

Synthesis of ZnO Nanoparticles & Biological Evaluation

Using Millettia Pinnata Leaf Extract

ELLA.RAJESH¹, Prof .B. VENKATESWARA RAO², Dr.M. PADMA³, Dr. CH. MADHU⁴

Department of engineering chemistry, Andra University, Visakhapatnam – 530003

Abstract:

In this study Zinc oxide nps were promptly synthesized from $Zn(NO_3)_2$ solution and formation of nps observed with in green method. Characterize Zinc oxide nps using IR, Scanning electron Microscope (Fe-SEM), AND X- ray diffract meter. Additional its antimicrobial activity against S.aureus, Escherichia coli is studied.

Key words:Nanoparticles, Zinc oxide, S.aureus, Escherichia coli.

1. INTRODUCTION

Nanomaterials find a huge range of applications due to their exciting physical, chemical and catalytic properties [1-14]. The effect of zinc oxide nanoparticles on antibiotics has been studied keeping in mind the fact that zinc oxide nanoparticles have an intrinsic bactericidal effect of their species [15-21]. Nanocrystalline ZnO particles have been found multiple range of applications in the fields of high sensitivity biomolecular detection, diagnostics, antimicrobials, therapeutics, catalysis, and micro-electronics [22-27]. The Millettia Pinnata is used as a traditional medicine in the treatment and prevention of several kinds of ailments in many countries such as for treatment of piles, skin diseases, and wounds [28-34].zinc oxide(ZnO) nanoparticles have a particular focus on plant-based synthesis [35-38]. Leaf extracts of a diverse range of millettia Pinnata have been successfully used in making ZnO nanoparticles [39-42].

2. MATERIALS AND METHOD

2.1. PREPARATION OF MILITIA PINNATA LEAF EXTRACT



Fig1:Millettia pinnate leaves

Initially, Indian Beech tree leaves (Millettia pinnate) leaves are cleaned several times using distilled water to remove the dust particles and residual moisture. 20g of thoroughly washed finely cut militia pinnate leaves in 500ml Erlenmeyer flask along with 100ml of distilled water and then boiling the mixture at 70°C until it turns into a brownish colour. Further, the extract was filtered with Whatman no:1 filter paper, and the extracted solution was used for further process.

2.2. Synthesis of ZnO Nanoparticles

To do this firstly we take 20grams of Zinc Nitrate and dissolved in 20ml of distilled water. Now add the above prepared leaf extract solution to this Zinc nitrate solution drop by drop for about 5ml.Both the solutions are mixed together on magnetic stirrer for about 30 min.Thus formed solution is centrifuged further at 300rpm. The solution is separated and dried in an oven for 80°C for about 20hrs. The precipitate is washed several times using distilled water and is filtered using nano filter paper. Finally, Zinc oxide nanopowder is obtained and used for further process.

3. Results and Discussions

3.1. X-ray Diffraction Analysis

X-ray diffraction is an analytical technique generally used for phase identification of a crystalline material and can provide information on a unit cell dimensions as well. X-ray diffraction is now a common technique for studying crystal structures and atomic spacing. Although single-crystal X-ray crystallographic investigation is the most precise source of information regarding the structure of a complex, the difficulty of obtaining crystalline complexes renders this method unsuitable for that study. However, a variety of other spectroscopic techniques could be used with good effect for characterizing the metal complexes in X-ray powder diffraction. The X-ray powder diffraction (XRD) measurements of three complexes are performed. The diffractogram obtained complexes have been given in figures and the observed diffraction data, with the help of the data obtained from the powder XRD, the particle size calculations are performed using Scherrer equation. The diffraction pattern is recorded and radius 2θ values of 36.088 and 18.768 are observed, which corresponds to Bragg reflections of hexagonal wurtzite structure.

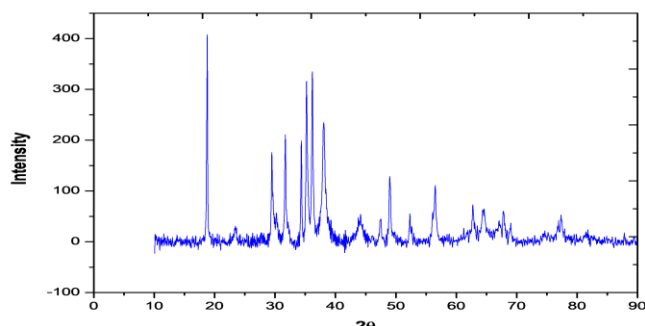


Fig 2: XRD Pattern for ZnO by phytochemical method

3.2 Field Emission-Scanning Electron Microscopy (FE-SEM)

The most widely used type of electron microscopic technique is Field emission scanning electron microscope (FE-SEM). It resolves and examines the microscopic structure of the sample by scanning the surface/fractured surface of materials with higher resolution and much greater depth of field. The most important feature of FE-SEM is the three-dimensional appearance of its images because of its large depth of field. FE-SEM enables us to obtain chemical information from the specimen by using various techniques, including the X-ray energy dispersive spectrometer (EDS). The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface, topography, composition, and electrical conductivity.

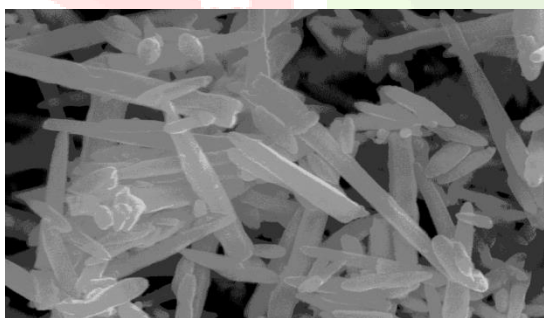


Fig 3: FE-SEM image of ZnO nanoparticles via phytochemical method

The synthesized ZnO nanoparticles were examined by FE-SEM and we can observe that rod shaped nanoparticles were formed in fig 3.

3.3 Energy Dispersive X-ray Spectroscopy (EDS):

Energy-dispersive X-ray spectroscopy is an analytical technique used for the elemental analysis or chemical characterization of a sample. It focuses on the investigation of an interaction of some source of X-ray excitation and a sample. Its characterization capabilities are placed in large part to the fundamental principle that each element has a unique atomic structure allowing a unique set of peaks on its X-ray spectrum. To stimulate the emission of characteristic X-rays from a specimen, a high-energy beam of charged particles such as electrons or protons or a beam of X-rays is focused into the sample being studied. At rest, an atom within the sample contains ground state (or unexcited) electrons in discrete energy levels or electron shells bound to the nucleus. The

incident beam may excite an electron in an inner shell, ejecting it from the shell while creating an electron-hole where the electron is an electron from an outer, higher-energy shell then fills the hole, and the difference in energy between the higher-energy shell and the lower energy shell may be released in the form of an X-ray. The number and energy of the X-rays emitted from a specimen can be measured by an energy-dispersive spectrometer. As the energy of the X-rays is characteristic of the difference in energy between the two shells, and of the atomic structure of the element from which they are emitted, this allows the elemental composition of the element is measured.

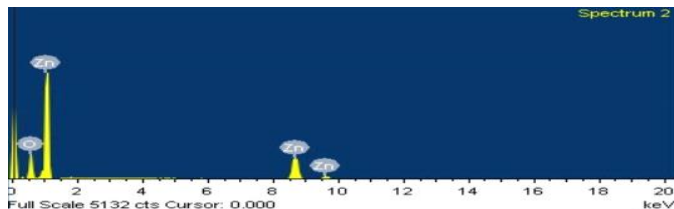


Fig 4EDX of ZnO

The energy dispersive spectrum figure revealed that the clear identification of elemental composition profiles of the synthesized nanoparticles. Zinc nanoparticles typically show optical absorption peak at 1 KeV to the surface Plasmon resonance. However other electrical signals along with Zinc Oxide nanoparticles are also recorded, which are not observed in the many other biosynthesis of nanoparticles. The elemental composition of Zinc and Oxygen is shown above Table 1.

| Element | Weight% | Atomic% |
|--------------|--------------|--------------|
| O | 24.62 | 57.17 |
| Zn | 75.38 | 42.83 |
| Total | 100.0 | 100.0 |

Table 1 Elemental Composition of Zinc Oxide NP

3.4 Fourier Transform Infrared Spectroscopy

ZnO nanoparticles of two milligrams are prepared by mixing with 200 mg of spectroscopic grade KBr.

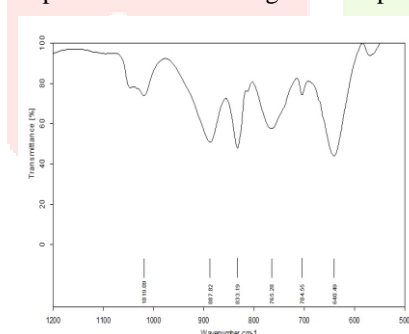


Fig 5:FTIR for Zno

| S.No | Wavenumber (cm ⁻¹) | Peak Assignments |
|----------|--------------------------------|----------------------------------|
| 1 | 1019 | O-C Stretching |
| 2 | 887 | CH₂ Stretching |
| 3 | 833 | P-O Stretching |
| 4 | 640 | C-H Bending |

Table 2Peak Assignments of Zinc Oxide

FTIR spectra are recorded using a Nicolet 520P spectrometer with the detector at 1200-400 cm^{-1} resolution and 20 scans per sample. FTIR Spectra of aqueous Zinc oxide nanoparticles prepared from the Ganuga Leaves extract was carried out to identify the possible biomolecules responsible for capping and efficient stabilization of the metal nanoparticles synthesized by leaf broth. The presence of biomolecules are in the range observed from 1200 cm^{-1} to 400 cm^{-1}

3.5. Antimicrobial screening of

This is screened in vitro for antibacterial activities against *E. coli* and *S. aureus* antifungal activity against *C. albicans* by Agar-well diffusion method. The antibacterial and anti-fungal activities of complex are listed in table 2.



Figure 6. Inhibition zones for complex against *S. aureus* and *E. coli*

| Bacteria | Inhibition zone (mm) |
|------------------|----------------------|
| <i>E. coli</i> | 9 |
| <i>S. aureus</i> | 12 |

Table 3. Antimicrobial activities of ZnO

According to above table the ZnO showed antibacterial activity against *E. coli* and *S. aureus*. The obtained inhibition zones are 9 and 12 mm.

4. Conclusion

In this work, Zinc Oxide nanoparticles are synthesized by phytochemical method which also known as green synthesis. The prepared ZnONP's are characterized by using spectroscopic, microscopic, conductivity, photoluminescence, and photodegradation studies such as XRD, FE-SEM, FT-IR. From the XRD results, the crystal size of the Green Synthesis method ZnO is less. FE-SEM results revealed the rod-like morphology of the phytochemical method. From the Energy Dispersive Spectroscopy (EDS) the atomic percentage and weight percentage of Zinc and Oxygen of ZnO particle.

5. Acknowledgment

One of the authors (ELLA. RAJESH) is grateful to the Council of Scientific & Industrial Research (CSIR), India, for their financial support.

6. REFERENCES

1. Mohanmad, Vaseem., Ahmad, Umar., Yoon-Bong, Hahn., American scientific publishers, **2010**, 1-36.
2. Manikandan, S., Karthikeyan, N., Silambarasan, M., Rajan, KS., ApplThermEng, **2012**, 44:1-10.
3. Silambarasan, M., Manikandan, S., Rajan, KS., Int. J. Heat MassTransf, **2008**, 55, 7991-8002.
4. Chen, C., Liu, P., Lu., C., Chem Eng J, **2008**, 144:509-13.
5. Khorrami, SA., Mahmoudzadeh, G., Madani, SS., Gharib, F., J. CeramProcess Res, **2011**, 12:504-8.
6. Sudha, M and Rajarajan, M., IOSR Journal of Applied chemistry, ISSN-2278-**2013**, 5736, 3.
7. Kolekar, T., Yadav, H., Bandgar, S., nRaskar, A., Rawal, C., Mishra, G., Indian Streams Research Journal, **2011**, 678-791.
8. NuralSyahiahSabri, T., Ahmad Kamal, Yahya., NurAimi, Jani., MohanmadKamal Harun and Mahesh Kumar, Talari., International Journal of the Institute of Materials Malaysia (IJINM), **2013**, 1.
9. Sharma, D., Sharma, S., Kaith, B.S., Rajput, J., Kaur, M., Appl.Surt.Sci., **2011**, 257,9661-9672.
10. Amekura, V., Plaksin, O., Umeda, N., Takeda, Y., Kishimoto, T., Nand Buchal, C., Mater, H., Res. Soc. Symp, **2006**, 786-795.
11. Chan, H and Ming-Hsun Tsai., Rev. Adv. Mater. Sci, **2008**, 18, 7.
12. Abhulimen, U., Mater, Res. Soc, Symp, **2005**, 2456-66.
13. Shah, M and Al-Shahry, M., JKAU Sci, **2009**, 61.
14. Chen, C.H., Chang, S.J., Chang, S.P., Li, M.J., Chen, I.C., Hsueh, T.J., Hsu, A.D., Hsu, C.L., J. Phys. Chem. C, **2010**, 114, 12422-12426
15. Hsu, C.L., Chen, K.C., J. Phys. Chem. 116, **2012**, 9351-9355.
16. Gao, P.X., Ding, Y., Wang, I.L., Nano Lett. 3, **2003**, 1315-1320.
17. Hu, Y., Zhang, Y., Chang, Y., Snyder, R.L., Wang, Z.L., ACS Nano 4, **2010**, 4220-4224.
18. Yang, J.L., An, S.J., Park, W.I., Yi, G.C., Choi, W, Adv. Mater. 16, **2004**, 1661-1664.
19. Hsueh, T.J., Hsu, C.L., Sens. Actuators B Chem. 131, **2008**, 572-576
20. Spanhel, J., Sol-Gel Sci. Technol , **2006**, 39-7
21. Satoh, Y., Ohshio, Sandm Saitoh, H, **2005** Sci. Technol. Adv. Mater. 6215
22. Meulenkamp, E.A., J. Phys. Chem. B 102, **1998**, 5566-5572
23. Baxter, J.B., Walker, A.M., Van Ommering, K., Aydil, E.S., Nanotechnology 17, **2006**, S304-S312.
24. Wang, Z.L. Mater. Sci. Eng. Rep. 64, **2009**, 33-71.
25. Jiang, C.Y., Sun, X.W., Lo, G.Q., Kwong, D.L., Wang, J.X. Appl. Phys. Lett. 90, **2007**, 263501-263503.
26. Yan, W., Ohtani, K., Kasai, R., Yamasaki, K., phytochem, **1996**, 45(5):1417-1422.
27. Sangeeta, D., Sidhu, H., Thind, S.K., Nath, R., J. Ethnopharmacol, **1994**, 44(2):61-66.
28. Anand, R., Patnaik, G.K., Kulshreshtha DK., Dhawan BN., Ind. J. Exper. Biol, **1994**, 32(8):548-552.
29. Prakash, D., Singh, P.N., Wahi, SP, Indian Drugs, **1985**, 22(6):332-333.
30. Dhar, ML., Dhar, MM., Dhawan, BN., Mehrotra, BN., Ray, C., Indian J. Exp. Biol, **1968**, 6:232-247.
31. Zhang, JD., Xu, Z., Cao, YB., Chen, HS., Yan, L., An, MM., Gao, PH., Wang, Y., Jia, XM., Jiang, YY., J. Ethnopharmacol, **2006**, 103(1): 76-84.
32. Ojha, SK., Nandave, M., Kumari, S., Arya, DS, Indian Drugs, **2006**, 43(2):136-139.
33. Phillips, OA., Mathew, KT., Oriowo, MA, J. Ethnopharmacol, **2006**, 104(3):351-355.
34. Singh, R P., Magesh, S., Rakkiyappan, C. Nanotechnology and application, **6**, **2012**, 43- 51.
35. Liu, X., Chen, N., Xing, X. RSC Advances. 5, **2015**, 54372-54378.
36. Parisi, C., Vigani, M and Rodriguez-Cerezo, E. Nano. **2015**, 10, 124-127.
37. Bedi, P and Kaur., **2015**, 4, 1177-1196.
38. Taheri, M., Qarache, H and Yoosefi, M., STEM Fellowship Journal., **2015**, 1, 1034-46.
39. Elmer, W.H and White, J.C., Environmental Science. Nano 3, **2016**, 1072-1079.
40. Liu, J., Feng, X., Wei, L., Chen, L., Song, B and Shao, L., Critical Reviews in Toxicology, **2016**, 46, 348-384.
41. Kuang, H., Yang, P., Yang, L., Aguilar, Z.P and Xu, H., Journal of Hazardous Materials, **2016**, 317, 119-126.
42. Feng, X., Yan, Y., Wan, B. Environmental Science and Technology, **2016**, 50, 5651.