Synthesis, characterization with optical property of Zn-O NPs using sol-gel route in alkaline medium

D. Tamilselvi [a, c *], N. Velmani [b], K. Rathidevi [c, d], Dr. V. Geethalakshmi [a], R. Nandhini [a]

[a] *Department of Science and Humanities, Rathinam Technical Campus, Coimbatore – 641 021, Tamilnadu, India*

[b] *Department of Chemistry, Government Arts College, Coimbatore -641 018, Tamilnadu, India*

[c] *Research & Development Centre, Bharathiar University, Coimbatore – 641 046, Tamilnadu, India.*

[d] *School of Foundational Sciences, Department of Chemistry, Kumaraguru College of Technology, Coimbatore – 641 049, Tamilnadu, India*

Zn-O nanoparticles were successfully synthesized by sol-gel route using $ZnSO_4.7H_2O$ as precursor in alkaline medium. Structural, morphological and optical property of Zn-O NPs was analyzed. Zn-O NPs were characterized by using X-ray diffraction (XRD), Fourier transform infrared spectral (FTIR), UV-Visible spectrophotometer, scanning electron microscopy (SEM) and Energy Dispersive X-ray analysis (EDAX).XRD pattern shows the purity of synthesized Zn-O nanoparticles particle size. Scanning electron microscopy (SEM) observations revealed remarkable change in morphological structure of Zn-O NPs. Analysis studies was confirmed, the functional groups and chemical bonding present in the synthesized Zn-O nanoparticles.

(Received January 25, 2022; Accepted May 30, 2022)

Keywords: Zn-O nanoparticles,Sol-gel method, XRD, SEM, Optical properties

1. Introduction

Nanosized particles of semiconductor materials have attracted much more interest in the research field due to their properties and applications in various field such as optoelectronics, catalysis, solar cells, ultraviolet light emitter, piezoelectric device, chemical gas sensors etc[1-6.]Zn-O nanoparticles is a n-type semiconductor material with a wide band gap of 3.37 eV and large excitation binding energy of 60 meV[7-14.]Zn-O nanoparticles have used in different application field such as varistors, photocatalysis, gas sensors, solar cells, pigment, transducers, etc. due to its unique physical, chemicall and biological properties of biocompatible, environmentally friendly, low cost and non-toxic nature. Zn-O nanoparticles have several advantages which include unique chemical and thermal stability, robustness, and long shelf life over other metal oxides such as TiO_2, WO_3, SiO_2, and Fe_2O_3. Zinc oxide exists in the following phases: hexagonal quartzite, cubic zinc blende, and cubic rock salt. Zinc oxide as one of the safest metal oxides, Food and Drug Administration (FDA) includes also it can be used in food industries. Zn-O nanoparticles are classified into based on the number of dimensions, such as zero-dimensional (0D), one-dimensional (1D), two-dimensional (2D), and three-dimensional (3D)[15].Zinc oxide nanoparticles were synthesized by using different chemical and physical methods such as, sol–gel, hydrothermal, precipitation and co-precipitation, chemical vapour deposition, spray pyrolysis, magnetic sputtering, microwave-assisted technique, solvothermal etc.

Zn-O nanoparticles having a lot of morphology structures have been reported due to their physical and chemical properties of crystals, such as nano-combs[15, 16], nanotubes[17], nano-springs[18], nanorods[19], nanorings[20] nanowires[21] and nanoflower[22],Differenttype of zinc salts were used as a

* Corresponding author: tamilselviupdate@gmail.com
https://doi.org/10.15251/JOBM.2022.142.69

precursor to synthesize Zn-O nanoparticles such as zinc acetate dehydrate ($Zn(CH_3COO)_2 \cdot 2H_2O$)), zinc nitrate hexahydrate ($Zn(NO_3)_2 \cdot 6H_2O$), zinc chloride ($ZnCl_2$) and zinc sulphate ($Zn(SO_4)_2 \cdot 7H_2O$).

In this present paper, the aim of this work is to synthesize Zn-O nanoparticles by sol-gel method using $ZnSO_4.7H_2O$ as precursor in alkaline medium. Synthesized Zn-O nanoparticleswere characterized by XRD, FT-IR, UV-Visible, SEM, EDAX analysis and the results of their structural, morphological, optical properties and elemental analysis has been discussed.

2. Materials and methods

2.1. Materials Required

The high purity chemicals (>99% purity) such as ($Zn(SO_4)_2 \cdot 7H_2O$), and Sodium Hydroxide (NaOH) were purchased from Sd. AR grade fine chemicals were used as the precursors without further purification.

2.2. Synthesis of Zn-O NPs.

Zn-O NPs was synthesized by using sol-gel route method in the following manner. To synthesis pure Zn-O, 1M Zinc sulphate $ZnSO_4.7H_2O$ was dissolved in 100ml double distilled water under vigorous stirring for 10 minsby using magnetic stirrer (solution A). Next add drop wise 0.2M sodium hydroxide (NaOH) solution into aqueous solution (A) with vigorous stirring for 2hrs to attain p^H level-12. The gelatinous white precipitate was formed. After that precipitate filtered and washed with distilled water, ethanol, dry for overnight at room temperature and dried in oven at 100°C for six hours. The dried white precipitates then annealed at 500°C for two hours followed by grinding to get fine particles. These were used for various characterization techniques. [15-25].The flow chart of the synthesized Zn-O nanoparticles is shown in Fig.1

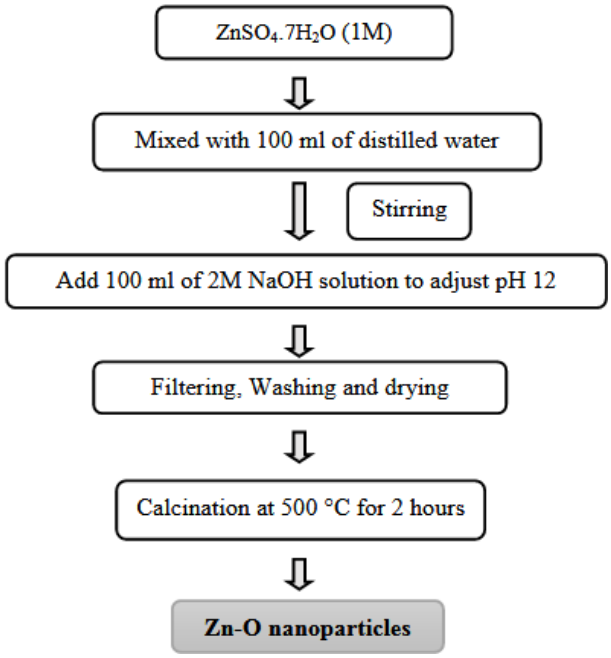

Fig.1. Flow chart of synthesized Zn-O nanoparticles.

3. Characterization techniques

A synthesized Zn-O nanoparticle has been analyzed at normal room temperature by XRD with 2θ scanning angle range was maintained between 30 - 90˚. The powder samples have been examined for identification of functional groups by using FTIR measurements range from 400 – 4000 cm^{-1}. The optical studies recorded by UV-visible Spectro-photometer from 100 to 900 nm. The structure and Surface morphology of the particles also have been examined using (HR-SEM) HR-field emission scanning electron operated at 10kV. The synthesized nanoparticles have been examined with Energy-Dispersive X-ray Spectrometer (EDAX) to identify elemental composition analysis.

4. Results and discussion of Zn-O NPs.

4.1.X-ray diffraction (XRD) analysis

Fig.2. indicates the XRD analysis of Zn-O NPs.XRD analysis clearly shows the crystalline nature and well-defined sharp diffraction peaks corresponding to planes of (100), (002), (101), (102), (110), (103), (200),(112),(201), (004) and (202) were observed. XRD peaks clearly show wurtzite - hexagonal structure matched with card No.36-1451of JCPDS data. [22-29].

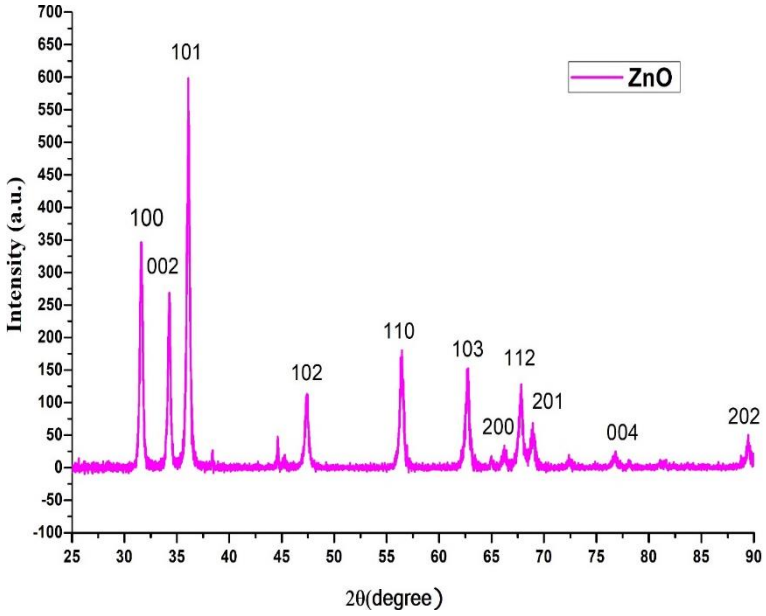

Fig. 2. XRDanalysis ofsynthesized Zn-O nanoparticles.

De-bye-Scherer formula was used to calculate the average crystallite size of Zn-O NPs.

$$D = 0.94(K)*1.54(\lambda)/\beta \cos \theta$$

where D – average crystallite size
K – Scherer's constant (0.94)
λ – Wavelength (1.54)
β – FWHM
θ – Bragg angle

The average crystallite size was calculated as 71 – 87 nm for synthesized Zn-O NPs depending on the crystal growth condition.

4.2. Fourier transform infrared (FTIR)–analysis

The chemical bonding and constituting elements in the samples can be analyzed by using FTIR spectrum. Fig. 3 shows FTIR spectrum of Zn-O NPs was observed from 400 - 4000 cm^{-1}. The peak observed at 686 cm^{-1} corresponds to Zn-O vibration of stretching[22].The broad peaks observed around 3609 $cm^{-1}$for stretching vibration of water molecule.The peak observed around 1693 $cm^{-1}$for bending vibrations of water molecule.The peak observed at 2926 cm^{-1} corresponds to C-H symmetric stretching vibration modes[25-28].The peak observed at 2308 cm^{-1} O = C = O symmetric stretching vibration mode [60]. The peak observed at 1514 cm^{-1} for antisymmetric vibration of mode of (COO^{-}) [29].

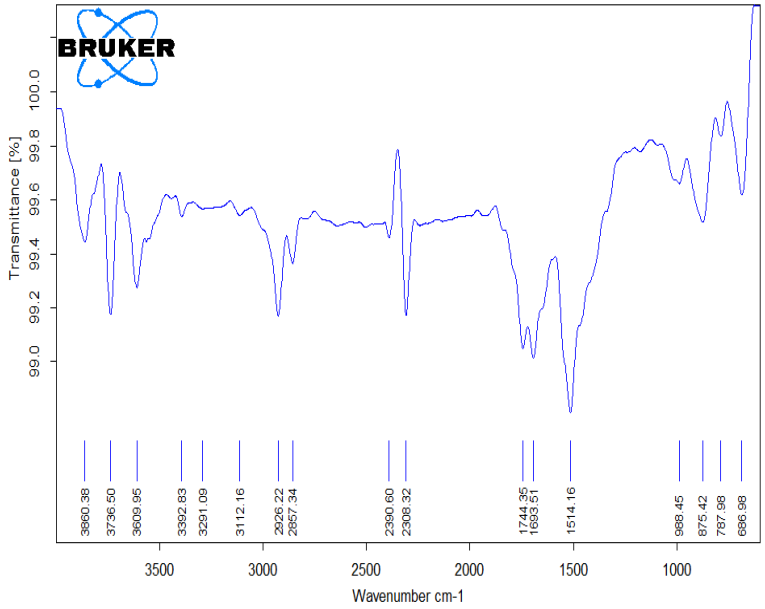

Fig. 3. FTIR analysis ofsynthesized Zn-O nanoparticles.

4.3. SEMAnalysis

The Surface morphology of Zn-O NPs was exhibited in Fig 4. From the SEM image hexagonal crystal shape was observed for Zn-O NPs.

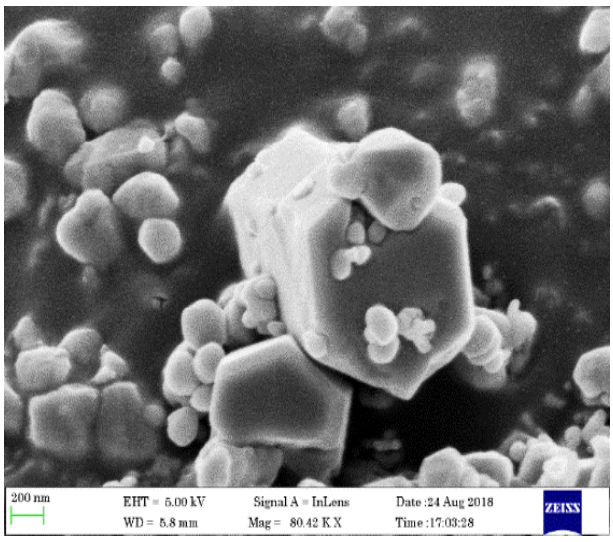

Fig. 4. Surface morphology ofZn-O NPs.

4.4. EDAX Analysis

Fig. 5.indicates the composition of element present inZn-O NPs was measured by using EDAX spectrum. From this spectrum, the elements of Zn, O, and C present in the samples. The C peaks correspond to their origin in copper grid. EDAX spectrum shows no impurities present in Zn-O samples.Weight percentage of the element was also confirmed by EDAX spectrum and presented in Table 1.

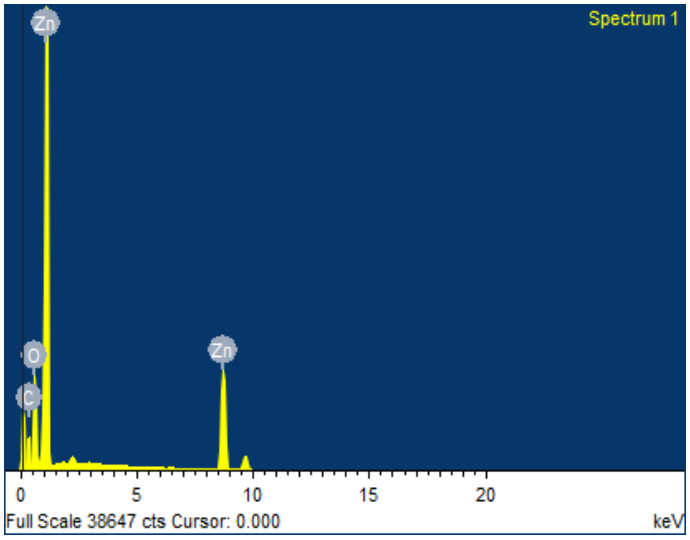

Fig. 5. EDAX analysis ofsynthesized Zn-O nanoparticles.

Table 1. Weight percentage of synthesized Zn-O nanoparticles from EDAX spectrum.

Element	Weight %
Zn	55.98
O	22.88
C	21.13
Total	100

4.5. Ultra violet -visible(UV-VIS)Analysis

The ultra violet -visible absorption peak of synthesized Zn-O NPs was exhibited in Fig 6. Generally, the position of absorbance value is depending on the size of the nanoparticles, oxygen deficiency, grain structure, band gap etc. The absorption peak of the Zn-O nanoparticles appeared at 321 nm clearly observed[30-36].

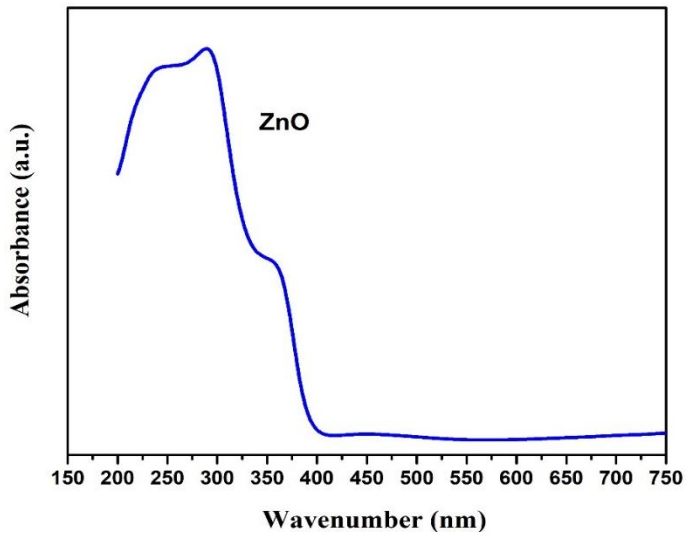

Fig. 6. Ultra violet -visible analysisofZn-O NPs.

5. Conclusion

Zn-O NPs are synthesizedin alkaline medium byusing sol-gel method. XRD analysis reveals structure of hexagonal wurtzite-type with calculated average crystallite size in range of 71-87 nanometer. FTIR analysis reveals absorption peak around 686 cm^{-1} corresponds to Zn-O stretching vibration is observed.SEM observations reveal the hexagonal shape crystal structure for Zn-O NPs. Purity of Zn-O NPs was confirmed by EDAX analysis. Zn-O absorption peak appeared at 321 nm clearly observed in the UV-Visible spectral analysis.

References

[1] Y. M. Hao, S. Y. Lou, S. M. Zhau, R. J. Yuan. G. Y. Zhu, N. Li, Nanoscale Res. Lett. 7, 100 (2012); https://doi.org/10.1186/1556-276X-7-100

[2] P. M. Shirage, Materials Today 16, 505 (2013); https://doi.org/10.1016/j.mattod.2013.11.007

[3] N. Goswami, A. Sahai, Mat. Res. Bull. 48, 346 (2013); https://doi.org/10.1016/j.materresbull.2012.10.045

[4] J. Jadhav, M. Patange, S. Biswas, Carbon Sci. Tech. 5, 2 (2013).

[5] G. Srinet, R. Kumar, V. Sajal. J. App. Phy. 114, 033912 (2013); https://doi.org/10.1063/1.4813868

[6] G. Srinet, R. Kumar, V. Sajal, Cer. Int. 39, 7557 (2013); https://doi.org/10.1016/j.ceramint.2013.03.008

[7] Yu-Cheng Chang, RSC Adv. 4, 56241 (2014); https://doi.org/10.1039/C4RA10243F

[8] S. Inbasekaran, R. Senthil, G. Ramamurthy, T. P. Sastry, EngTechnol 3(1), 8601 (2014).

[9] S. Sabir, M. Arshad, S. K. Chaudhari, 2014, zinc oxide nanoparticles for revolutionizing agriculture: synthesis and applications; https://doi.org/10.1155/2014/925494

[10] A. Shrishti, P. S. Kumar, S. Monika, Int J Chem Pharma Anal 1(3), 141 (2014).

[11] M.M. Rahman et al. Journal of Industrial and Engineering Chemistry, 2014, 20, 1071; https://doi.org/10.1016/j.jiec.2013.06.044

[12] Xingyan Xu, Chuanbao Cao, Journal of Magnetism and Magnetic Materials 377, 308 (2015); https://doi.org/10.1016/j.jmmm.2014.10.130

[13] Kyung Ho Kimn, ZhuguangJin, Yoshio Abe, Midori Kawamura, Materials Letters 149, 8

(2015); https://doi.org/10.1016/j.matlet.2015.02.109

[14] Rana et al., AIP Advance 5, 097118 (2015); https://doi.org/10.1063/1.4930598

[15] Y. Kumar, A. K. Rana, P. Bhojane, M. Pusty, V. Bagwe, S. Sen, P. M. Shirage, Materials Research Express 2, 105017 (2015); https://doi.org/10.1088/2053-1591/2/10/105017

[16] Rana et al. Applied Surface Science 379, 23 (2016); https://doi.org/10.1016/j.apsusc.2016.04.066

[17] Parasharam M. Shirage, Amit Kumar Rana, Yogendra Kumar, Somaditya Sen, S. G. Leonardi, G.Neri, RSC Adv. 6, 82733 (2016); https://doi.org/10.1039/C6RA15891A

[18] Qiaoqiao Yin et al., RSC Adv. 6, 38653 (2016); https://doi.org/10.1039/C5RA26631A

[19] M. Sh. Abdel-Wahab, AsimJilani, I.SYahia, Attieh A. Al-Ghamdi. Superlattices and microstructures 94, 108 (2016)l https://doi.org/10.1016/j.spmi.2016.03.043

[20] Amit Kumar Rana, Prashant Bankar, Yogendra Kumar, Mahendra A. Dattatray, J. Parasharam, M. Shirage, RSC Adv. 6(106), 104318 (2016); https://doi.org/10.1039/C6RA21190A

[21] R. Saravana Kumar, S. H. S. Dananjaya, Mahanama De Zoysa, Minyang Yang, RSC Adv. 6, 108168 (2016).

[22] Kun Xu, Changzhen Liu, Rui Chen, Xiaoxiang Fang, Xiuling Wu, Jie Liu, Physica B 502, 155 (2016); https://doi.org/10.1016/j.physb.2016.07.017

[23] D. Guruvammal, S. Selvaraj, S. MeenakshiSundar, Journal of Alloys and Compounds 682, 850 (2016); https://doi.org/10.1016/j.jallcom.2016.05.038

[24] Z. A. Fattah, Int. J. Eng. Sci. Res. Tech. 5(7), 418 (2016).

[25] Y. Wang et al, J. Mater. Res. 31(15), 15 (2016); https://doi.org/10.1557/jmr.2016.191

[26] S. Fabbiyola, V. Sailaja, L. Johnkennedy, M. Bououdina, J. Judith Vijaya, Journal of Alloys and Compounds 694, 522 (2017); https://doi.org/10.1016/j.jallcom.2016.10.022

[27] UmadeviGodavarti, V. D. Mote, MadhavaprasadDasari,Modern Electronic Materials 3, 179 (2017); https://doi.org/10.1016/j.moem.2017.10.006

[28] R. Sankar Ganesh, E. Durgadevi, M. Navaneethan, V. L. Patil, S. Ponnusamy, C. Muthamizhchelvan, S. Kawasaki, P. S. Patil, Y. Hayakawa, Chemical Physics Letters, 2017.

[29] G. Pallavi, Shankar Undre, D. Birajdar, R. V. Kathare, K. M. Jadhav, Procedia Manufacturing, 477 (2018)

[30] Madhava P. Dasari, UmadeviGodavarti,VishwanathMote,Processing and ApplicationofCeramics 12(2), 100 (2018); https://doi.org/10.2298/PAC1802100D

[31] D Tamilselvi, N Velmani, K Rathidevi.Egyptian Journal of Chemistry,Vol. 62, No. 6. pp. 785 - 795 (2019).

[32] S. Satheeskumar, V. Jeevanantham, D. Tamilselvi.Journal of Ovonic Research 14, 9-15.(2018).

[33] D Tamilselvi, N Velmani, K Rathidevi.Mediterranean Journal of Chemistry,9 (5),403-410,(2019).

[34] D Tamilselvi, N Velmani, K Rathidevi.Journal of Ovonic Research.16, p. 123 - 130. (2020).

[35] K. Rathidevi, N. Velmani, D. Tamilselvi. Journal of Ovonic Research 16, p.337 - 343, (2020).

[36] E. Y. Shaba · J. O. Jacob · J. O. Tijani· M. A. T. Suleiman, Applied Water Science (11) 48 (2021); https://doi.org/10.1007/s13201-021-01370-z