SYNTHESIS AND CHARACTERIZATION OF POLYMER CAPPED METALOXIDE NANOPARTICLES

(1) Prasaant kumar D- Assistanat professor,

(2) Palepu Rajendar-Assistant professor

Department of physics, Siddhartha Institute of Technology & Sciences

Narapally, Ghatkesar Hyderabad, Telangana 500088

Abstract: *Multi-composite nanomaterials with wide range of compositions and tunable morphology displays multiple functions and novel properties that makes them good candidates for applications in biological detection and sensing, drug delivery, magnetic data storage photoelectric conversions etc. In the present work, the synthesis and characterization of polymer capped zinc oxide nanoparticles. Cellulose is acted as polymer. The synthesized nanoparticles were characterized by X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Analysis (EDAX), Fourier Transform Infrared Spectroscopy (FTIR), Atomic Force Microscopy (AFM) and Transmission Electron Microscopy (TEM). The morphological and elemental compositions were analyzed using FESEM with EDX. The XRD results revealed that the zinc oxide nanoparticles are highly crystalline; It can be clearly observed that the diffraction peaks appear in the pattern corresponding to the wurtzite hexagonal structure. The nanocomposites size can be calculated from Debye-Scherrer's formula.*

Keywords: Zinc oxide, Cellulose, Nanoparticles, SEM, EDAX and XRD.

1. INTRODUCTION

In recent years, nanoscience and technology have potential applications in the field of science and technology. Particularly nanocrystalline materials are treated as the suitable material for those applications. Intensive investigations were carried out for most of the applications of these new classes of materials [1]. Among the various nanocrystalline materials, ZnO with particle size in the range of several nm are treated as exclusively suitable material for various applications because of their unique properties. Zinc oxide (ZnO) is an n-type inorganic semiconductor which crystallizes in hexagonal wurtzite structure, has bulk direct band gap at about 3.37 eV [2], and exciton binding energy of 60 meV. Due to those unique natures, particularly at the nano-scale, ZnO have potential applications in optoelectronic devices such as solar cells optical wave guide, light emitting diodes (LED), etc. The main obstacle in any applications of ZnO NPs (event any NPs) is a tendency of NPs suspension to agglomerate. To resolved this problem one used to modified the surface of ZnO and others NPs by capping agent [3-5]. Many types of capping agents have been considered to serve as a stabilizer [6,7]. The stabilizer could change the size, shape and morphology [8,9] of the particles consequently affect the optical and electrical properties due to change in the band gap of NPs [10,11]. In this paper we have reported the cellulose capped metal oxide nanoparticles. Cellulose, as one of the most abundant biopolymers, has many merits like biodegradability, biocompatibility and low cost.

The advantages of capping nanoparticles by polymer molecules were the following: (1) the passivation of surface defects, which decreased the nonradiative recombination center, such as dangling bonds, and increased fluorescence intensity [12-14]; (2) the aggregation inhibition of nanoparticles by a steric effect; 3) the utilization of nanoparticles for a drug delivery system (DDS) by the improvement in

immunoaffinity; and 4) the probability of surface functionalization of nanoparticle, such as targeting. The main objective of the research work is to synthesis and characterization of polymer capped zinc oxide nanoparticles.

2. MATERIALS AND METHODS

2.1. SYNTHESIS OF POLYMER CAPPED ZINC OXIDE (ZnO) NANOPARTICLES

The capping agent cellulose is dissolved in 100 ml of distilled water and stirred at 90ºC for one hour and then cooled to room temperature. 2.8754 g of $ZnSO_4.7H_2O$ (0.1M) was dissolved in 100 mL of distilled water. Meanwhile, Cellulose solution was prepared and was added to $ZnSO_4.7H_2O$ (0.1M) solution. Then 1.6802 g of $NaHCO_3$ (0.2 M) was prepared separately and was added drop wise into the above solution under constant stirring. After being stirred at room temperature for 2-3 hours, the as formed precipitates were filtered, washed with distilled water and ethanol and finally dried in hot air oven at 80ºC to get cellulose capped zinc oxide nanoparticles [1].

The biopolymer capped ZnO nanoparticles were characterized by Fourier Transform Infrared Spectroscopy (FTIR) to determine composition and molecular structure, the morphologies were investigated by Scanning Electron Microscope and the crystallographic structure of the sample was examined using X-ray diffraction (XRD).

3. RESULTS AND DISCUSSION:

The XRD of the synthesized cellulose capped zinc oxide shows broad peaks at values of 31.9 (100), 34.5 (002), 36.3 (101), 47.5 (102), 56.7 (110), and 62.9 (103) which are typical for the zinc oxide structure. All of the indexed peaks in the obtained spectrum are well matched with that of bulk zinc oxide (JCPDS code no. 36-1451), which confirms that the synthesized nanoparticles have wurtzite hexagonal structure with high purity. Notable line broadening of the diffraction peaks is an indication that the synthesized materials are in nanometer range. The average particle size has been determined from full width at half maximum (FWHM) of the diffraction peaks using Scherrer's equation. The particle size of zinc oxide nanoparticles has been found to be 40.2 nm.

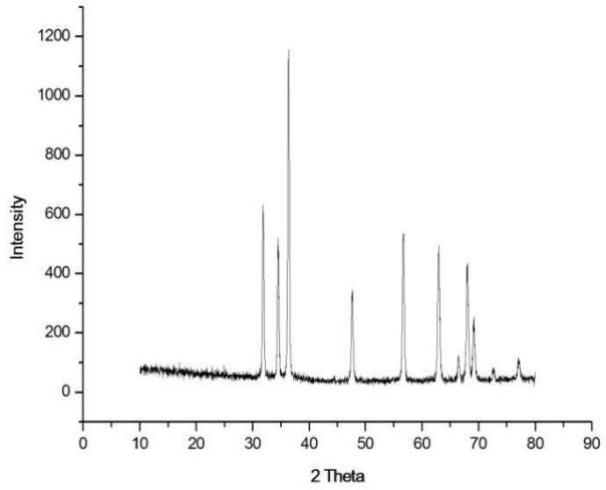

Fig. 1. XRD Spectrum of Cellulose capped ZnO nanoparticles

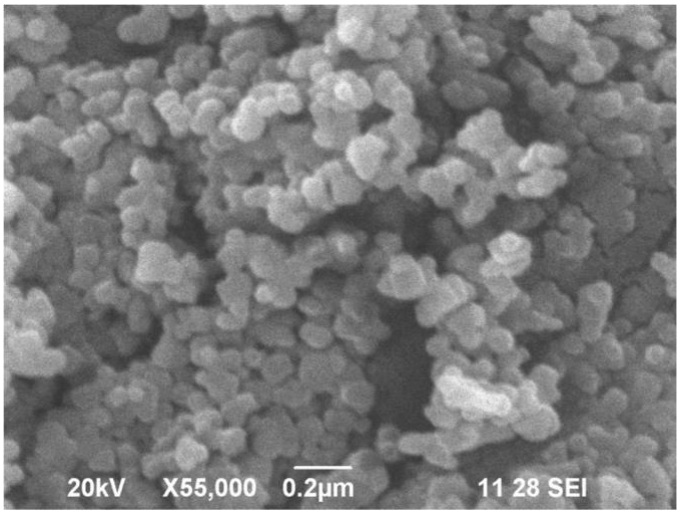

Fig. 2.a. SEM image of Cellulose capped ZnO nanoparticles

The surface morphology of the synthesized Cellulose capped zinc oxide nanoparticles was identified by Scanning Electron Microscope. This material can be clearly seen that the cellulose capped ZnO nanoparticles are almost uniformly dispersed in the polymer matrix (Fig.2.a). However, ZnO aggregates were also observed at some location due to its hydrophilic nature [15] and higher specific surface area. The EDAX spectra of polymer capped zinc oxide nanoparticles are presented in Fig.2.b. The peaks corresponding to Zn and O are clearly observed in the EDX spectrum at their normal energy and the results are clearly indicating the formation of zinc oxide nanoparticles.

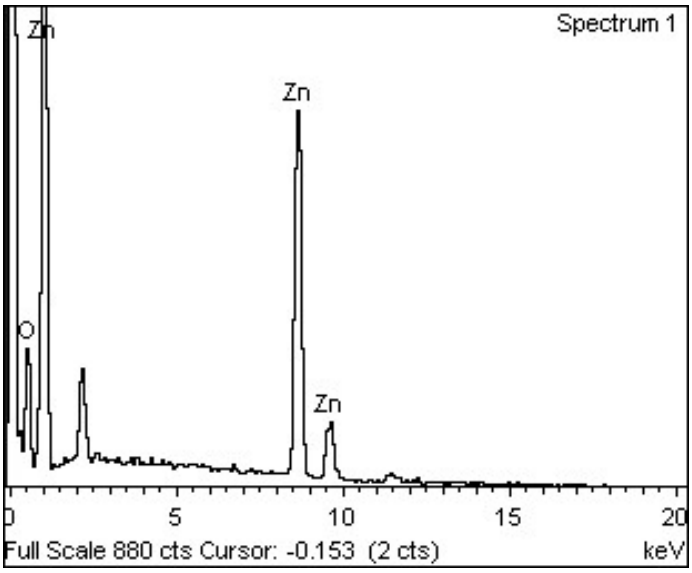

Fig. 2.b. EDAX image of Cellulose capped ZnO nanoparticles

The FTIR of the cellulose capped ZnO nanoparticles is shown in Fig. 3. The observed main peaks below 500 cm^{-1} corresponds to Zn-O confirms the formation of polymer capped zinc oxide nanoparticles. The peaks are observed at 1380 cm^{-1}, 1097 cm^{-1} and 1032 cm^{-1} corresponds to the symmetric stretching of C-O bond and the bond observed at 2360cm^{-1} is due to CO_2 molecule in air. The characteristic band at 3450 cm^{-1} corresponds to the stretching vibration of O-H groups.

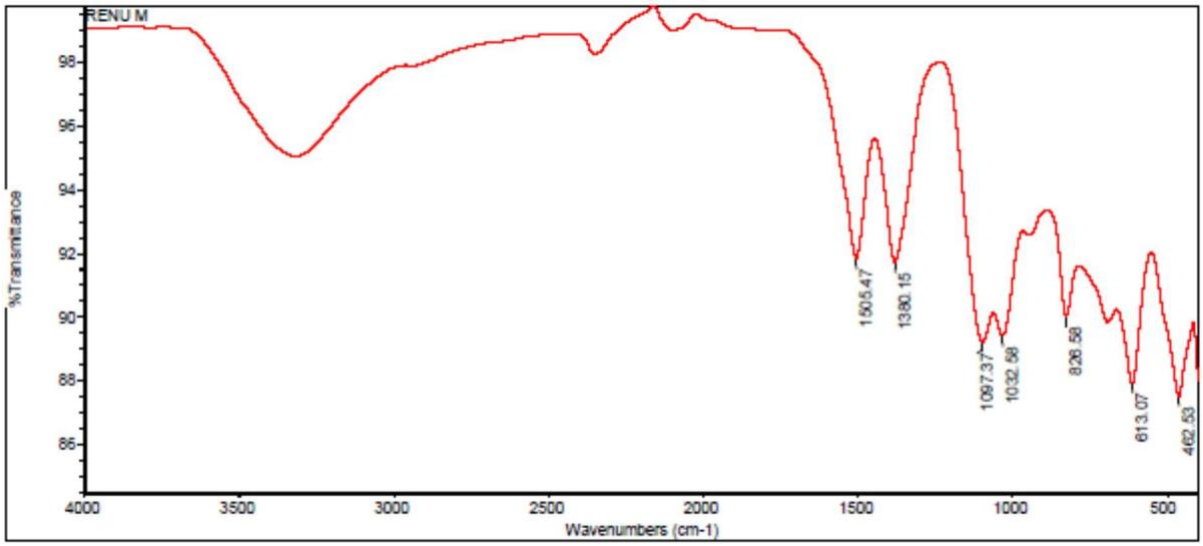

Fig. 3. FTIR image of Cellulose capped ZnO nanoparticles

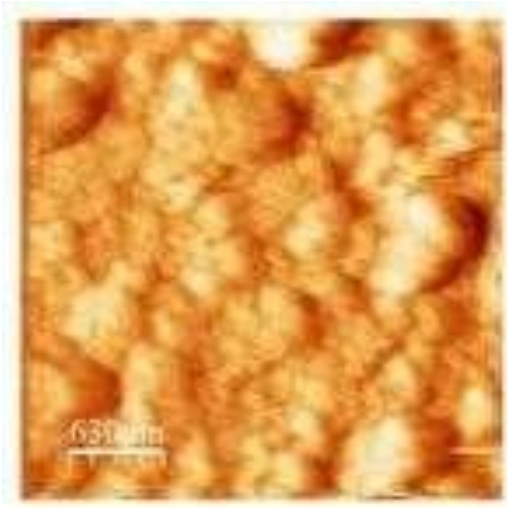

Fig.4. AFM image of Cellulose capped ZnO nanoparticles

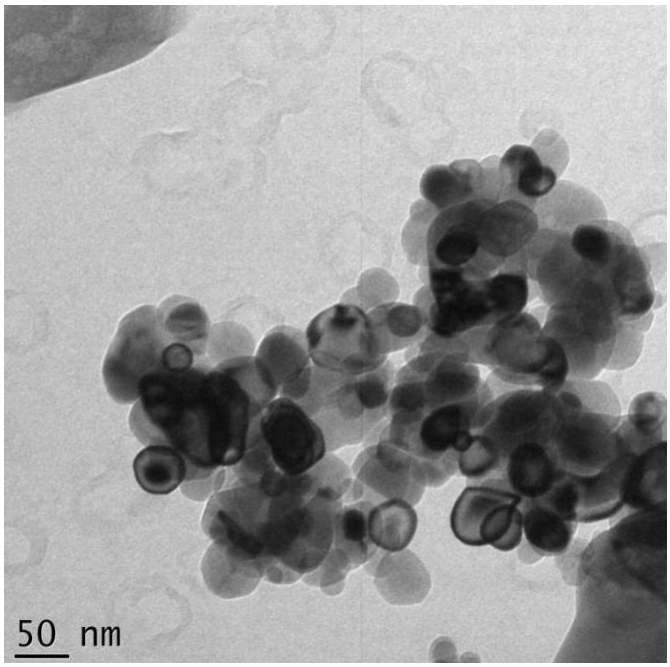

Fig.5. TEM image of Cellulose capped ZnO nanoparticles

AFM technique is used to study the morphology of cellulose capped zinc oxide nanoparticles. AFM image is showed the spherical morphology for the polymer capped metal oxide nanoparticles (fig.4). The surface roughness and RMS value were determined by AFM analysis. Particle size is determined using the transmission electron microscopy. The TEM image of the cellulose capped zinc oxide nanoparticles showed spherical shaped metal oxide nanoparticles are distributed within the size range of 30-50 nm and is shows in fig.5.The TEM image of the cellulose capped zinc oxide nanoparticles confirm the formation of metal oxide nanoparticles.

4. CONCLUSION

Polymer capped Zinc oxide nanoparticles were successfully prepared using cellulose as the capping agent and the prepared nanomaterial was characterized using X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM) with Energy Dispersive X-ray Analysis (EDAX), and Fourier Transform Infrared Spectroscopy (FTIR). According to the XRD characterization the average particle size has been determined using Scherrer's equation. The average particle size of zinc oxide nanoparticles has been found to be 40.2 nm and that's good enough for the preparation of polymer capped nanoparticles. This material can be clearly seen that the cellulose capped ZnO nanoparticles are almost uniformly dispersed in the polymer matrix, which proves the role of polymer capping in the size and morphology of zinc oxide nanoparticles and the peaks corresponding to Zn and O are clearly observed in the EDX spectrum at their normal energy and the results are clearly indicating the formation of zinc oxide nanoparticles. The vibrational modes of zinc oxide nanoparticles were studied from FTIR analysis. The synthesized zinc oxide nanoparticle is a promising candidate for the applications in various fields especially in water purification. The TEM image of the cellulose capped zinc oxide nanoparticles showed spherical shaped metal oxide nanoparticles are distributed within the size range of 30-50 nm

References

1. *T. Anantha kumar, S. Malathi, C.V. Mythili and M. Jeyachandran, "Structural, Morphological and Optical Properties of Zinc Oxide Nanoparticles by Polymer Capping", International Journal of ChemTech Research, Vol.11 No.08, (2018), pp. 48-57.*
2. *L. Spanhel and M. A. Anderson, "Semiconductor clusters in the sol-gel process: quantized aggregation, gelation, and crystal growth in concentrated zinc oxide colloids", J. Am. Chem. Soc. Vol. 113, no. 8, (1991), pp. 2826–2833.*
3. *T. K. Kundu, N. Karak, P. Barik and S. Saha, "Optical Properties of Zno Nanoparticles Prepared by Chemical Method Using Poly (VinylAlcohol) (PVA) as Capping Agent", Int. J. Soft Comp. Eng. (IJSCE) vol. 1, (2011), pp. 19-24.*
4. *M. Thirumavalavan, K. L. Huang and J. F. Lee, "Preparation and Morphology Studies of Nano Zinc Oxide Obtained Using Native and Modified Chitosans", Materials, vol. 6, no. 9, (2013), pp. 4198-4212.*
5. *Y. L. Wu, A. I. Y. Tok, F. Y. C. Boey, X. T. Zeng and X. H. Zhang, "Surface modification of ZnO nanocrystals", Appl. Surf. Sci. vol. 253, no. 12, (2007), pp. 5473–5479.*
6. *B. Bochev, G. Yordanov, H. Yoshimura and C. Dushkin, "Synthesis of ZnO nanocrystals in the presence of different additives," in Nanoscience and Nanotechnology, Proceedings of National Conference, Bulgaria,Sofia, (2011), pp. 25–26.*
7. *I M. Joni, A. Purwanto, F. Iskandar and K. Okuyama, "Dispersion Stability Enhancement of Titania Nanoparticles in Organic Solvent Using a Bead Mill Process", Ind. Eng. Chem. Res. vol. 48,no. 15, (2009), pp. 6916–6922.*
8. *M. Kaur, K. L. Singh and P. Kumar, "Effect of PVA capping on the optical and structural properties of hydrothermally synthesized ZnS nanocrystals", Int. J. IT Eng. Appl. Sci. Res. vol. 2, no. 1,(2013), pp. 20–24.*
9. *I M. Joni, R. Balgis, T. Ogi, T. Iwaki and K. Okuyama, "Surface Functionalization for Dispersing and Stabilizing Hexagonal Boron Nitride Nanoparticle by Bead Milling", Colloids and Surfaces A: Physicochem. Eng. Aspects, vol. 388, no. 1-3, (2011), pp. 49– 58.*
10. *I M. Joni, T. Ogi, A. Purwanto, K. Okuyama, T. Saitoh and K. Takeuchi, "Decolorization of beads-milled TiO_2 nanoparticles suspension in an organic solvent", Adv. Powder Technol. vol. 23,no. 1,(2012), pp. 55–63.*
11. *N. Faleni and M. J. Moloto, "Effect of glucose as stabilizer of ZnO and CdO nanoparticles on the morphology and optical properties", Int. j. recent res. appl. Stud, vol. 14, no. 1,(2013), pp. 127–135.*
12. *J.I. Pankove, "Optical Processes in Semiconductors", Dover Publications: New York, NY, USA, 1971.*
13. *O.I. Micic, J.R. Sprague, Z. Lu and A. Nozik, "Highly efficient band-edge emission from InP quantum dots", Appl. Phys. Lett, vol. 68, no. 22, (1996), pp. 3150-3152.*
14. *M. Kuno, J.K. Lee, B.O. Dabbousi, F.V. Mikulec and M.G. Bawendi, "The band edge luminescence of surface modified CdSenanocrystallites: Probing the luminescing state", J. Chem. Phys, vol. 106, no. 23, (1997), pp.9869-9882.*
15. *A. Abdolmaleki, S. Mallakpour and S. Borandeh, "Preparation, characterization and surface morphology of novel optically active poly(ester-imide)/functionalized ZnO bionanocomposites via ultrasonication assisted Process", Applied surface science, vol. 257, no. 15, (2011), pp. 6733-6735.*