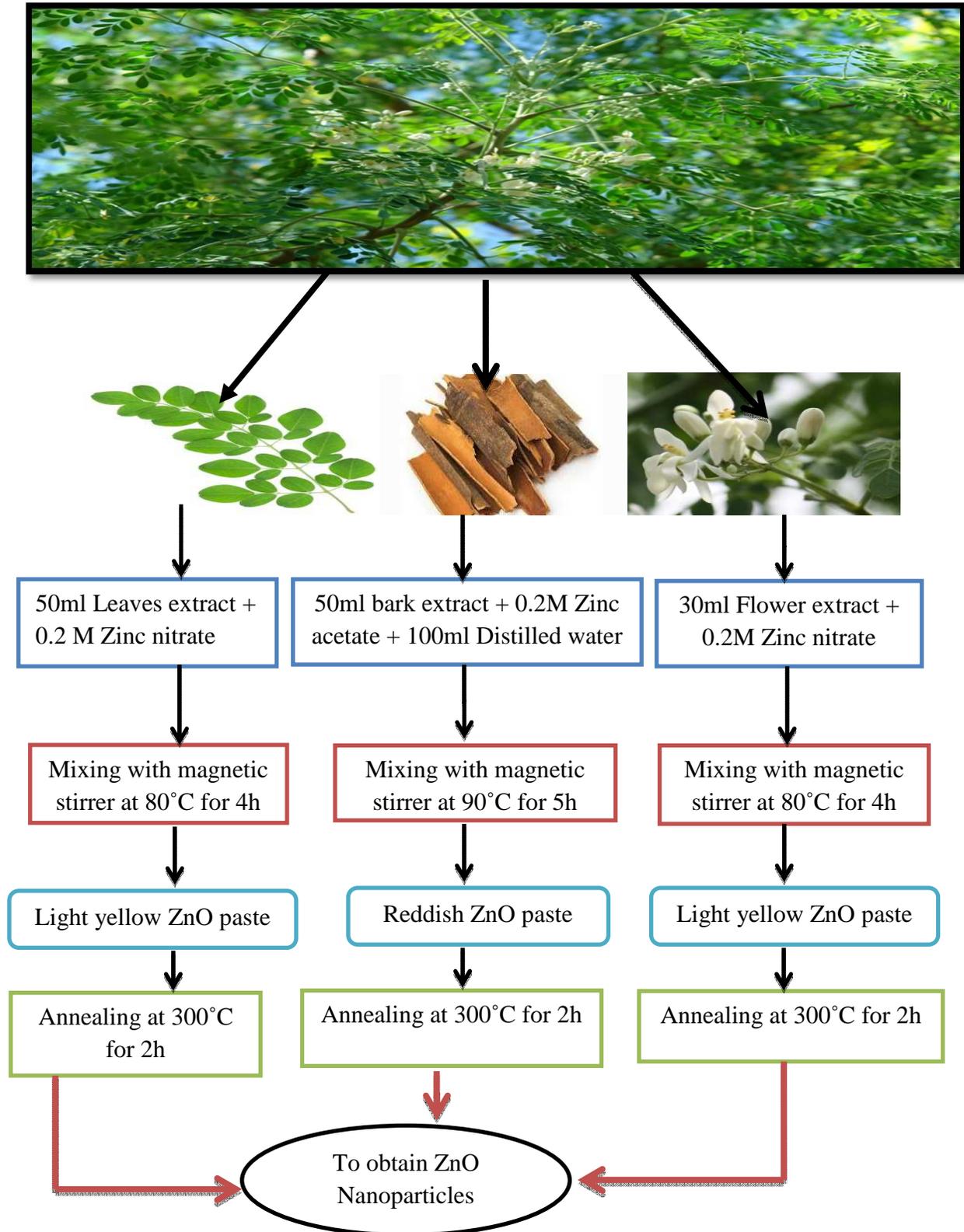


Figure No.2: Synthesis of ZnO nanoparticles using *Moringa Oleifera* plant

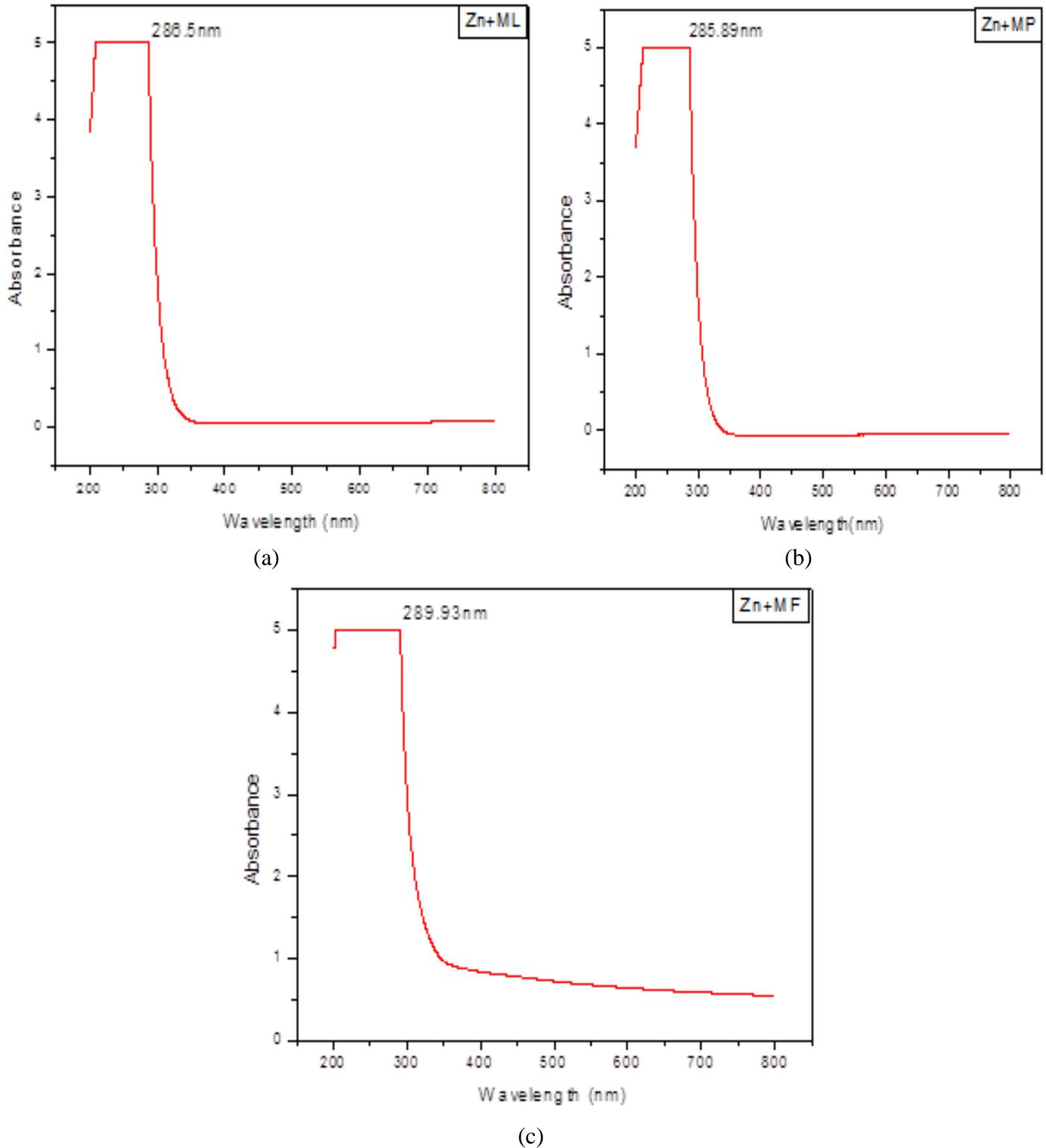
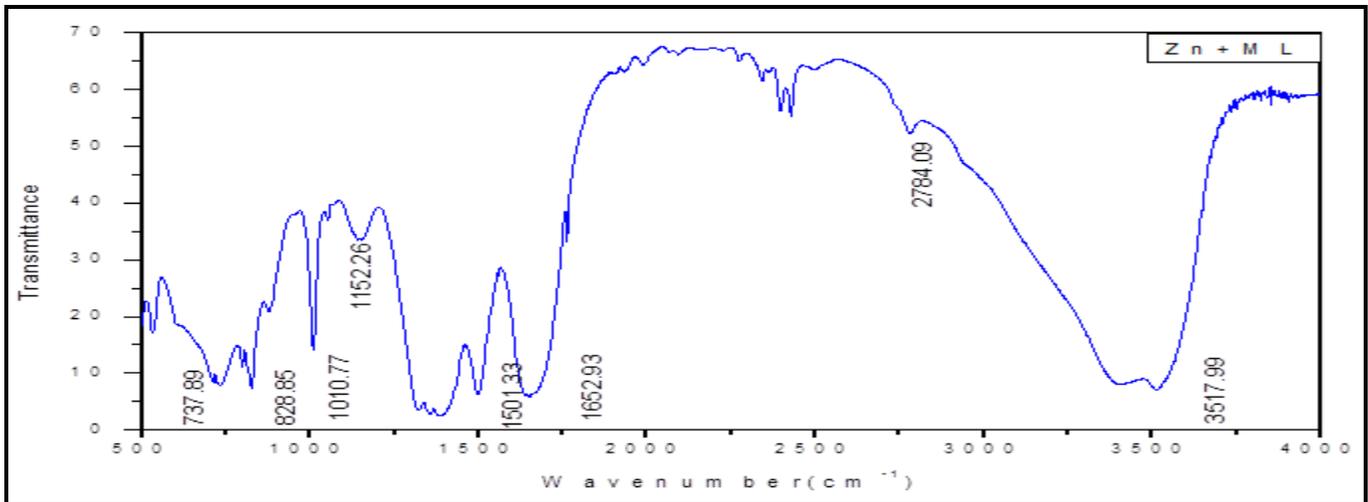
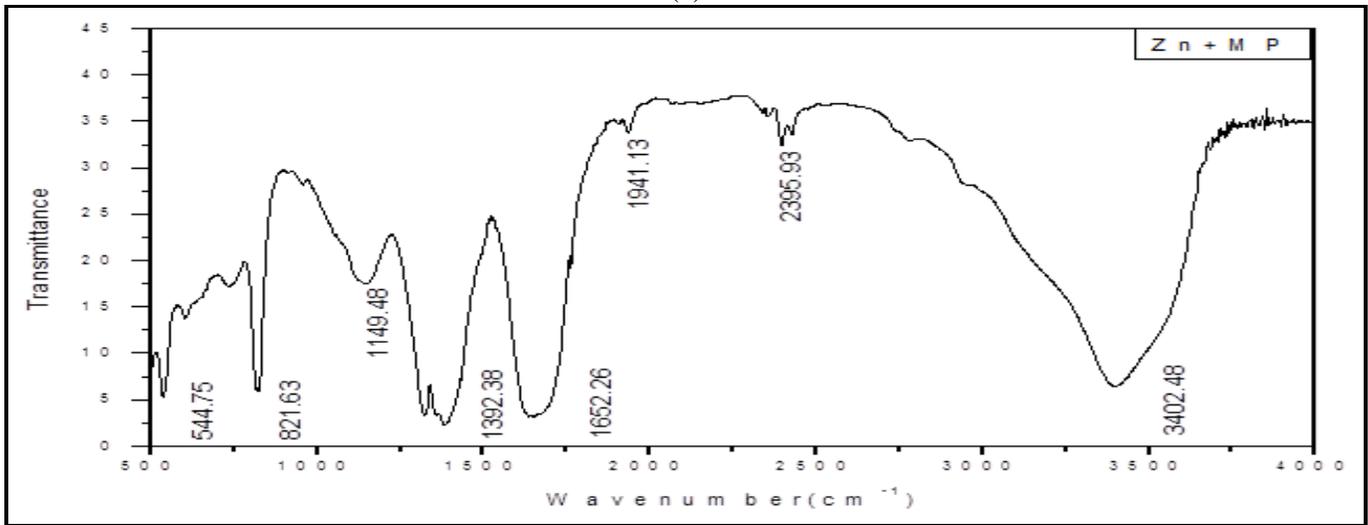


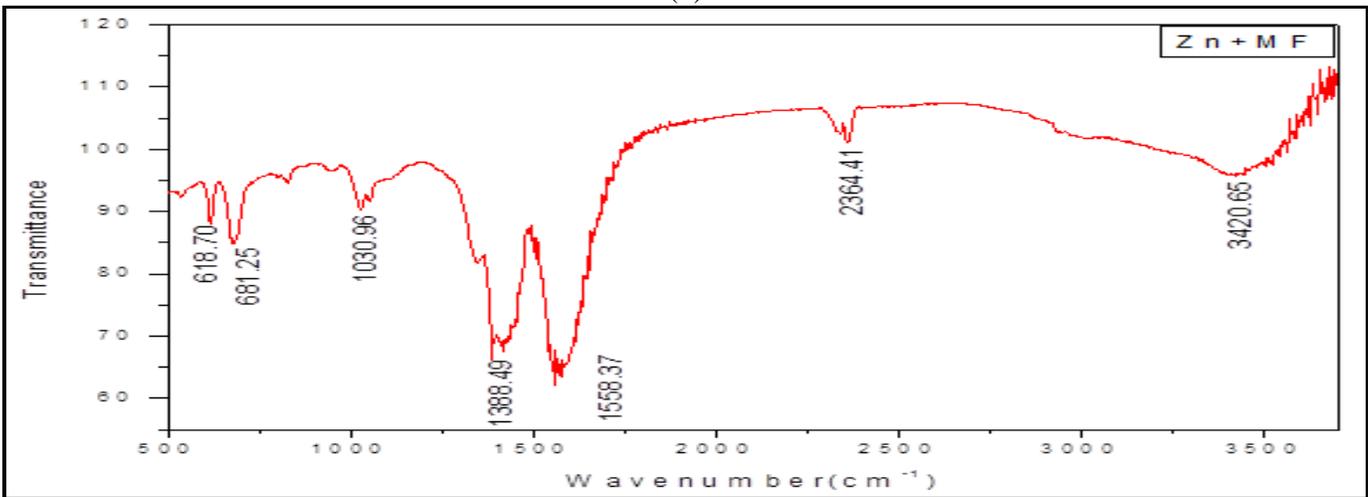
Figure No.3: (a) Leaf Extract (b) Inner Bark Extract (c) Flower Extract



(a)

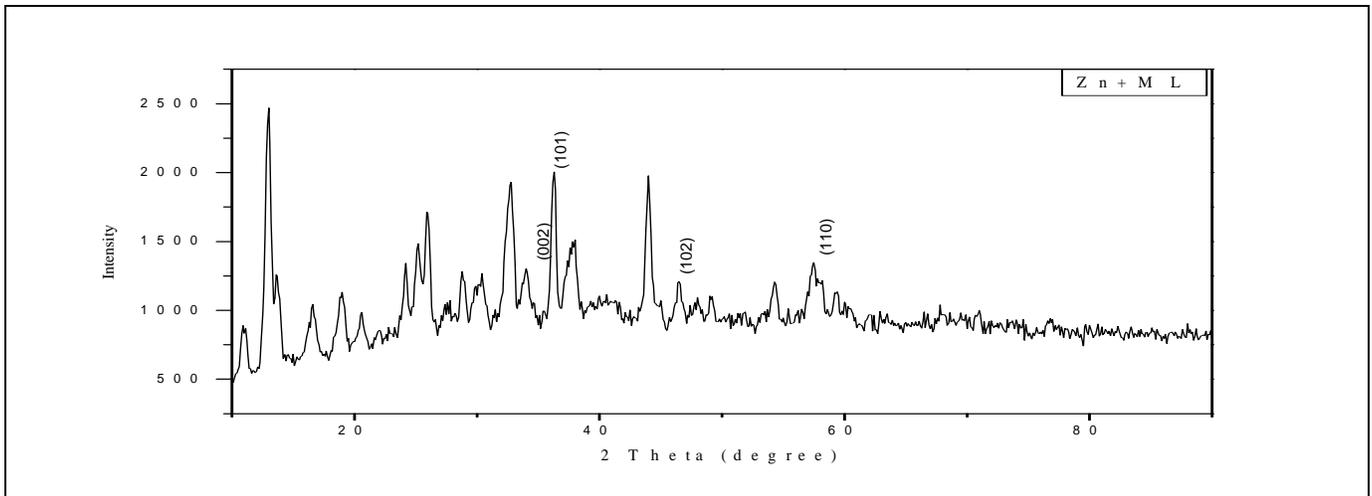


(b)

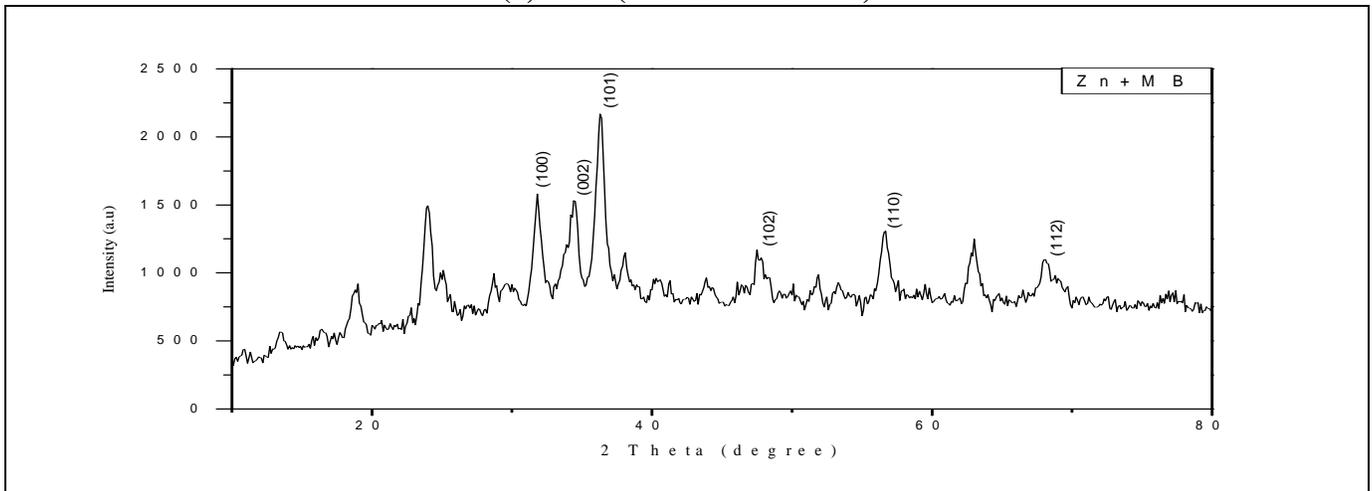


(c)

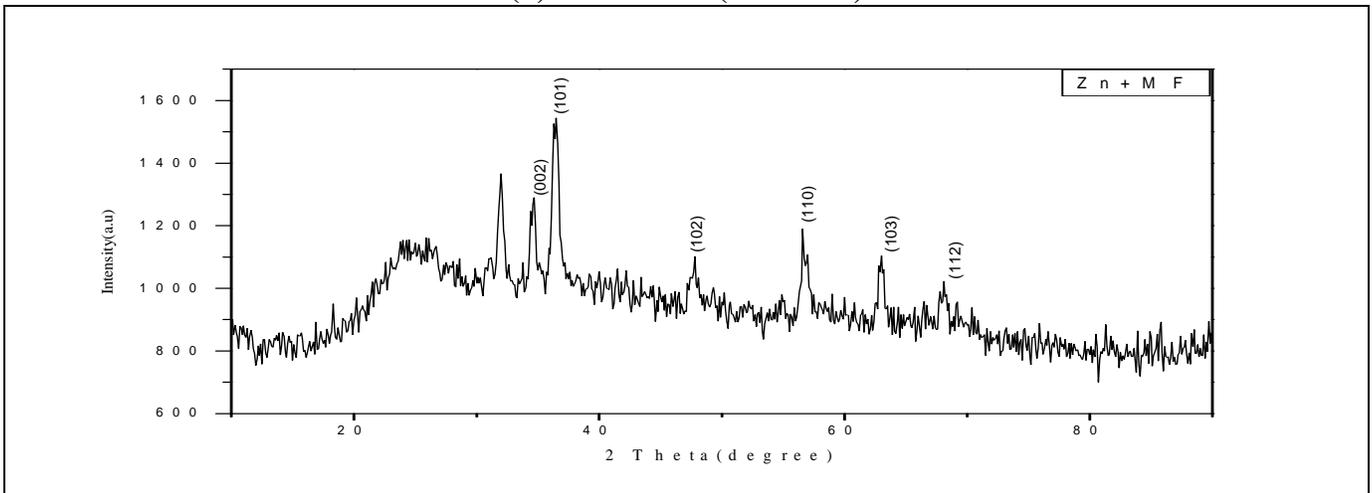
Figure No.4: (a) Leaf Extract (ZnO NPs) (b) Inner Bark Extract (ZnO NPs) (c) Flower Extract (ZnO NPs)



(a) Leaf (Extract ZnO NPs)



(b) Inner Bark (ZnO NPs)



(c) Flower Extract (ZnO NPs)

Figure No.5: XRD patterns of ZnO NPs using *Moringa Oleifera* (a) Leaf Extract, (b) Bark Extract and (c) Flower Extract

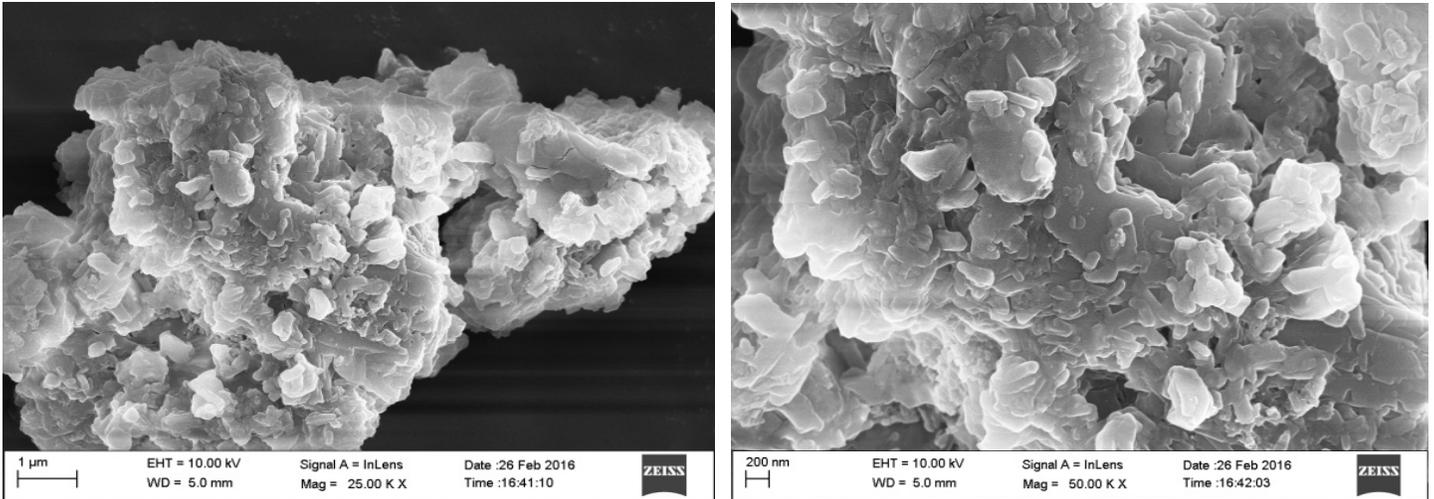


Figure No.6: SEM Image of ZnO NPs (leaf extract)

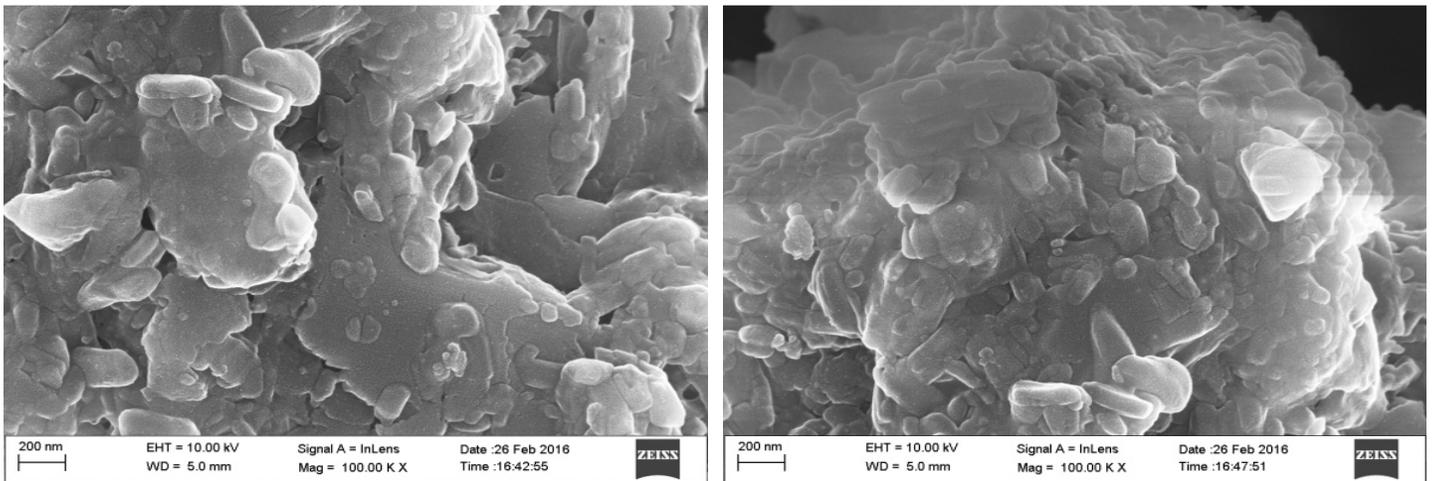


Figure No.7: SEM Image of ZnO NPs (inner bark extract)

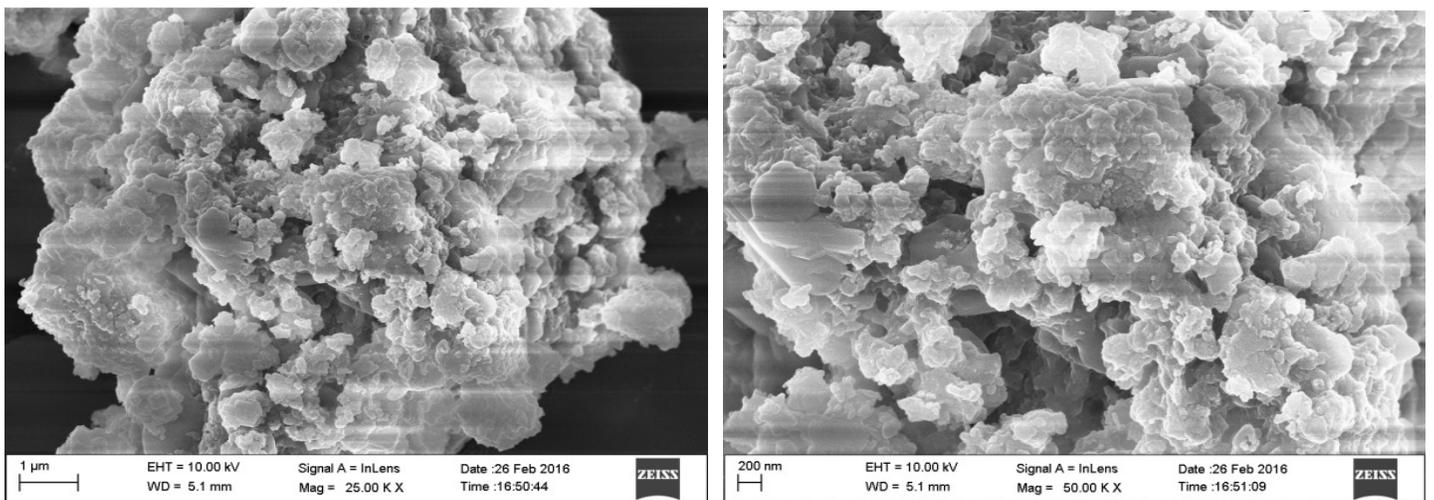


Figure No.8: SEM Image of ZnO NPs (flower extract)

CONCLUSION

Zinc oxide nanoparticles using the leaf bark and flower extract of *Moringa Oleifera* is synthesized by sol-gel heating method. From the UV-Vis spectroscopic study, the energy band gap reached the maximum value of 4.3eV at 286.5nm. Diffraction peaks observed in the XRD spectrum reveals the hexagonal wurtzite structure and confirmed with the values from JCPDS data. Size of the nanoparticles is dependent on the annealing temperature and the value of the size of the nanoparticle is in the range of 14-21 nm. FTIR results confirm the presence of functional group such as carboxylic acid, alcohol, and O-amino group.

ACKNOWLEDGEMENT

The authors very thankful to my guide, Faculty members and M.Sc students in the department of physics, Karpagam Academy of Higher Education. I would also like to thank the management for providing laboratory facilities.

CONFLICT OF INTEREST

We declare that we have no conflict of interest.

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Please cite this article in press as: Thirunavukkarasu C et al. Preparation and characterization of ZnO nanoparticles using *moringa oleifera* extract by green synthesis method, *Asian Journal of Phytomedicine and Clinical Research*, 4(3), 2016, 121 - 132.