Est. 1984

ORIENTAL JOURNAL OF CHEMISTRY
An International Open Access, Peer Reviewed Research Journal

www.orientjchem.org

ISSN: 0970-020 X
CODEN: OJCHEG
2021, Vol. 37, No.(6):
Pg. 1447-1451

Synthesis and Characterization of Zincoxide Nanoparticles of Average Diameter 10nm in Aqueous Medium

ARPITA BISWAS

Department of Chemistry, Shibpur Dinobundhoo Institution (College), 412/1, G. T. Road (South), Howrah-711102, West Bengal, India.
*Corresponding author E-mail: abiswas83@gmail.com

http://dx.doi.org/10.13005/ojc/370625

(Received: November 15, 2021; Accepted: December 16, 2021)

ABSTRACT

Synthesis of Zincoxide nanoparticles (ZnO NPs) with definite size and shape and their morphological characterization rationally is really a challenging aspect at present due to the ongoing demand of these nanosize particles for their divergent use in different field of science and technology. Reduction of Zinc acetate dihydrate by sodium hydroxide was performed to produce ZnO NPs by following precipitation method. Here the whole reaction was completed in aqueous medium in low temperature. To characterize the synthesized ZnO NPs some recent techniques like X-ray diffraction study (XRD), Ultra-violet Visible (UV-Vis) spectroscopy, Field emission scanning electron microscopy (FESEM), Transmission electron microscopy (TEM), Selected area electron diffraction (SAED) and Electron diffraction X-ray (EDX) were used systematically.

Keywords: ZnO NPs, Aqueous medium, XRD, SEM, TEM.

INTRODUCTION

It is observed that the last two decades there is an explosive growth in the field of research work of nanotechnology and nanoscience of which transition metal paid an attractive importance. Zinc is one of the transitional metals and ZnO is one of the transition metal oxides which shows high activity in diverse ground when we get it in its nano dimension range. In this nano range ZnO NPs occupy some physical and chemical properties which is absent in bulk ZnO.[1] The human beings are getting benefit by using these ZnO NPs as in food additives, food packing, UV-inhibiting sun screen creams, cosmetics, solar cells, organic light emitting-diodes(OLEDs), chemical sensor, photo catalytic applications, antibacterial applications, biocompatible applications, biomedical applications, textiles, removal of organic dyes, acrylic paints and agriculture etc.[2-14] There are several synthetic methods like mechanochemical process, physical vapour synthesis, thermal decomposition, precipitation and hydrothermal to synthesize ZnO NPs.[15-19] Geometrical shapes of ZnO NPs produced by precipitation process will be different as rod-like, flower-like, flake-like, needle like and spherical etc.[20-24]

In this present article spherical ZnO NPs were prepared of average diameter 10±2 nm in aqueous medium which has some advantages mentioned point wise here. Firstly, the precipitation

method gets preference over the other processes because it is a simple process and consumes low energy. Moreover it has a control to show a discrepancy in the size of nanoparticles just by manipulating any condition of the reaction.[25] Secondly, among all the above mentioned geometry spherical geometry has minimum interfacial energies because it occupies least surface/volume and hence it gains structural stability.[26] Thirdly, there is very good economic benefit as the aqueous medium is very cheaper than the costly organic solvent medium.[27] Considering these above advantages it can be said that spherical ZnO NPs can be prepared easily with a low cost solvent which is very helpful economically and hence the present article should get importance in the field of nanoparticle synthesis.

MATERIALS AND METHODS

Reagent and chemicals used

In this study chemicals like Zincacetate dihydrate [$Zn(OAc)_2.2H_2O$], Sodium hydroxide [NaOH], Pyridine [C_6H_5N] and ethanol [EtOH] were used. All these above mentioned chemicals were used without any further purification. For reaction purpose double distilled water was used.

Synthesis of ZnO NPs

Few drops of 0.3M Pyridine were added slowly into a 25 mL of 0.2 M Zinc acetate dihydrate aqueous solution with continuous stirring. A colorless solution was produced in which drop wise 25 mL of 0.4M sodium hydroxide was added slowly. The white colour precipitation appeared which was washed by deionized water and 95% (v/v) ethanol. Then it was air dried at 60°C for about 3 hours. The synthetic procedure was used here is nearly similar with Kawano *et al.,*[28] The key reaction in this present study for the synthesis of ZnO NPs is represented stepwise as following:

$Zn(OAc)_2.2H_2O + \text{double distled water} \rightarrow Zn(OAc)_2(aq.)$

$NaOH + \text{double distled water} \rightarrow NaOH\ (aq.)$

$Zn(OAc)_2\ (aq.) + 2NaOH\ (aq.) \rightarrow Zn(OH)_2\ (aq.) + 2CH_3COONa(aq.)$

$Zn(OH)_2\ (aq.) + 2H_2O \rightarrow Zn(OH)_4^{-2}(aq.) + 2H^+$

$Zn(OH)_4^{-2}(aq.) \rightarrow ZnO \downarrow + H_2O + 2OH^-$

X-ray diffraction

Record of XRD pattern of synthesized ZnO NPs was done by Brucker AXS, Model D8, WI, USA. The CuKα radiation of λ =1.5409 Å was used during the diffractogram record. The scan rate and the 2θ range were set up were 5°/min and (20° to 80°) respectively. This experiment was performed at room temperature by keeping the voltage 40Kv and current 40mA.

UV-Visible spectroscopic measurement

Shimazdu TCC 240A UV-Vis spectrophotometer instrument was used to measure the SPR (surface Plasmon resonance) of the prepared aqueous solution containing ZnO NPs at 25°C. The path length of quartz cubate cell was 1 cm.

Field emission scanning electron microscope (FESEM)

Field emission scanning electron microscope was done by using (HITACHI S-4800, JAPAN) to study the surface morphology and size of the synthesized ZnO NPs. The voltage of the instrument was adjusted to 20kV. A dilute aqueous solution containing 1 or 2 drops of etanol of the powered ZnO NPs was prepared to make a film on carbon tape. Vacuum desiccator was used for the evaporation purpose and before taking FESEM the dried sample was gold coated.

Transmission electron microscopy (TEM)

Transmission electron microscopy was used to determine the core morphology and size of the synthesized ZnO NPs. This study was performed with the help of a high resolution transmission electron microscope (Jeol-HRTEM-2011, Tokyo, Japan) within the accelerating voltage range 80 to 85 kV. A droplet of diluted (50 times) sample solution containing ZnO NPs was casted on carbon coated copper grid (mesh size 300C, PRO Sci Tech). This sample containing Cu grid was incubated at least 3hrs before taking final TEM images. This sample containing Cu grid was further utilized for the selected area electron diffraction (SAED) and energy dispersive X-ray (EDX) studies which are very much necessary to characterize the synthesized ZnO NPs.

RESULTS AND DISCUSION

X-ray diffraction (XRD)

The XRD pattern of synthesized ZnO NPs was represented by Fig. 1. The recorded data with 2θ (in degree) and their corresponding planes were given by Table 1. This result indicates the nature of

the nanoparticles is crystalline and it matched with JCPDS file no. 00-036-1451.

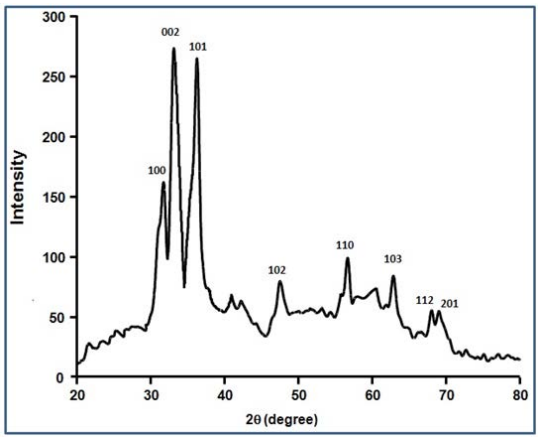

Fig. 1. XRD pattern of ZnO NPs

Table 1: The value of 2θ (in degree) with corresponding plane (hkl)

2θ (degree)	31.74	34.48	36.23	47.47	56.57	62.88	68.06	69.02
hkl	100	002	101	102	110	103	112	201

UV-Vis spectroscopic study

The SPR band of synthesized ZnO NPs was obtained at 347nm which is a good indication of formation of ZnO NPs. This result is also helpful to understand that the diameter of these NPs should be 10±2 nm which is further verified by FESEM and TEM images.

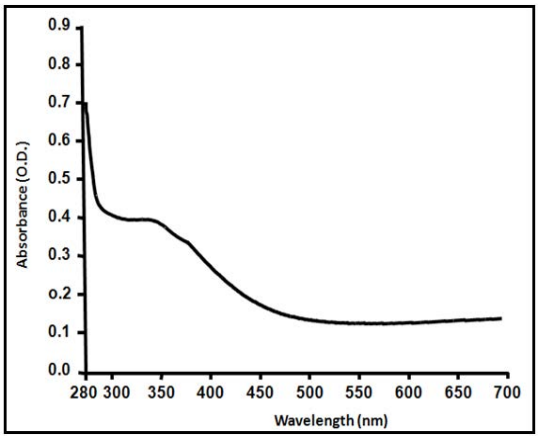

Fig. 2. The UV-Visible absorbance spectra of synthesized ZnO NPs

Field emission scanning electron microscope

FESEM pictures of synthesized ZnO NPs were shown by (Fig. 3a). The zoomed in pictures were represented by (Fig. 3b-c). The spherical shape of the produced nanoparticles was represented by (Fig. 3c). In this picture it has a clear view of the ZnO NPs of average diameter 10±2 nm.

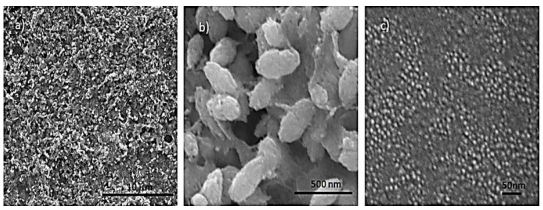

Fig. 3. SEM images of Zno nanoparticles (a-c) with different magnifications

Transmission electron microscopy (TEM)

TEM pictures were represented by (Fig. 4d-e) where it was observed that the morphology of the synthesized ZnO NPs were spherical and the average diameter of the nanoparticles were 10±2 nm. SAED pattern of the produced nanoparticles were shown by (Fig. 4f) which is bright in nature and this nature confirms that the nanoparticles are crystalline in nature.

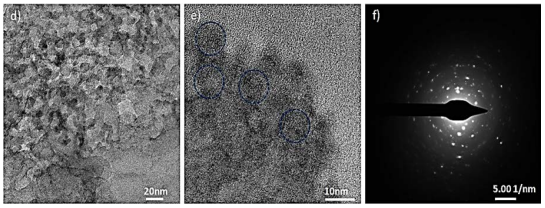

Fig. 4. TEM images of ZnO nanoparticles (d-e) with different magnification. SAED pattern is represented by f

Electron diffraction X-ray (EDX)

The EDX was performed to understand the elemental composition of the synthesized ZnO NPs (Fig. 5.) which shows optical absorption only for zinc and oxyzen element. There are two strong absorption peaks at 1.0 keV and 8.6 keV out of four optical absorption peaks of zinc and one for oxygen at 0.5 keV.[29] This result clearly reveals that ZnO NPs which were formed are of highest purity.

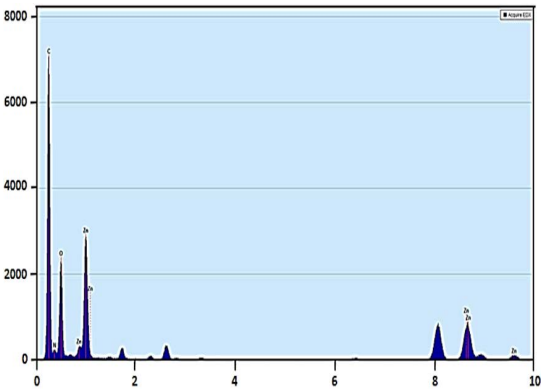

Fig. 5. EDAX of ZnO nanoparticles

CONCLUSION

The XRD study confirms the production of ZnO NPs with definite planes and very good crystalinity. The UV-Visible study gives the information of production of ZnO NPs as there is peak at 347nm. This result is also an indication of about the diameter of the nanoparticles which is further confirmed by SEM and TEM studies. Both of these studies suggest that the diameter of synthesized ZnO NPs is of 10±2 nm and their geometrical shape is spherical. The nature of these ZnO NPs is crystalline which was proved by SAED. The EDX study was an evident to proof the purity of synthesized ZnO NPs. So here pure ZnO NPs were produced of average diameter10nm in aqueous medium and are spherical in nature which indicates that this particular method may be helpful economically to boost the chemicophysical properties of synthesized nanoparticles as this synthetic method has a control over size and shape of the nanoparticles.[30]

ACKNOWLEDGEMENT

The author delightedly acknowledges Prof.U.C. Halder, Department of Chemistry, Organic Chemistry section, Jadavpur University for providing the essential research amenities. The author also thanks Department of Chemistry, Shibpur Dinobundhoo Institution (College) for initial research work facilities.

Conflict of Interest

The author declares that there is no conflict of interest about the publication of this article.

REFERENCES

1. Ozgur, U.; Alivov, Y. I.; Liu, C.; Teke, A.; Reshchikov, M. A.; Doan, S.; Avrutin V.; Cho, S.-J.; Morkoc, H. *J. Appl. Phys.*, **2005**, *98*, 1–104.
2. Chaudhry, Q.; Scotter, M.; Blackburn, J.; Ross, B.; Boxall, A.; Castle, L.; Aitken, R.; Watkins, R. *Food Additives & Contaminants:* Part A., **2008**, *25*, 241–258.
3. Li, Q.; Mahendra, S.; Lyon, D. Y.; Brunet, L.; Liga, M. V.; Li, D., Alvarez, P. *J. Water Res.,* **2008**, *42*, 4591–4602.
4. Singh, E.; Nalwa, H. S.; *RSC Adv.*, **2015**, *5*, 73575–73600.
5. Lee, H.; Park, I.; Kwak, J.; Yoon, D. Y.; Lee, C. *Appl. Phys. Lett.*, **2010**, *96*, Article ID 153306.
6. Chaudhary, S.; Umar, A.; Bhasin, K. K.; Baskoutas, S. *Materials.*, **2018**, *11*, 287–325.
7. Bhuyan, T.; Mishra, K.; Khanuja, M.; Prasad, R.; Varma, A. *Materials.*, **2015**, *32*, 55–61.
8. Sirelkhatim, A.; Mahmud, S.; Seeni, A.; Kaus, N. H. M.; Ann, L. C.; Bakhori, S. K. M.; Hasan, H.; Mohamad, D. *Nano-Micro Lett.*, **2015**, *7*, 219–242.
9. Zahra, J.; Iqbal, S.; Latif, M.; Ali, M.; Shad, M. A.; Tabish, T. A.; Ashiq, M. N.; Iqbal, F. Nanosci. and Nanotechnol. Lett., 2017, 9, 1–8.
10. Jiang, J.; Pi, J.; Cai, *J. Bioinorg. Chem. Applications.*, **2018**, 1–18,
11. Rajendran, R.; Balakumar, C.; Ahmmed, H. A. M.; Jayakumar, S.; Vaideki, K.; Rajesh, E. *Int. J. Eng. Sci. Technol.*, **2010**, *2*, 202–208.
12. Chaudhury, S.; Kaur, Y.; Umar, A.; Chaudhury, G. R. *J. Mol. Liquids.*, **2016**, *220*, 1013–1021.
13. Fiori, J. J.; Silva, L. L.; Picoli, K. C.; Ternus, R.; Ilha, J.; Decalton, F.; Mello, J. M. M.; Riella, H. G.; Fiori, M. A. *Materials Science Forum.,* **2017**, *899*, 248–253.
14. Sabir, S.; Arshad, M.; Chaudhari, S. K.; *The Scientific World Journal.*, **2014**, Article ID 925494:8.
15. Ao, W.; Li, J.; Yang, H.; Zeng, X.; Ma, X. *Powder Technology.,* **2006**, *168*, 148–151.
16. Swihart, M. T.; *Current Opinion in Colloid & Interface Science.*, **2003**, *81*, 127–133.
17. Ni, Y. H.; Wei, X. W.; Hong, J. M.; Ye, Y. Mater. Sci. Eng., B, *Solid State Mater. Adv. Technol.,* **2005**, *121*, 42–47.
18. Fakhari, S.; Jamzad, M.; Fard, H. K. *Green Chem. Lett. Rev.*, **2019**, *12*, 19–24.
19. Baruah, S.; Dutta, *J. Sci. Technol. Adv. Mater.,* **2009**, *10*, Article ID 013001.
20. Khoshhesab, Z. M.; Sarfaraz, M.; Houshyar, Z. *Synthesis and Reactivity in Inorganic, Metal-Organic, and Nano-Metal Chemistr.*, **2012**, *42*, 1363-1368.

21. Kumra, K. M.; Mandal, B. K.; Naidu, E. A.; Sinha, M.; Kumar, K. S. Reddy, P. S. Spectrochimica Acta Part A: *Molecular and Biomolecular Spectroscopy*., **2013**, *104*, 171-174.
22. Cao, Z.; Zhang, Z.; Wang, F. Wang, G. *Colloids Surf. A: Physicochem. Eng. Asppects*., **2009**, *340*, 161-167.
23. Hasanpoora, M.; Aliofkhazraeia, M.; Delavaria, H. *Procedia Materials Science*., **2015**, *11*, 320–325.
24. Geetha, K.; Chellapandian, M.; Arulnathan, N.; Ramanathan, *A. Vetworld*., **2020**, *13*, 121–126.
25. Romadhan, M. F.; Suyatma, N.; Taqi, F. *Indones. J. Chem*., **2016**, *16*, 117–123.
26. Liu, Y.; Tan, J.; Thomas, A.; Ou-Yang, D.; Muzykantov, V. R. *Ther Deliv*., **2012**, *3*,181–194.
27. Talapin, D.; Shevchenko, E., *Chem. Rev.,* **2016**, *116*, 10343–10345.
28. Kawano, T.; Imai, H. *Colloids Surf.* A., **2008**, *319*, 130–135.
29. Fakhari, S.; Jamzad, M.; Kabiri F. H. *Green Chem. Lett. Rev*., **2019**, *12*, 19–24.
30. Al Kordy H. M. H; Sabry, S. A.; Mabrouk, M. E. *M. Sci. Rep*., **2021**, *11*, 10924–10938.