



Synthesis and characterization of ZnO nanoparticles by co-precipitation method at room temperature

A. Jafar Ahamed* and P. Vijaya Kumar

PG and Research Department of Chemistry, Jamal Mohamed College (Autonomous), Tiruchirappalli – 620 020, Tamil Nadu, India

ABSTRACT

ZnO nanoparticles were synthesized by co-precipitation method. The synthesized particles were characterized by X-ray diffraction (XRD), High resolution transmission electron microscopy (HRTEM), Energy dispersive X-ray spectroscopy (EDX) and UV-visible spectroscopy. The X-ray diffraction study reveals that the synthesized ZnO nanoparticles have wurtzite structure and the particle size varies from 35 to 40 nm. HRTEM investigation reveals that the surface morphology of ZnO nanoparticle is spherical in co-precipitation. The UV-Visible spectrum of the nanoparticles shows a red shift compared to that of the bulk sample.

Keywords: Nanoparticles, Zinc oxide, X-ray diffraction, co-precipitation method.

INTRODUCTION

In recent years, noble metal oxide nanoparticles have been the subject of focused research due to their unique electronic, optical, mechanical, magnetic and chemical properties

That are significantly different from those of bulk counterpart [1]. The nano zinc oxide has found wide ranging applications in various areas due to its unique and superior physical and chemical properties compared with bulk ZnO. The large specific surface area, high pore volume, nanostructured properties, low cost and low toxicity of nano ZnO [2] make it a promising candidate, particularly in catalysts [3], photocatalysis, electrostatic dissipative coating, transparent UV protection films, and chemical sensors [4-7].

Various methods such as thermal decomposition, chemical vapor deposition, sol gel, spray pyrolysis, and precipitation have been developed for the fabrication of nanosized ZnO particles with uniform morphology and size [8-10]. Among these synthetic routes, precipitation approach compared with other traditional methods provides a facile way for low cost and large-scale production, which does not need expensive raw materials and complicated equipments [11].

In the present study, we report the synthesis of ZnO nanoparticles using co-precipitation method and the characterization of ZnO nanoparticles using X-ray diffraction, transmission electron microscopy (TEM), selected area electron diffraction (SAED), UV-vis absorbance spectra is discussed.

EXPERIMENTAL SECTION

Zinc nitrate, sodium hydroxide, and ethanol were purchased and used without further purification. Zinc oxide nanoparticles were synthesized by co-precipitation method using zinc nitrate and sodium hydroxide precursors. In this experiment, a 0.1M aqueous solution of zinc nitrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) was kept under constant stirring using a magnetic stirrer to completely dissolve the zinc nitrate for one hour and 0.8M aqueous solution of sodium hydroxide (NaOH) was also prepared in the same way with stirring of one hour. After complete dissolution of zinc nitrate, 0.8M NaOH aqueous solution was added under high speed constant stirring, drop by drop (slowly for 45 min) touching the walls of the vessel. The reaction was allowed to proceed for 2 h after complete addition of sodium hydroxide. The beaker was sealed at this condition for 4 h. After the completion of the reaction, the solution was allowed to settle for overnight and further, the supernatant solution was separated carefully. The remaining solution was centrifuged for 10 min, and the precipitate was removed. Thus, precipitated ZnO NPs were cleaned three times with deionized water and ethanol to remove the byproducts which were bound with the nanoparticles and then dried in air atmosphere at about 60°C . During drying, $\text{Zn}(\text{OH})_2$ is completely converted into ZnO. The prepared ZnO nanoparticles were characterized for their optical and nanostructured properties.

Characterization techniques

Powder XRD measurements were performed using the X-ray diffractometer (Shimadzu XD-3A) in the diffraction angle ranges $20 \leq 2\theta \leq 80^\circ$, with monochromatic $\text{CuK}\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$) source. High-resolution transmission electron microscope (HRTEM, JEM 2100) was used with a high resolution pole piece operates at 200 kV accelerating voltages. It works under a vacuum of $\sim 10^{-6}$ Pa. These conditions give a lattice resolution of 0.14 nm and a point to point resolution of 0.23 nm which help the instrument to be a perfect technique for imaging materials on the atomic scale. The optical properties of the nanoparticles in solutions are studied using UV-Visible spectrophotometer (Shimadzu, UV-2450) in the wavelength range of 200–900 nm.

RESULTS AND DISCUSSION

Figure 1 represents the X-ray diffraction pattern of ZnO nanoparticles. A definite line broadening of the XRD peaks indicates that the prepared material consists of particles in nanoscale range. From this XRD patterns analysis, we determined peak intensity, position and width, full-width at half-maximum (FWHM) data. The diffraction peaks located at 31.72° , 34.36° , 36.19° , 47.50° , 56.56° , 62.77° , 67.96° , and 69.04° have been keenly indexed as a hexagonal wurtzite phase of ZnO [12, 13] with lattice constants $a = b = 0.325 \text{ nm}$ and $c = 0.521 \text{ nm}$ (JCPDS card number: 36-1451) [14], and further it also confirms the synthesized nanopowder was free of impurities as it does not contain any characteristics XRD peaks other than ZnO peaks.

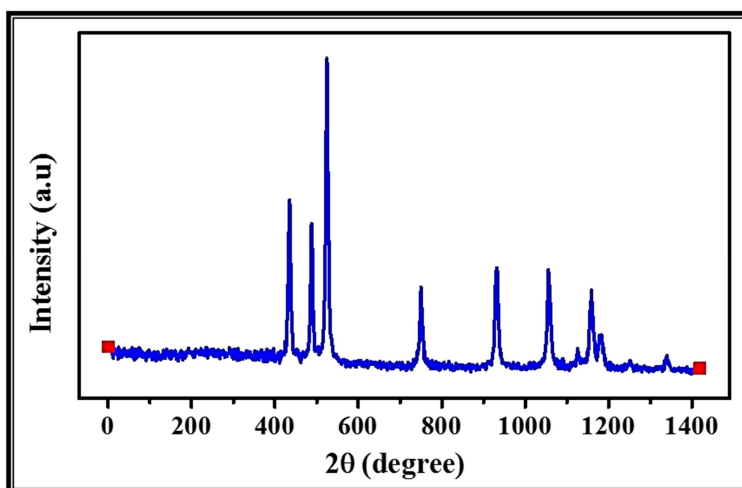


Fig.1 XRD pattern of ZnO nanoparticles

The average crystallite size of the ZnO NPs was estimated from X-ray line broadening of the diffraction peaks using

Debye Scherrer's relation.

$$\text{Average crystallite size (D)} = \frac{0.9\lambda}{\beta \cos \theta} \quad \text{--- (1)}$$

Where, λ is the wave length of X-ray used (1.54060 Å), β is the angular peak width at half maximum in radians and θ is Bragg's diffraction angle. The average crystallite size is calculated as 39 nm for ZnO NPs.

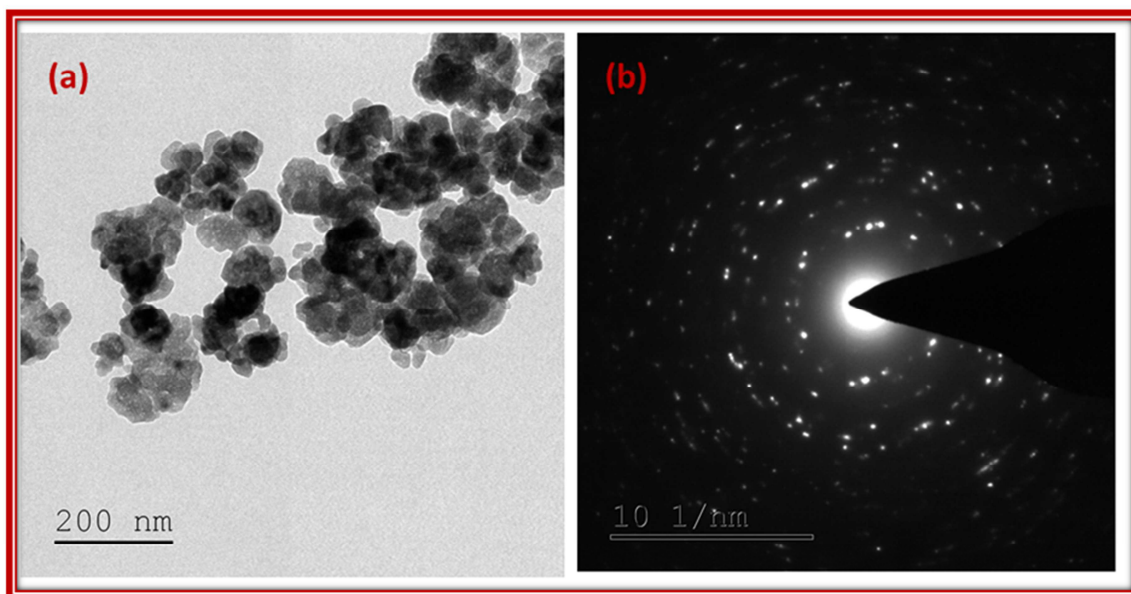


Fig.2 (a) HRTEM image of ZnO nanoparticles and Fig.2 (b) SAED pattern of ZnO nanoparticles

Figure 2 (a) shows the HRTEM image of ZnO nanoparticles. From the TEM image spherical shape ZnO nanoparticles were observed and the average size in the range of 35-40 nm which is in the better agreement with the size calculated by XRD. Figure 2 (b) shows the selected area diffraction pattern (SAED) of ZnO nanoparticles. It shows that the particles are well crystallized. The diffraction rings on SAED image matches with the peaks in the XRD pattern which also proves the hexagonal wurtzite structure of ZnO nanoparticles [15].

Figure 3 shows the EDAX spectrum of ZnO nanoparticles prepared by co-precipitation method. The strong peaks observed in the spectrum related to Zinc and oxygen. The elemental constitution of ZnO nanoparticles with two major peaks was found to have a weight percentage of 70.43 of Zinc and 39.29 of oxygen. The prepared ZnO nanoparticles have an atomic percentage of 36.82 of Zinc and 63.18 of oxygen. This study confirmed the formation of ZnO nanoparticles in the co - precipitation process.

Figure 4 shows the optical absorption of ZnO nanoparticles. It shows absorption peaks at 370 nm. Even though these spectra were taken at room temperature, the ZnO nanoparticle samples exhibit salient exciton absorption features due to the relatively large binding energy of the exciton (60 mV). As the annealing temperature is increased, the absorption peak exhibits a progressive redshift. The corresponding redshifts are consistent with the observed variation of the NPs sizes as reported earlier [16]. Since the size of ZnO nanoparticles is nearly 40 nm, that is much more than the exciton Bohr radius in ZnO ($r_B=2.34$ nm), we observe a small shift of the peak due to quantum confinement effect.

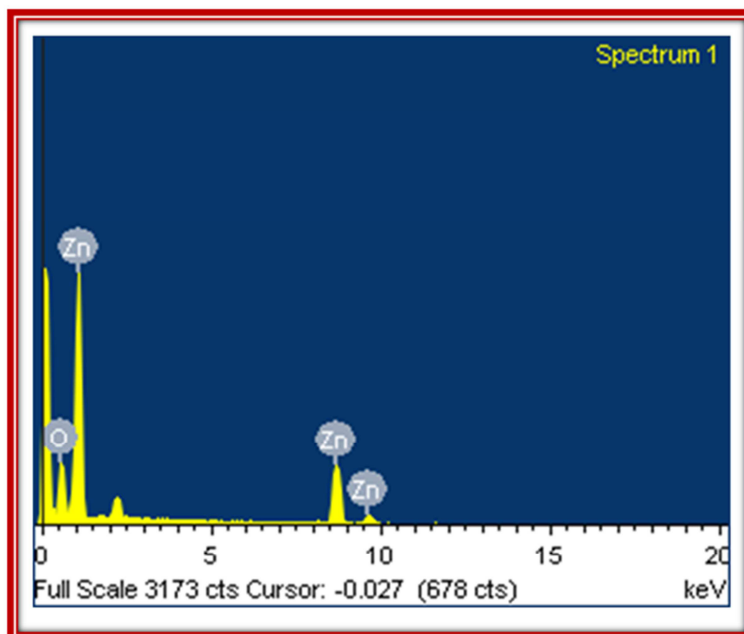


Fig.3 EDAX spectrum of ZnO nanoparticles

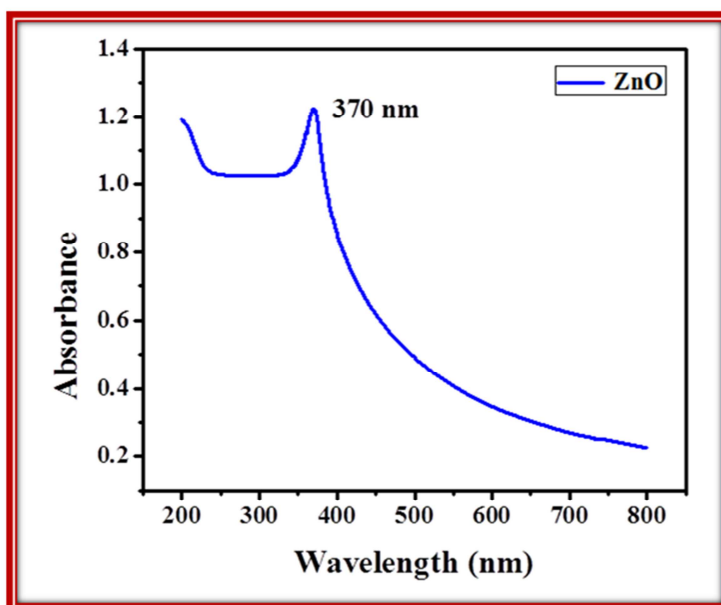


Fig.4 UV-visible absorption spectra of ZnO nanoparticles

CONCLUSION

ZnO nanoparticles were prepared via co-precipitation method. XRD results revealed that all the samples are polycrystalline and hexagonal wurtzite structure. The crystallite size of the prepared nanoparticles was determined by Debye-Scherrer's equation and it was found to be in the nanometer range 39 nm. HRTEM result shows the synthesized ZnO nanoparticles were highly spherical in shape. SAED studies confirm the synthesized ZnO nanoparticles are highly crystalline nature. The EDAX spectral data evident that the formation of ZnO nanoparticles through co-precipitation method. The UV-visible study shows red shift absorption at ~370 nm.

REFERENCES

- [1] NM Franklin; NJ Rogers; SC Apte; GE Batley; GE Gadd; PS Casey, *Environmental Science Technology*, **2007**, 41, 8487.
- [2] HJung; Choi H; *Applied Catalysis B*, **2006**, 66, 288.
- [3] Maciej Mazur; *Electrochemistry Communications*, **2004**, 6, 400-403.
- [4] ZL Wang; J Song, *Science*, **2006**, 312(5771), 242-246.
- [5] DC Look, *Materials Science and Engineering B*, **2001**, 80(1-3), 383-387.
- [6] M. Kitano; M. Shiojiri, *Powder Technology*, **1997**, 93(3), 267-273.
- [7] IO. Sosa; C Noguez; RG Barrera, *The Journal of Physical Chemistry B*, **2003**, 107(26), 6269-6275.
- [8] Y Yang; H Chen; B Zhao; X Bao; *Journal of Crystal Growth*, **2004**, 263(1-4), 447-453.
- [9] JH Lee; KH Ko; BO Park; *Journal of Crystal Growth*, **2003**, 24, 7(1-2), 119-125.
- [10] ZM Dang; LZ Fan; SJ Zhao; CW Nan, *Materials Science and Engineering B*, **2003**, 99(1-3), 386-389.
- [11] C Wang; WX Zhang; XF Qian; XM Zhang; Y Xie; YT Qian; *Materials Letters*, **1999**, 40(6), 255-258.
- [12] J Zhou; F Zhao; Y Wang; Y Zhang; L. Yang; *Journal of Luminescence*, **2007**, 122-123(1-2), 195-197.
- [13] ZMKhoshhesab; MSarfaraz; MA Asadabad; *Metal-Organic and Nano-Metal Chemistry*, **2011**, 41(7), 814-819.
- [14] JCPDS, Powder Diffraction File, Alphabetical Index, Inorganic Compounds, International Centre for Diffraction Data, Newtown Square, Pa, USA, **1977**.
- [15] GVoicu; OOprea; BS Vasile; EAndronescu; *Digest Journal of Nanomaterials and Biostructures*, **2013**, 8, 667-675.
- [16] Lirimpan; VPN Nampoore; P Radhakrishnan; A Deepthy; B Krishnan; *Journal of Applied Physics*, **2007**, 102, 063524-063529.