

# Preparation and Spectroscopic Characterization of ZnS nanoparticles

O P Chimankar<sup>1\*</sup>, N N Padole<sup>1</sup>, N R Pawar<sup>1</sup> and P G Agone

<sup>1</sup>Department of Physics, RTM Nagpur University, Nagpur-440033, India  
R.G. Engineering College, Chandrapur, India

**Abstract:** *The aim of the present study was to prepare nano-sized ZnS. The ZnS semiconducting nanoparticles were synthesized by simple chemical reaction of Zinc salt like Zinc Acetate [Zn(CH<sub>3</sub>COO)<sub>2</sub>], sodium sulfide (Na<sub>2</sub>S) and DMF [H-CO-N(CH<sub>3</sub>)<sub>2</sub>] as a stabilizing agent. The main advantage of this method is to synthesized semiconductor nanomaterials with wide band gap and nanoparticles are chemically stable over a long time. The structural, morphological, chemical composition and optical properties of synthesized nanoparticles have been investigated by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), UV-Vis spectroscopy (UV) and Scanning electron microscopy (SEM). XRD analysis shows that sample prepared were the wurtzite structure and the crystallite size estimated using Debye Scherer's formula was found to be in the range of 35 to 40 nm. The SEM analysis revealed the formation of ZnS nanoparticles with almost spike like shape. In optical characterization the spectra becomes transparent for visible radiation.*

**Keywords:** Chemical Synthesis, ZnS Nanoparticles, XRD, FTIR, UV, SEM Spectroscopy

## 1. Introduction

Last two decades have witnessed a rapid advancement in various techniques for the fabrication of nanoparticles. Among various classes of nanoparticles, II-VI class inorganic semiconductor nanomaterials like CdS, ZnS and CdSe emerged as important materials because of the ability to synthesize them in numerous configurations for the applications in optoelectronics devices. ZnS is a direct wide bandgap (3.91 eV) compound semiconductor with a high index of refraction and a high transmittance in the visible range and it is one of the more important materials in photonics research. Nanostructure ZnS have unique physical, chemical, electrical, optical and transport properties, which differ from the bulk material and even single atoms. As the radius of the crystallite approaches the Bohr radius of an exciton, the energy gap begins to wide, this is due to quantum size effect. ZnS nanoparticles has wide band gap, it is widely used in photonics like optical switches, optical filters and sensors, in optoelectronics devices, LED, photocells, laser, address decoder, photocatalysis, water purification, hydrogen production, photovoltaic solar cell, paints and in engineered plastic for good thermal stability. It is also applicable for food and pharmaceutical product. These properties emerge from the high surface to volume ratio of nanoparticles. We anticipate inspiring interest in studying the novel physical, optical and chemical properties of ZnS nanostructures. ZnS is extensively studied as it has numerous applications to its credit. There are various chemical based methods available for the synthesis of ZnS nanomaterials.

In its bulk form, ZnS is typically found to have the zinc blend crystal structure at room temperature. The zinc blend structure is cubic. At elevated temperatures, bulk ZnS can undergoes a phase transformation from the cubic zinc blend structure to a hexagonal crystal structure known as the wurtzite structure. This transformation has been shown to occur at 1020°C. The zinc blend and wurtzite structures are very similar.

## 2. Experimental Details

### 2.1 Materials and Methods

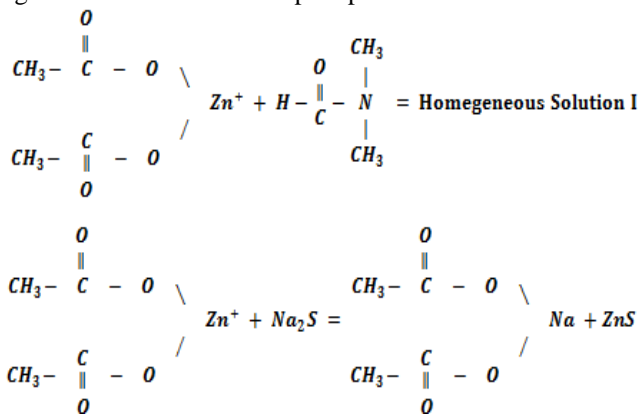
Zinc Acetate [Zn(CH<sub>3</sub>COO)<sub>2</sub>] and sodium sulfide (Na<sub>2</sub>S) were purchased from Merck Company. The XRD measurements of synthesized samples were carried out using a Philips X-pert PRO powder diffractometer with Cu-K $\alpha$  radiation ( $\lambda = 1.54060 \text{ \AA}$ ) in the scan range 0-100. The morphology of synthesized sample was studied using Scanning Electron Microscopy (GEOL 6380A) by a sputtering technique with gold as covering contrast material in VNIT Nagpur. The IR spectra were recorded using Bruker spectrometer with KBr pellets in the range from 400 to 4000 cm<sup>-1</sup> from Shivaji science college, Nagpur.

### 2.2 Synthesis of Nanoparticles

The synthesis of ZnS nanoparticles was carried out by chemical method using Zinc Acetate [Zn(CH<sub>3</sub>COO)<sub>2</sub>], DMF [H-CO-N(CH<sub>3</sub>)<sub>2</sub>] and sodium sulfide (Na<sub>2</sub>S) as source materials. All the reagents were of analytical grade and used without further purification. All steps of the synthesis were performed at low temperature and ambient conditions. The schematic of formation of ZnS nanoparticles as shown in figure 1. In a typical preparation, specific molar concentration of zinc acetate was prepared in 100 ml of DMF and kept it for a magnetic stirrer for 10 minutes. DMF used as a common solvent for chemical reaction to enhance reactivity and also stabilizing agent. Then the equimolar solution of sodium sulfide was taken in a burette and it was added drop wise to the solution which was kept for stirring using a magnetic stirrer at 70°C, which resulted in formation of ZnS nano colloid.

The stirring was continued for 2 hours for the completion of precipitation reaction. Nanoparticles were collected by centrifugation at 2000 rpm for 15 minutes. The precipitate was filtered, washed with acetone dried in a vacuum oven at 60°C. Gray powder of ZnS nanocrystallites has been obtained. In this process as ZnS is a less electropositive

element and hence less reactive. Sodium metal is highly electropositive and hence more reactive. Therefore sodium replaces Zn<sup>2+</sup> ions from zinc acetate and converted into sodium acetate and zinc sulfide. Zinc sulfide is insoluble in organic solvent so it will be precipitate.



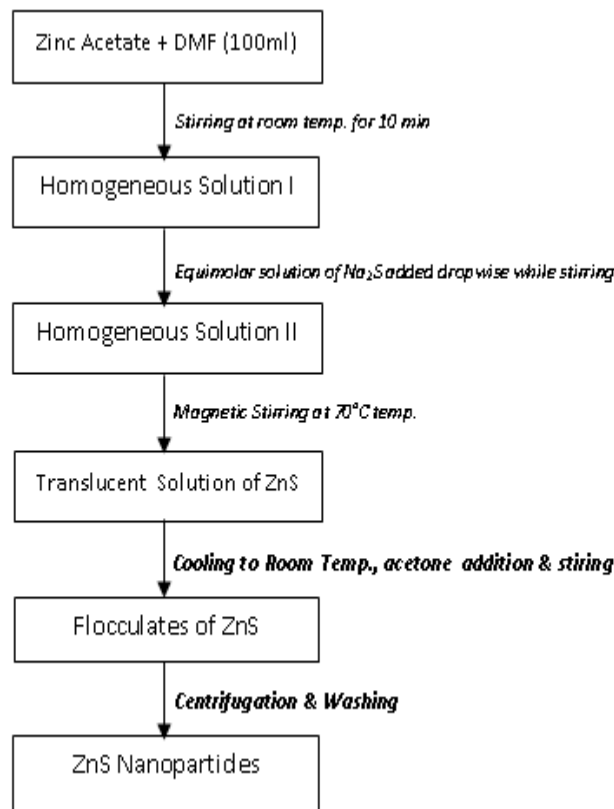
### 3. Results and Discussion

Figure 2 shows the XRD patterns of the prepared ZnS nanoparticles. The ZnS nanoparticles were characterized by X-ray diffraction (XRD) (Model: Philips XPERT PRO type diffractometer) with Cu K $\alpha$  radiation. X-ray diffraction technique is used to determine the crystallite size and structure of ZnS nanoparticles. The lattice parameters were also calculated. It reveals the hexagonal structure for the prepared nanoparticles and the features correspond to the (111), (200), (220), (311) and (422) planes, which show obvious size broadening effects, indicating the finite size of the nanoparticles.

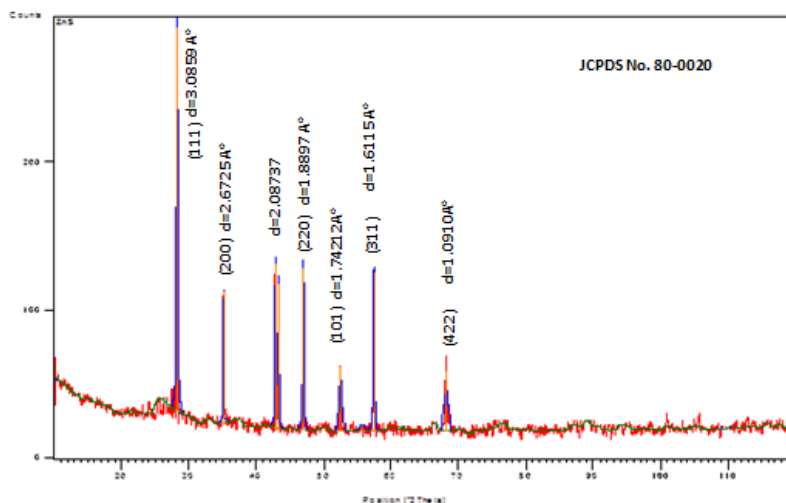
The obtained pattern is found to match with the earlier reported pattern (JCPDS CAS No. 80-0020). The peaks corresponding to each plane is identified and indexed. From XRD it is evident that the obtained ZnS has hexagonal wurtzite phase. The mean crystallite size of the sample is calculated using Scherer's formula

$$D = \frac{0.94 \lambda}{\beta \cos \theta}$$

where D is the average crystallite size,  $\lambda$  is the x-ray wavelength (1.5405 Å) and  $\beta$  is full width at half maximum in radian (B. D. Cullity, 2001). The average crystallite size of ZnS nanoparticles is 37.46 nm. The details pertaining to the crystallite size calculation is given in table 1.



**Figure 1:** A Schematic of the formation of ZnS Nanoparticles



**Figure 2:** XRD Spectra of ZnS Nanoparticles

**Table 1:** Structural parameters of ZnS Nanoparticles

FWHM(Degree2Th)	K	Cos $\theta$	$\beta$ (Radian)	L	Hkl
0.2244	0.914	0.969809732	0.003914533	38.13 nm	(111)
0.1632	0.914	0.953482319	0.002846933	53.3284 nm	(200)

0.2448	0.914	0.930743428	0.0042704	36.4181 nm	(121)
0.2040	0.914	0.917309749	0.003558667	44.345 nm	(220)
0.4896	0.914	0.896935259	0.0085408	18.8947 nm	(101)
0.2040	0.914	0.877128211	0.003558667	46.376 nm	(311)
0.9792	0.914	0.82822261	0.0170816	10.2323 nm	(422)

#### 4. Fourier Transform Infrared Spectrum (FTIR)

Although the synthesized ZnS was a white gray powder, its purity was further examined by means of FTIR spectrometry. The FTIR spectroscopy measures the bond vibration frequency in a molecule and use to determine the functional group. The FTIR- spectra of ZnS Nanopowder were recorded in the range of 600 - 4000  $\text{cm}^{-1}$  are shown in figure3. It is to

be noted that all the absorption peaks of the ZnS nanoparticles used in the present work, totally matches with those reported in the literature. The medium strong band observed at 2935  $\text{cm}^{-1}$  suggests the presence of C-H stretch. The spectra shows the peak at 1600  $\text{cm}^{-1}$ , which is due the presence of O-H bending. The region from 636  $\text{cm}^{-1}$  to 1355  $\text{cm}^{-1}$  shows finger print region.

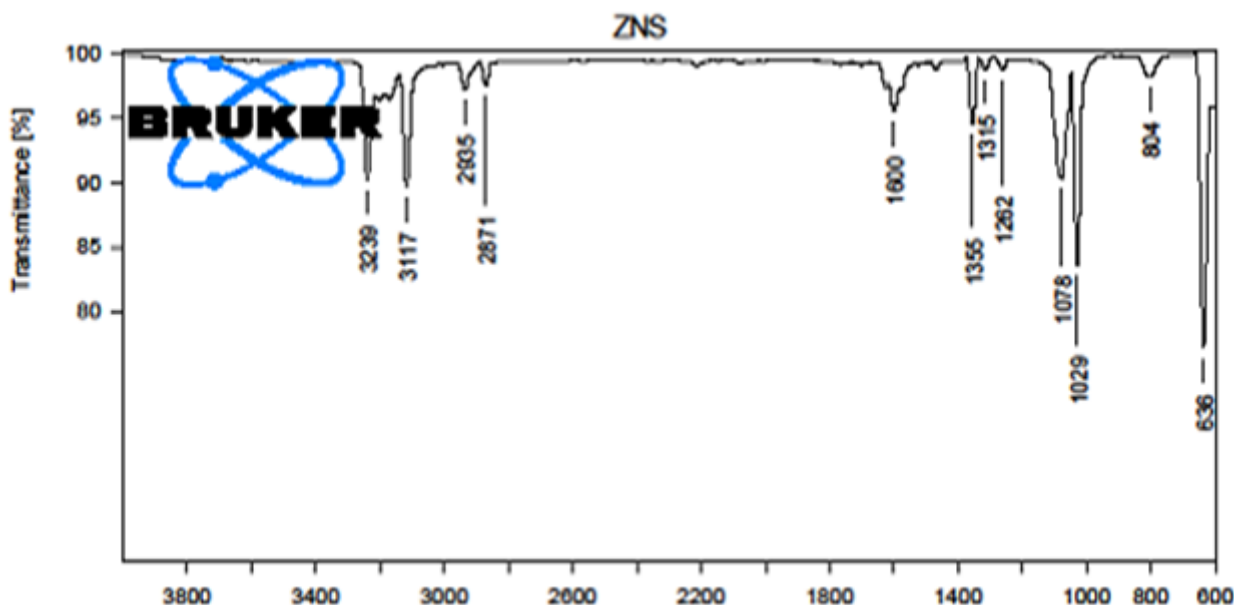


Figure 3: FTIR spectra of ZnS Nanoparticles

#### 5. Scanning Electron Microscopy (SEM)

The morphology of synthesized sample was studied using Scanning Electron Microscopy (GEOL 6380A) by a sputtering technique with gold as covering contrast material. The High Resolution Scanning Electron Microscopic image of nano-Zinc sulfide powder is shown in the Figure 4. It can be observed that the particles are of spike like shape. The grain size of ZnS nanoparticles ranges from 35 nm - 40 nm.

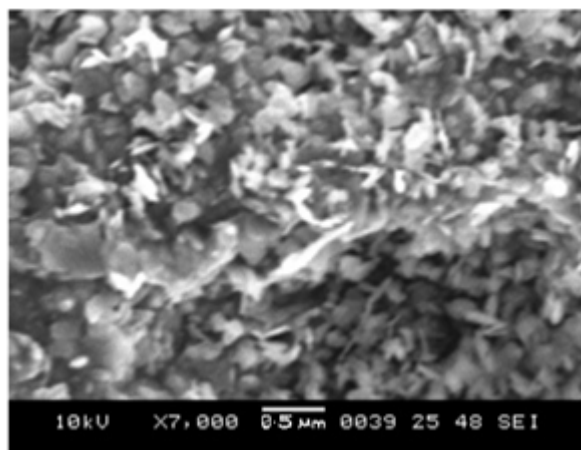


Figure 4: SEM image of ZnS Nanoparticles

#### 6. UV-Visible Spectroscopy

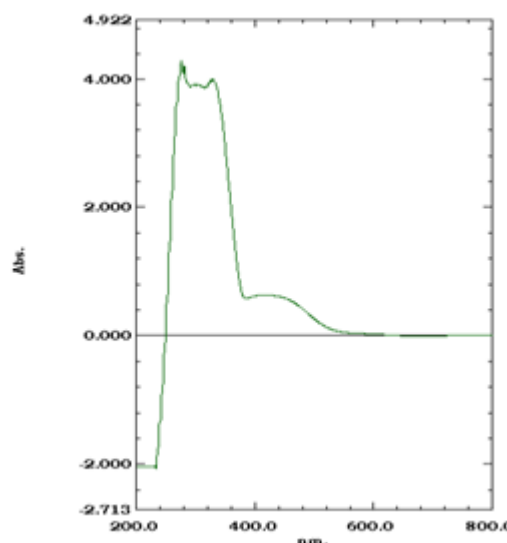


Figure 5: Absorption spectra of ZnS nanoparticles

The study of UV-Vis absorption spectra is important to understand the behavior of nanocrystal . A fundamental property is the band gap. Optical excitation of electrons

across the band gap is strongly allowed, producing an abrupt increase in absorption at the wavelength corresponding to the band gap energy. Fig.(5) shows the optical absorption spectra of prepared ZnS nanoparticles recorded in the range of 200 nm to 800 nm. The absorption edge found to be at 270 nm. In this compound Zn is present as a  $Zn^{2+}$ . Hence its d orbital is completely filled. Therefore excitation of electron from d orbital is not possible.

## 7. Conclusion

ZnS nanoparticles were prepared by chemical method. The average crystallite size of the sample was calculated using Scherrer equation and found in the range of 35 nm to 40 nm. The sample is characterized by XRD, SEM, FTIR, and UV vis spectroscopy. The SEM analysis showed the formation of spike like nanoparticles. It is noteworthy that the chemical method is effective for obtained pure phase nanomaterial's with controllable size, uniform morphology and shape.

## 8. Acknowledgement

The authors OPC grateful to the university grand commission, New Delhi, for providing financial assistance in the form of Major Research Project.

## References

- [1] Bhaskarjyoti Bodo, Ranjit Singha, and Sukumar Chandra "Structural and Optical Properties of Chemically Synthesized ZnS Nanostructures" Das International Journal of Applied Physics and Mathematics, Vol. 2, No. 4, July 2012
- [2] Anoop Chandran, Nimmi Francis, Tibi Jose and K C George "Synthesis, structural characterization and optical bandgap determination of zns nanoparticles" ISSN: 0973-7464 Vol. XVII: No.1 & 2 SB Academic Review 2010 : 17-21
- [3] Anita Jain, Sanjay Panwar, Sunil Kumar "Synthesis & Characterisation of Core Shell ZnS/ZnO Nanoparticles" International Journal of Applied Engineering Research, ISSN 0973-4562 Vol.7 No.11 (2012) © Research India Publications; <http://www.ripublication.com/ijaer.htm>
- [4] Senapati U.S.1, Jha D.K. and Sarkar D. "Green Synthesis and Characterization of ZnS nanoparticles" Research Journal of Physical Sciences ISSN 2320-4796 Vol. 1(7), 1-6, August (2013) Res. J. Physical Sci.
- [5] D. amaranatha reddy, g. murali, a. divya, r.p. vijayalakshmi, b.k. reddy "Investigations on ZnS nanoparticles based on synthesis temperature for optoelectronic device applications" journal of optoelectronics and advanced materials Vol. 12, No. 11, November 2010, p. 2185 - 2189
- [6] Shubhangi D. Bompilwar, Subhash B. Kondawar, Vilas A. Tabhane "Impedance Study of Nanostructure Cadmium Sulfide and Zinc Sulfide" Available online at [www.Scholarsresearchlibrary.com](http://www.Scholarsresearchlibrary.com) Archives of Applied Science Research, 2010, 2 (3): 225-230
- [7] rita john, s. sasi florencea chalcogenide "Optical, structural and morphological studies of bean- like zns nanostructures by aqueous chemical method" letters vol. 7, no. 4, april 2010, p. 269 – 273

- [8] M. Bilge a, S. Özdemir Kart a, H.H. Kart a, T. Cagin "Mechanical and electrical properties of ZnS" under pressure of Achievements in Materials and Manufacturing Engineering VOLUME 31 ISSUE 1 November 2008
- [9] Gopa Ghosh, Milan Kanti Naskar, Amitava Patra , Minati Chatterjee "Synthesis and characterization of PVP-encapsulated ZnS nanoparticles" Optical Materials 28 (2006) 1047–1053
- [10] G. Nabyouni, R. Sahraei, M. Toghiani, M. H. Majles ara and K. Hedayati "Preparation and characterization of nanostructured zns thin films grown on glass and n-type si substrates using a new chemical bath deposition technique" rev.adv.mater.sci. 27(2011) 52-57
- [11] Xiaoyan Li a, Chenguo Hua,n, Hong Liu b, Jing Xu a, Buyong Wana, Xue Wanga "ZnS nanoparticles self-assembled from ultrafine particles and their highly photocatalytic activity" Physica E 43 (2011) 1071–1075
- [12] Dagui Chen,a Feng Huang, a Guoqiang Ren,a Dongsong Li,b Meng Zheng,a Yongjing Wangb and Zhang Lin "ZnS nano-architectures: photocatalysis, deactivation and regeneration" [www.rsc.org/nanoscale](http://www.rsc.org/nanoscale) | Nanoscale
- [13] Mahsa Mobarraz, Mohammad Javad Chaichi1, Amin Karimi Zindashti2, Mohammad Reza Ganjali "Application of Functionalized ZnS Quantum Dots as a Fluorescence Probes for Determination of Amino Acids" 2011 International Conference on Nanotechnology and Biosensors IPCBEE vol.25(2011) © (2011) IACSIT Press, Singapore