# Spherical Shaped ZnO Nanoparticles Synthesized using Chemical Route Assisted by Microwave Irradiation

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#### ABSTRACT

The ZnO nanoparticles have been synthesized using a simple chemical route assisted by microwave irradiation. The synthesized ZnO nanomaterials particle size, functional group and morphology were characterized by means of X-ray diffraction analysis (XRD), Fourier transform infrared spectroscopy (FTIR) and scanning electron microcopy (SEM). The average crystalline size was calculated and found to be 40-42 nm. The spherical shape morphology was confirmed by SEM analysis.

Keywords: Zinc oxide nanoparticles, Structural, Morphological properties.

#### **1.INTRODUCTION**

Nanostructure ZnO materials compared with bulk size materials exhibits the possible quantum confinement effect and large surface areas, showing different electronic, optical, thermal and chemical properties. ZnO nanoparticles having a wide band gap oxide n-type semiconductor materials (3.37eV) with large exciton binding energy of (60meV) at room temperature [1-3]. In recent years ZnO nanostructure materials have found the numerous applications such as pH sensors [4], biosensors [5], gas sensors [6], UV photodiodes [7], transparent electrodes [8], and acoustic wave devices [9]. The ZnO nanomaterials synthesized by various techniques such as microemulsion, solvothermal, chemical precipitation, hydrothermal, electrospinning, and sol gel process [10-17] were widely reported.

In this research work reported the synthesized ZnO nanostructure by a microwave irradiation assisted process is simple, cheap, and fast and is characterized by a different irradiation time interval. As compared to conventional heating method, microwaves cause the uniform distribution of temperature between the surface and bulk material there by leading to the fast formation of ZnO nanoparticle. To investigate the synthesis of ZnO nanoparticle in this method, the obtained ZnO nanomaterial were characterized by various techniques such as X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM).

#### 2. EXPERIMENTAL PROCEDURE

Zinc Chloride and ammonia solution were supplied from (Merck, 98%) Mumbai, India. All chemicals were of analytical grade and used as received without further purification. The synthesis of ZnO nanoparticles by the microwave irradiation method was carried out as follows. First, a 0.1M of zinc hydroxyl solution was prepared by dissolving zinc chloride in deionized water. Then the pH of the solution was maintained at 8 by adding ammonia solution dropwise. The resulting product was filtered and washed with deionized water and ethanol until becomes free from chlorine impurities. The precipitate was irradiated for 5 minutes and 30 minutes in household microwave (radiation frequency 2.45GHZ, power up to 1KW) with convection mode, finally giving a white product of ZnO nanoparticles.

## **3. RESULT AND DISCUSSION**

## 3.1 X-ray diffraction analysis (XRD)

The microstructure of spherical shape ZnO nanostructure has been characterized by XRD as shown in fig .1. All the diffraction peaks are indexed as orthorhombic phase with lattice parameters a=4.905Å, b=5.143Å and c=8.473Å, which are confirm from the standard card (JCPDS card no-89-0138). The average crystalline size was calculated by Scherrer's formula,  $d=K\lambda/\beta\cos\theta$ , where d is the mean crystalline size, K is a grain shape dependent constant (0.9),  $\lambda$  is the wavelength of the incident beam,  $\theta$  is a Bragg reflection angle, and  $\beta$  is the full width at half maximum (FWHM) of the mean diffraction peak. The calculated average particles size of ZnO nanoparticles around 42-41 nm.



Figure 1. XRD patterns of microwave synthesized samples: (a) sample A, (b) sample B.

# **3.2 Fourier Transform Infrared Spectroscopy (FTIR)**

Figure 2 shows the FTIR spectrum of ZnO nanoparticles. The sharp peaks at 606 and 611cm<sup>-1</sup> corresponding to the stretching mode of Zn-OH bond it is due the presence of zinc hydroxide. The small peak at 439 cm<sup>-1</sup> corresponding to the stretching mode of Zn-O bond it is indicate the presence of zinc oxide. The recorded values are tabulated in table 1.

International Journal of Science and Engineering Applications Special Issue NWLM ISSN-2319-7560 (Online)



Figure 2. FTIR spectrum of microwave synthesized samples: (a) sample A, (b) sample B.

Table 1. FTIR	peaks assignments of microwave	e synthesized samples: (	a) sample A, (b) sample B
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Wavenumber(cm <sup>-1</sup> )		Pook assignments
Sample A	Sample B	_ reak assignments
3198	3204	O-H stretching
1644	1644	C=O stretching
1427	1434	C=C stretching
1063	1067	C-O stretching
606	611	Zn-OH stretching
439	439	Zn-O stretching

### 3.3 scanning electron microscopy (SEM)

SEM images of ZnO nanoparticles are reported in fig 3. SEM images of ZnO nanoparticles were irradiated at 5 minutes and 30 minutes are shown in fig 3. The morphology of 5 minutes irradiated ZnO show the agglomeration of small crystallites and attributed at uncontrolled coagulation during precipitation, at higher irradiation time interval, small crystallites are form a large agglomerated spherical shaped structure.



Figure 3. SEM images of microwave synthesized samples: (a) sample A, (b) sample B.

#### **4. CONCLUSION**

Spherical shaped ZnO nanoparticles were successfully synthesized by microwave irradiation method. XRD patterns confirm the orthorhombic phase structure with average crystalline size 40-42 nm. FTIR spectrum confirms the presence of zinc oxide network. A SEM image shows the formation of spherical shape agglomerated structure. In summary, microwave irradiation method is a cheap and fast method for production of ZnO nanoparticles.

#### **5. REFERENCE**

- S. Hingorani, V. Pillai, P. Kumar, M.S. Multani, D.O. Shah, Microemulsion mediated synthesis of zinc-oxide nanoparticles for varistor studies, Materials Research Bulletin 28 (1993) 1303–1310.
- [2] S. Sakohara, M. Ishida, M.A. Anderson, Visible luminescence and surface properties of nanosized ZnO collids prepared by hydrolyzing zinc acetate, Journal of Physical Chemistry B 102 (1998) 10169–10175.
- [3] X. Zhao, S.C. Zhang, C. Li, B. Zheng, H. Gu, Application of zinc oxide nanopowder for two-dimensional micro-gas sensor array, Journal of Materials Synthesis and Processing 5 (1997) 227.
- [4] Asif, Nur, Willander, and Danielsson, "Selective calcium ion detection with functionalized ZnO nanorods-extended gate MOSFET," Biosensors and Bioelectronics, vol. 24, no. 11, pp. 3379–3382, 2009.
- [5] C. Xia, N. Wang, L. Lidong, and G. Lin, "Synthesis and characterization of waxberry-like microstructures ZnO for biosensors," Sensors and Actuators B, vol. 129, no. 1, pp. 268–273, 2008.
- [6] B. B. Rao, "Zinc oxide ceramic semi-conductor gas sensor for ethanol vapour," Materials Chemistry and Physics, vol. 64, no. 1, pp. 62–65, 2000.
- [7] Liu, Zhang, Lu et al., "Fabrication and characterization of ZnO film based UV photodetector," Journal of Materials Science, vol. 20, no. 3, pp. 197–201, 2009.
- [8] W. H. G. Horsthuis, "ZnO processing for integrated optic sensors," Thin Solid Films, vol. 137, no. 2, pp. 185–192, 1986.

- Krishnamoorthy and Iliadis, "Development of high frequency ZnO/SiO2/Si Love mode surface acoustic wave devices," Solid- State Electronics, vol. 50, no. 6, pp. 1113–1118, 2006.
- [10] H. Zhang, D. Yang, T.S. Li, X. Ma, Y. Ji, J. Xu, D. Que, Synthesis of needle like nickel nanoparticles in water-in-oil microemulsion, Materials Letters 59 (2005) 1696–1700.
- [11] D. Mandelaers, G. Vanhoyland, H. Van den Rul, J.D. Hean, M.K. VanBeal, J. Mullens, L.C. Van Poucke, Synthesis of ZnO nanopowder via an aqueous acetate–citrate gelation method, Materials Research Bulletin 37 (2002) 901–914.
- [12] N. Uekawa, S. Iahii, T. Kojima, K. Kakegawa, Formation of porous spherical aggregated structure of ZnO nanoparticles by low-temperature heating of Zn(OH)2 in diol solution, Materials Letters 61 (2007) 1729–1734.
- [13] J.A. Park, J. Moon, S.J. Lee, S.-C. Lim, T. Zyung, Fabrication and characterization ZnO nanofibers by electrospinning, Current Applied Physics 9 (2009) 210–212.
- [14] T. Krishnakumar, R. Jayaprakash, N. Pinna, V.N. Singh, B.R. Mehta, A.R. Phani, Microwave-assisted synthesis and characterization of flower shaped zinc oxide nanostructures, Materials Letters 63 (2009) 242–245.
- [15] N. Pinna, G. Neri, M. Antonietti, M. Niederberger, Nonaqueous synthesis of nanocrystalline semiconducting metal oxides for gas sensing, Angewandte Chemie International Edition 43 (2004) 4345–4349.
- [16] X. Gao, X. Li, W. Yu, Flowerlike ZnO nanostructures via hexamethylenete via hexamethylenetetramine-assisted thermolysis of zinc-ethylenediamine complex, Journal of Physical Chemistry B 109 (2005) 1155–1161.
- [17] L.P. Bauermann, J. Bill, F. Aldinger, Bio-friendly synthesis of ZnO nanoparticles in aqueous solution at near-neutral pH and low temperature, Journal of Physical Chemistry B 110 (2006) 5182–5185.