International Journal of Advanced Research in Physical Science (IJARPS)
Volume 2, Issue 1A, Month 2014, PP 104-111
ISSN 2349-7874 (Print) & ISSN 2349-7882 (Online)
www.arcjournals.org

Microwave-Assisted Method for ZnO Nanoparticles Synthesis Using Ionic Liquids

CH. Ashok
Centre for Nano Science and Technology
Institute of Science and Technology
Jawaharlal Nehru Technological University
Hyderabad/Hyderabad-85/Telangana/India
chinthakuntlaashok@gmail.com

K. Venkateswara Rao
Centre for Nano Science and Technology
Institute of Science and Technology
Jawaharlal Nehru Technological University
Hyderabad/Hyderabad-85/Telangana/India
kalagadda2003@gmail.com

V. Rajendar
Centre for Nano Science and Technology
Institute of Science and Technology
Jawaharlal Nehru Technological University
Hyderabad/Hyderabad-85/Telangana/India
rajendar.nano@gmail.com

CH. Shilpa Chakra
Centre for Nano Science and Technology
Institute of Science and Technology
Jawaharlal Nehru Technological University
Hyderabad/Hyderabad-85/Telangana/India
shilpachakra.nano@gmail.com

K. Ganapathi Rao
Centre for Nano Science and Technology
Institute of Science and Technology
Jawaharlal Nehru Technological University
Hyderabad/Hyderabad-85/Telangana/India
ganapathi.kandregula@gmail.com

Abstract: *Metal oxides exhibit extraordinary properties in the nano level. ZnO nanoparticles have high refractive index, thermal, conductivity, binding, antibacterial, UV-protection etc., due to these properties ZnO nanoparticles are used in many industrial applications such as ceramics, glass, cement, rubber, lubricants, paints, ointments, sealants, pigments, foods, batteries, ferrites and fire retardants, etc. Generally many synthesis methods exist to synthesize ZnO nanoparticles. This present paper is focused on ZnO nanoparticles synthesis by Microwave-Assisted Synthesis method using ionic liquids. Microwave-Assisted synthesis method is very simple, fast, rapid heating and eco-friendly technique. Ionic liquids are having high ionic conductivity, high heat capacity, low toxicity nature and high dissolving capability. The obtained ZnO nanoparticles have been characterized by X-ray diffractometer (XRD), Particle Size Analyzer (PSA), Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectroscopy (EDS), Transmission Electron Microscopy (TEM) and Thermo Gravimetric and Differential Thermal Analyzer (TG/DTA) for crystal structure, average particle size, shape, elemental compositions, morphology and thermal properties respectively.*

Keywords: *ZnO Nanoparticles, Microwave-Assisted Method, XRD, TEM, TG/DTA.*

1. INTRODUCTION

Metal oxide nanomaterials display variety of applications in the field of optoelectronics, catalytic, coatings, semiconductors, sensors, solar cells, ceramics, spintronics and biological [1]. ZnO metal oxide is a polar inorganic crystalline material having very interesting properties such as good electrical, good optical, non-toxicity, high stability, piezoelectric and low cost [2]. In recent years the nano meter range material importance increases drastically [3]. ZnO belongs to II-VI semiconductor group material having large excitation binding energy of 60 MeV and wide band gap of 3.37 eV (at room temperature) due to this reason it is used in optoelectronic device fabrication [4, 5]. However the synthesis of nanomaterials is a big challenge for the researchers, many methods are available to prepare nano sized materials which includes hydrothermal synthesis, thermal decomposition, gas phase reaction, chemical vapor deposition, electro deposition, sol–gel method, chemical precipitation, mechano-chemical, solution combustion and microwave-assisted method. Microwave-assisted method has unique effects compared with the conventional heating because of its interesting properties such as a very short reaction time, rapid homogeneous heating, high purity, ability to produce small inorganic particles and energy efficient [6, 7]. Present work deals with ZnO nanoparticles preparation by Microwave-assisted method using ionic liquids. [8]. Ionic liquids include very fascinating properties like high ionic conductivity, heat capacity, electric conductivity, viscosity, chemical and thermal stability, non-flammability, low vapor pressure, low toxicity and ability to dissolve a variety of materials [9].

2. METHODS

2.1 Experimental Details

Zinc acetate and 1-ethyl-3-methyl-imidazolium tetrafluoro borate were used as initial precursor materials for the preparation of ZnO nanoparticles using Microwave-assisted method. In 100 ml of distilled water; add 0.2 M Zinc Acetate and 0.01 M NaOH alternately with vigorous stirring. After 15 minutes of stirring 1 ml of [bmim] BF4 solution was added drop by drop. Then transparent solution was formed. The obtained solution was kept in Microwave oven for 5 minutes (Microwave oven maintains 2.45 GHz frequency and 180°C temperature), milky white color solution was formed. The obtained solution was cooled up to room temperature and centrifuge at 3000 rpm for 10 minutes and then washed with ethanol and water for several times, dried at 90°C for one hour finally the ZnO nanoparticles were formed.

2.2 Characterization Techniques

The crystal structure and average crystalline size was measured by Bruker D8 X-ray diffractometer. The average particle size was obtained by HORIBA SZ-100 Particle size analyzer. Shape and elemental analysis of the nanoparticles were estimated by S 3400 N Scanning Electron Microscope. The morphology of the nanoparticles was observed by JEM-100 CXII Transmission electron microscope. S-II EXSTRAR 6000 TG/DTA 6300 used for measuring thermal properties.

3. RESULTS AND DISCUSSION

3.1 X-RAY DIFFRACTOMETER

The following Fig. 1 indicates the XRD pattern of ZnO Nanoparticles synthesized by Microwave-assisted method. As prepared Zinc Oxide Nanoparticles confirmed that the Hexagonal structure was formed. . The peaks were observed at 31°, 34°, 36°, 47°, 56°, 62°, 66°, 68°, 69° and 77° with corresponding (h k l) values (100), (002), (101), (102), (110), (103), (200), (112), (201) and (202) respectively. These results were matched with JCPDS card number 36-1451.

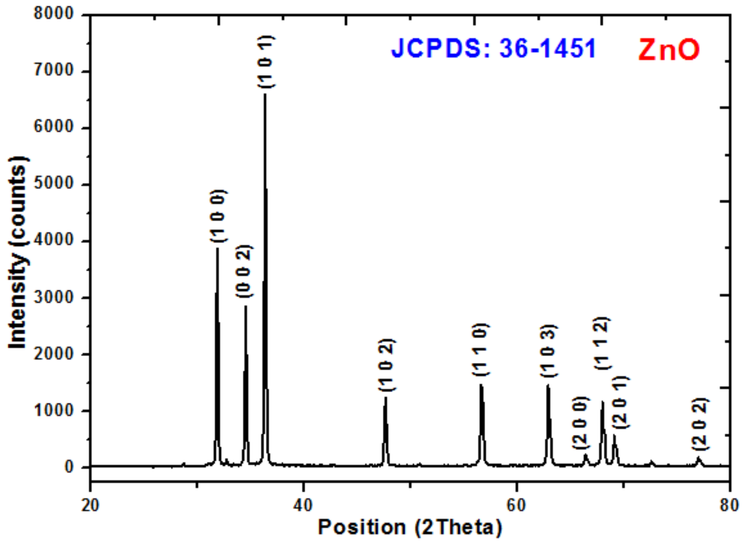

Fig. 1 XRD Pattern of ZnO Nanoparticles

The lattice parameters were a=b=0.3249 nm and c=0.5206 nm. The average crystallite size can be calculated by using Debye Scherer equation,

$$D = \frac{0.9\lambda}{\beta cos\theta} \qquad (1)$$

Where, D- is the average crystallite size of the particle, λ - is the wavelength of radiation of CuKα X-rays, β- is the full width half maximum (FWHM) of the peak, θ = the Bragg's angle. The crystallite size for ZnO nanoparticles is 21nm [10].

3.2 Particle Size Analyzer

Particle size analyzer is used to measure average particle size. Ethanol was used as dispersion medium for ZnO nanoparticles and sonicated using ultra-sonicator. The particles distribution was observed in the form of histogram.

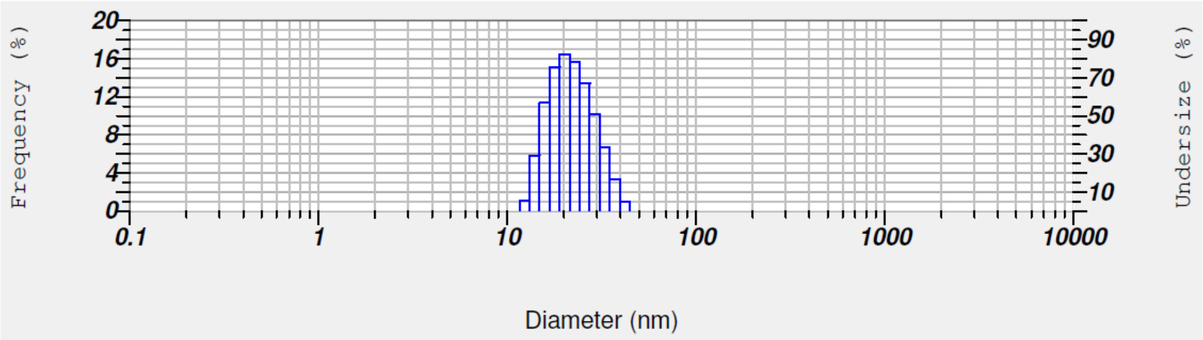

Fig. 2 Particles Distribution of ZnO Nanoparticles

The mean value of the histograms was taken as the average particle size. The average particle size obtained 29nm. This value is nearly equal to the XRD average crystallite size [11].

3.3 Scanning Electron Microscope

The SEM image of ZnO nanoparticles is shown in below Fig. 3. The morphology and size was observed in this SEM image. The image was observed with in the magnification of 5 μm.

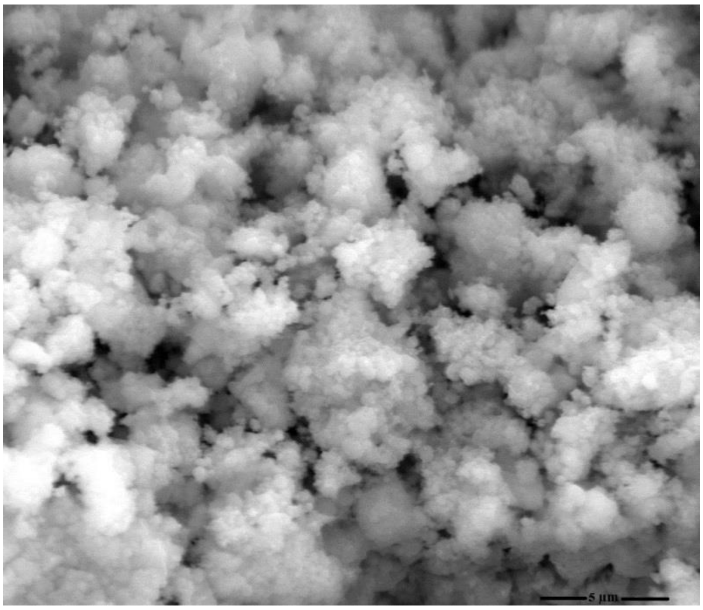

Fig. 3. SEM image of ZnO Nanoparticles

The spherical agglomerated bunch like structures was formed. This structure was formed due to involvement of ionic liquid in the reaction. The size range is 100 nm to 120 nm [12].

3.4 Energy Dispersive X-ray Spectroscopy

The elemental composition was investigated by EDS analysis. The EDS spectrum infers that pure ZnO nanoparticles were formed.

Table 1. Elemental Composition of ZnO Nanoparticles

Element	Weight (%)
Zinc (Zn)	**51.60**
Oxygen (O)	**48.40**

The weight percentages of Zinc and Oxygen are almost equal. The weight percentages were shown in above Table 1.

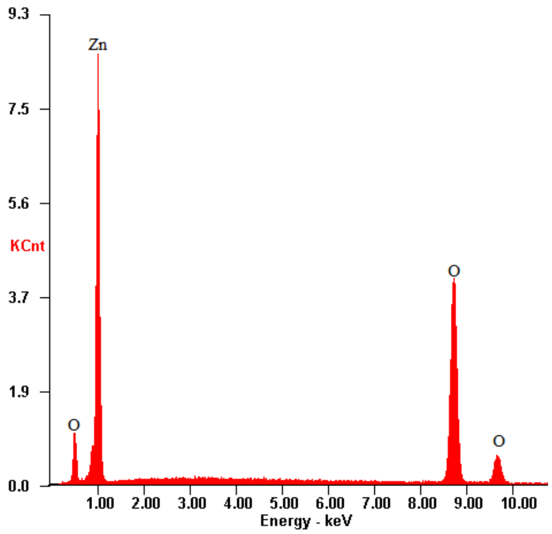

Fig. 4 EDS spectrum of ZnO Nanoparticles

3.5 Transmission Electron Microscopy

The morphology of the ZnO nanoparticles was observed in TEM image. The following TEM image infers that the spherically agglomerated nanoparticles were formed and also observed that uneven distribution of the nanoparticles.

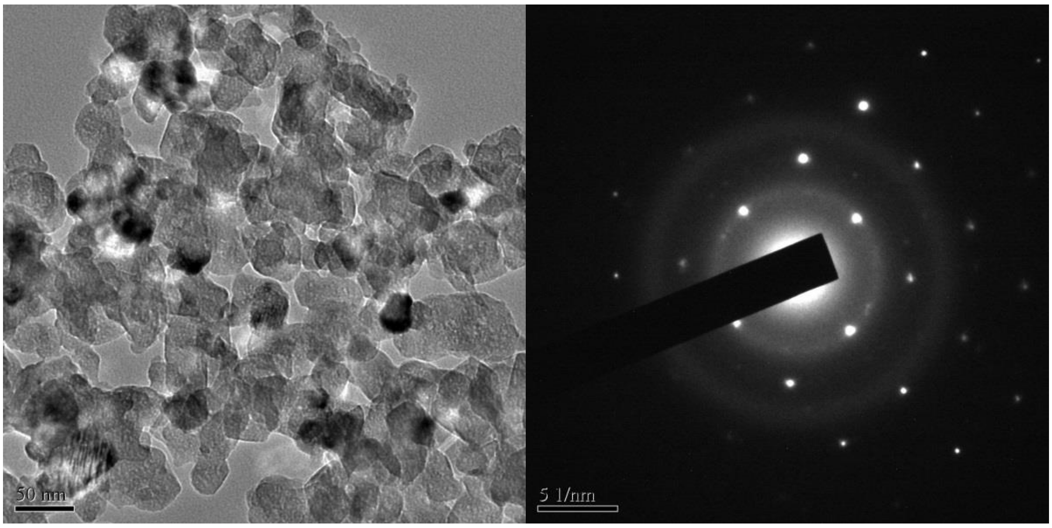

Fig. 5 TEM image and SAED pattern of ZnO Nanoparticles

The selected area electron diffraction infers that the ZnO nanoparticles prepared by Microwave-assisted synthesis using ionic liquids exhibit hexagonal structure. The SAED pattern supports the XRD crystal structure analysis.

3.6 Thermo Gravimetric & Differential Thermal Analysis

Thermal properties were studied with the assistance of TG/DTA curves. TG/DTA curves of ZnO nanoparticle is shown in below figure. The heat flow and weight loss were observed in presence of temperature.

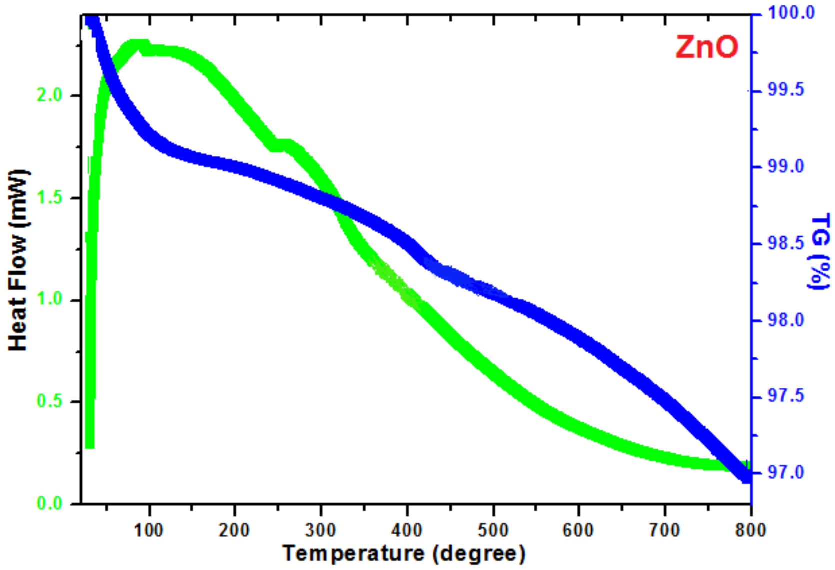

Fig. 6 TG/DTA curves of ZnO Nanoparticles

Heat flow curve infers that no phase transformation has occurred. The weight loss was observed by TG curve. From room temperature to 100°C weight loss is observed due to the evaporation of water molecules in the ZnO nanoparticles. Beyond this temperature weight loss was caused by the evaporation of unreacted organic and inorganic materials in the reaction. The total weight loss observed is 3.0%.

1. CONCLUSION

The ZnO particles were successfully synthesized by Microwave-assisted method using ionic liquids. The crystal structure and average crystallite size was measured using XRD pattern, i.e., the structure is hexagonal and average crystallite size is 21 nm. From the particle size analyzer, average particle size is 29 nm (this value is nearly equal to XRD size). The spherically agglomerated bunch like structures were observed in SEM and the EDS analysis concludes that the obtained particles were Zinc and Oxygen. The uneven distributed nanoparticles were observed in TEM and the selected area electron diffraction supports the hexagonal structure. Thermal properties conclude that no phase transformation has occurred and the weight loss observed is 3.0%. From the above information attained, ZnO nanoparticles were in the nano range.

ACKNOWLEDGEMENTS

My special thanks to **University Grants Commission** for providing the financial support.

REFERENCES

[1] Anne Aimable, Tomasz Strachowski, Ewelina Wolska, Witold Lojkowski and Paul Bowen, Comparison of two innovative precipitation systems for ZnO and Al-doped ZnO nanoparticle synthesis, Processing and Application of Ceramics. 4(3) 107 (2010).

[2] Askarinejad, Azadeh, Alavi, Mohammad Amin, Morsali and Ali, Sonochemically Assisted Synthesis of ZnO Nanoparticles: A Novel Direct Method, Iran. J. Chem. Chem. Eng. 30(3), 75 (2011).

[3] Mohd Farhan Khan, Hameedullah M., Akhter H. Ansari, Ejaz Ahmad, Lohani MB., Rizwan Hasan Khan, Mezbaul Alam M., Wasi Khan, Fohad Mabood Husain and Iqbal Ahmad, Flower-shaped ZnO nanoparticles synthesized by a novel approach at near-room temperatures with antibacterial and antifungal properties, Int. J. Nanomedicine. 9, 853 (2014).

[4] Prabhu Y.T., Siva Kumari B., Venkateswara Rao K., Kavitha V. and Aruna Padmavathi D, Surfactant Assisted Synthesis of ZnO Nanoparticles, Characterization and its Antimicrobial Activity against Staphylococcus aureus and Escherichia coli, Int. J. current Eng. and Technol. 4(2), 1038 (2014).

[5] Suhendro Purbo Prakoso and Rosari Saleh, Hydrogen Incorporation in Undoped ZnO Nanoparticles, World J. Condensed Matter Phys. 1, 130 (2011).

[6] Ruoyu Hong, Tingting Pan, Jianzhong Qian and Hongzhong Li, Synthesis and surface modification of ZnO nanoparticles, Chem. Eng. J. 119 (2-3), 71 (2006).

[7] Rameshwar Rao, Rajendar V and Venkateswara Rao K, Structural and Optical Properties of ZnO Nano Particles Synthesised by Mixture of Fuel Approach in Solution Chemical Combustion, Adv. Mater. Res. 629, 273 (2013).

[8] Amir Kajbafvala, Joshua P. Samberg, Hamed Ghorbani, Ehsan Kajbafvala and S.K. Sadrnezhaad, Effects of initial precursor and microwave irradiation on step-by-step synthesis of zinc oxide nano-architectures, Mater. Lett. 67, 342 (2012).

[9] Martyn J. Earle and Kenneth R. Seddon, Ionic liquids. Green solvents for the future, Pure Appl. Chem. 72(7), 1391 (2000).

[10] Firouz Matloubi Moghaddam and Hamdollah Saeidian, Controlled microwave-assisted synthesis of ZnO nanopowder and its catalytic activity for O-acylation of alcohol and phenol, Mater. Sci. and Eng. B. 139(2–3) 265 (2007).

[11] Ashok CH and Venkateswara Rao K, ZnO/TiO_2 nanocomposite rods synthesized by microwave-assisted method for humidity sensor application, Superlattices and Microstructures. 76, 46 (2014).

[12] Khorsand Zak A., Razali R., Abd Majid WH. and Majid Darroudi, Synthesis and characterization of a narrow size distribution of zinc oxide nanoparticles, Int. J. Nanomedicine. 6, 1399 (2011).

[13] Faheem Ahmed, Shalendra Kumar, Nishat Arshi, Anwar M.S. and Ram Prakash, Growth and characterization of ZnO nanorods by microwave-assisted route: green chemistry approach, Adv. Mat. Lett. 2(3) 183 (2011).

[14] Mohammad Ali Moghri Moazzen, Seyed Majid Borghei and Farshad Taleshi, Change in the morphology of ZnO nanoparticles upon changing, the reactant concentration, Appl. Nanosci. 3, 295 (2013).

[15] Ming-Guo Ma, Ying-Jie Zhu, Guo-Feng Cheng and Yue-Hong Huang, Microwave synthesis and characterization of ZnO with various Morphologies, Mater. Lett. 62(3), 507 (2008).

[16] Yendrapati Taraka Prabhu, Kalagadda Venkateswara Rao, Vemula Sesha Sai Kumar and Bandla Siva Kumari, Synthesis of ZnO Nanoparticles by a Novel Surfactant Assisted Amine Combustion Method, Adv. in Nanoparticles. 2, 45 (2013).

AUTHOR'S BIOGRAPHY

Mr. Ch. Ashok is a Research (Ph. D.) Scholar in Centre for Nano Science and Technology, Institute of Science and Technology, Jawaharlal Nehru Technological University Hyderabad, Telangana, India. He published five international peer-reviewed journals and one text book. He completed his M.Sc. (Physics) from Kakatiya University and M.Tech. (Nanotechnology) from JNTU Hyderabad. His research interest is on Synthesis and Characterization of Metal oxide Nanostructured materials for Sensor Applications.

DR. K. Venkateswara Rao is Associate Professor of Nanotechnology in CNST, IST and Additional Controller of Examinations-III JNTUH, Telangana, India. He is DST-Programme Coordinator, Board of studies member in many Universities. He published 82 international peer reviewed journals and published 14 books in the area of Nanotechnology and Physics. He is presently guiding 8 Ph.D. students. Under his guidance more than 50 M.Tech., M.Sc. and B.Tech. Project students submitted their dissertations. He is working in the area of Nanomaterials Synthesis, Characterization and Fabrication of thin films for various Applications.

Mr. V. Rajendar is a Research (Ph. D.) Scholar in Centre for Nano Science and Technology, Institute of Science and Technology, Jawaharlal Nehru Technological University Hyderabad, Telangana, India. He published eight international peer-reviewed journals. He completed his M.Sc. (Physics) from Acharya Nagarjuna University, M.Phil (Physics) from Vinayaka University. His research interest is on Synthesis and Characterization of Nanostructured materials for Diluted Magnetic Semiconductor Applications.

Ms. CH. Shilpa Chakra is Assistant Professor of Nanotechnology and Head of the department, Centre for Nano Science and Technology, IST, Jawaharlal Nehru Technological University Hyderabad, Telangana, India. She completed B.Tech. (Biotechnology) and M.Tech. (Nanotechnology) from JNTUH. She published 24 international peer reviewed journals and published 3 books in the area of Nanotechnology. Presently under her guidance more than 20 M.Tech., M.Sc. and B.Tech. Students are working in the same field. Her area of interest is Nanomaterials synthesis, Characterization and Applications from green methods.

Mr. K Ganapathi Rao is a Research student and currently he is pursuing his M.Tech. (Nanotechnology) in Centre for Nano Science and Technology, Institute of Science and Technology, Jawaharlal Nehru Technological University Hyderabad, Telangana, India. He completed his Bachelor's degree in Electronics and Communication Engineering from JNTUH, Telangana, India. His area of interest is on Synthesis and Characterization of Metal oxide Nanomaterials for Self-cleaning Applications