

Characterisation of Undoped ZnS and FeCl₃ Doped ZnS Nanoparticles are Synthesized by Chemical Precipitation Method

K.G. Aarthe¹, P. Kiruthika¹

ABSTRACT

Nanoparticles of Ferric chloride (FeCl₃) doped Zinc Sulphide (ZnS) and Undoped Zinc Sulphide (ZnS) had been synthesized successfully by simple chemical precipitation method. Particle sizes have been calculated from X-ray diffraction (XRD) analysis which confirms the nano structure of the samples. The Molecular structure of the compound was determined by the Fourier transform infrared spectroscopy (FTIR) analysis and the different vibrational bands confirmed the functional groups present in the sample. The bandwidth of the absorbance is examined by using (UV)-Visible Spectroscopy. The Morphological structures have been confirmed by using Scanning Electron Microscope (SEM). Energy Dispersive analysis of X-ray (EDAX) shows the composition of elements present in the nanoparticles. The applications of ZnS were used in the field such as Field Emitting Diodes (FET), sensors (gas sensors, biosensors), Flat panel displays, Electroluminescence.

Keywords: XRD, FTIR, UV, SEM, EDAX.

Author Affiliation: ¹Department of Physics, Vellalar College for Women, Erode, Tamil Nadu- 638012.

Corresponding Author: K.G. Aarthe. Department of Physics, Vellalar College for Women, Erode, Tamil Nadu- 638012.

Email: k.g.aarthe@vcw.ac.in

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1. INTRODUCTION

Nanoscience is an emerging area of science which involves the study of materials on an ultra-small scale. Recent advances in nanotechnology and various quantum size effects in nano-scale particles imply that many novel devices of the future will be based on properties of nanomaterials. Transition metal ions and rare earth ions doped ZnS semiconductor materials have a wide range of applications in electroluminescence devices, phosphors, Light Emitting devices and optical sensors. ZnS can be synthesized using several methods as like homogeneous precipitation, Microwave methods, Thermal evaporation, Pulsed laser deposition, Spray pyrolysis and Solution gelation (Sol-Gel).^[1-6]

2. EXPERIMENTAL PROCEDURE

The pure ZnS nanoparticles were synthesized by dissolving 4M of ZnCl₂ in 50 ml of distilled water and allowed to stirrer in magnetic stirrer for 1hour. Then the 4M of Na₂S was dissolved in 50ml distilled water. The above solution is added to the ZnCl₂ solution and stirrer for 1 hour. After few hours the white precipitate was obtained. The pure

ZnS nanopowder was prepared. Similarly, the same process for FeCl₂ doped ZnS nanoparticles were carried out. Now, the FeCl₂ doped ZnS were synthesized by 4M of FeCl₂ with 50ml of distilled water allowed to stirrer for 1 hour and the obtained solution is added drop by drop to the pure ZnS solution. Finally, the brown color precipitate was obtained. The obtained solution was filtered and dried in hot, oven at 100°C for 3 days. The FeCl₃ doped ZnS and pure ZnS nanoparticles were synthesized.

3. RESULTS AND DISCUSSIONS

3.1 X-ray diffraction:

The crystallinity of the synthesized nanopowder was investigated by XRD pattern. The XRD pattern of FeCl₃ doped ZnS and undoped of ZnS shown respectively. The straight line and sharp peaks shows that the synthesized powders were nano crystalline in nature.^[5] The crystalline sizes of the FeCl₃ doped ZnS were calculated by means of an X-ray line broadening method using the Scherrer equation:

$$\text{Debye Scherrer formula: } D = k\lambda / \beta \cos\theta$$

Where, D =Crystalline size in nanometer

λ =Wavelength of the radiation

k = Constant equal to 0.94

β = Full width at half maximum (FWHM) Intensity

θ =Peak radiations

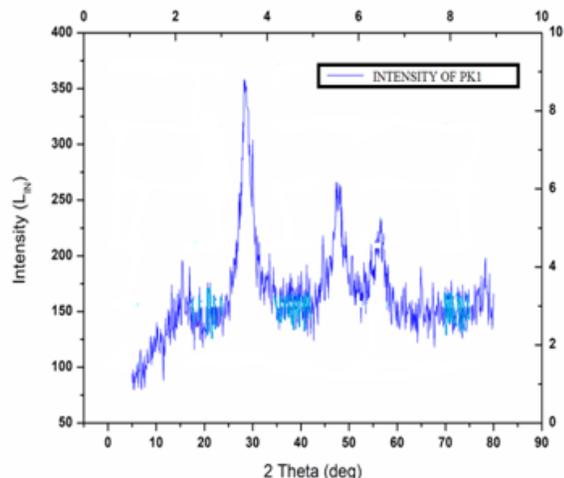


Fig.1: XRD Analysis of Undoped ZnS Nanoparticles

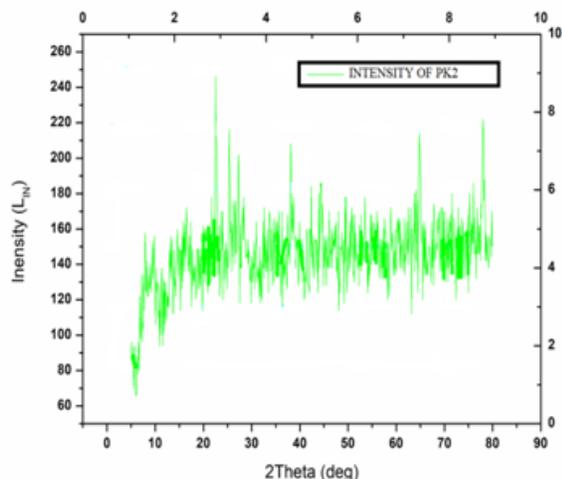


Fig. 2: XRD Analysis of FeCl₃ Doped ZnS Nanoparticles

The calculated average crystallite size of undoped ZnS concentration increased in crystallite size D , becomes increased due to size effect and the peak broaden becomes decreased with sharp peak intensity for FeCl₃ doped ZnS. The various parameters of XRD pattern are tabulated.

3.2 FTIR analysis

The FTIR spectrum of FeCl₃ doped ZnS nanoparticles synthesized by chemical precipitation method. The wavelength region was recorded in the range of 400-4000 cm⁻¹. The Peak at 3417.86 cm⁻¹ corresponds to the O-H Stretching present in the samples.^[7] The absorption peak absorbed at 1627.92 cm⁻¹ are corresponds to C=O Stretching

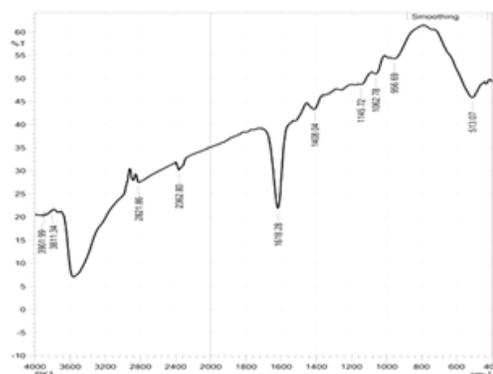


Fig. 2.1: FTIR Spectrum of Undoped ZnS Nanoparticles

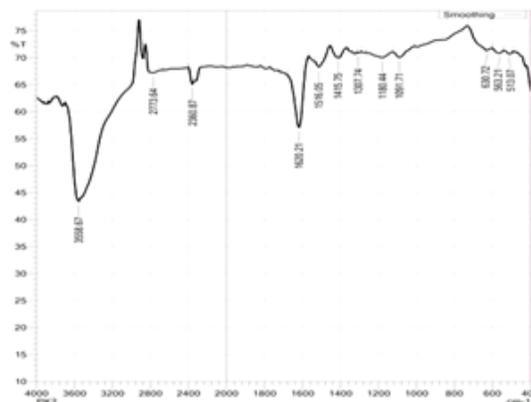


Fig. 2.2: FTIR Spectrum Of FeCl₃ Doped ZnS Nanoparticles

Table1: Structural parameters of ZnS Nanoparticles

Sample Name	2θ (deg)	Interplanar Spacing d(Å)	FWHM (deg)	Crystallite Size D (10 ⁻⁹) nm	Dislocation Density (10 ⁻³)	Micro Strain (10 ⁻³)
Undoped ZnS	28.6458	0.9689	56.8392	2.6295	144.6218	13.7681
	47.8000	0.9142	52.3300	3.0269	109.1439	11.9607
	56.1916	0.8821	53.7864	3.0520	107.3510	11.8620
FeCl ₃ doped ZnS	22.6003	0.9806	7.3772	0.2001	2.4954	1.8086
	64.8255	0.8442	6.0881	2.8176	1.2595	1.2849
	77.9149	0.7776	7.8848	2.3618	1.7925	1.5328

Vibration present in the samples. The absorption peak absorbed at 1089.78 cm^{-1} corresponds to C-O-C Stretching. The appearance of two peaks assigned at 1618.28 cm^{-1} and 3885.67 cm^{-1} are shown

3.3 UV Analysis

The optical properties of FeCl_3 doped ZnS and undoped ZnS nanoparticles are characterized by UV-visible spectrum. The UV absorption spectrum of FeCl_3 doped ZnS and undoped ZnS shown in the figure respectively. The optical absorption and transmission of the samples were recorded in the range of 200-1200 nm respectively. The band gap energy can be calculated using the formula.

$$E = h \cdot c / \lambda_{\text{max}}$$

Where, h = Planck's constant ($6.626 \times 10^{-34}\text{ J sec}$)

c = Wavelength of light ($3 \times 10^8\text{ m/s}$)

λ_{max} = Maximum wavelength (m)

The standard band gap energy was 3.5 which were decreased of undoped ZnS as 1.21 and FeCl_3 doped ZnS as 1.14. The absorption edge shift towards the lower value of

wavelength while increase in concentration of FeCl_3 . It is clear that the band gap increases with doping concentration. From the UV-Visible spectroscopy it was observed that the absorbance of ZnS nanoparticles decreases with increase in wavelength and while the transmission of ZnS nanoparticles, increase with decrease in wavelength. Because of good transparency, ZnS nanoparticles can be used as dielectric filter. The calculated band gap energy for the sample is tabulated.

3.4 SEM Analysis

The morphology of the prepared nanoparticles are examined using SEM. Nano-sized ZnS grains were observed in the SEM image of the prepared sample, having large surface area and the images are shown in the figure. The closure examination of FeCl_3 doped ZnS and undoped ZnS images reveal a well defined particle like cauliflower structure.^[8]

Table 2: Optical parameters of ZnS Nanoparticles

Samples	Absorptions	Band gap in eV
Undoped ZnS	0.042	1.218
FeCl_3 Doped ZnS	0.063	1.148

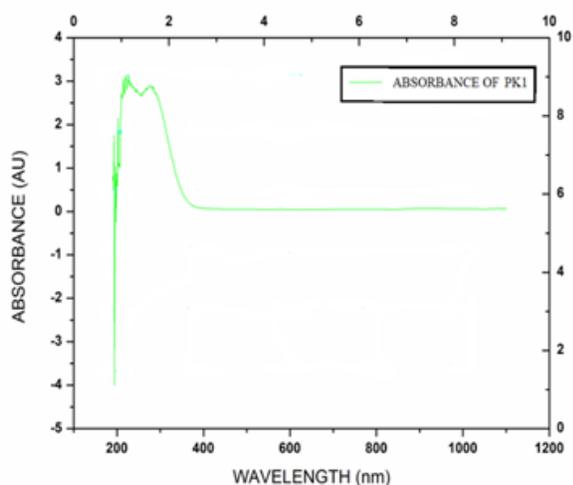


Fig. 3.1: Absorbance Spectra of Undoped ZnS Nanoparticles

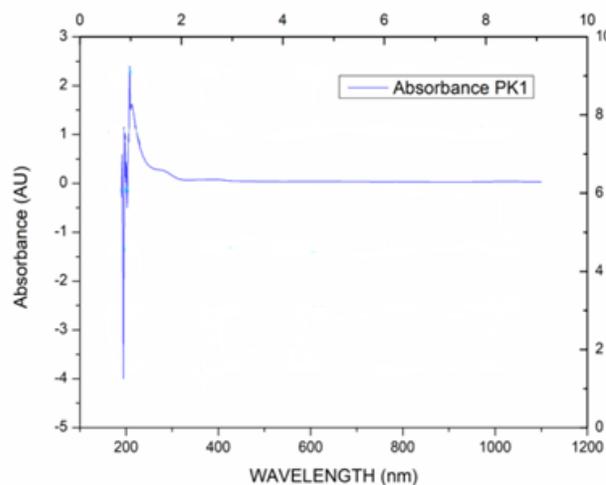


Fig. 3.2: Absorbance Spectra of FeCl_3 Doped ZnS Nanoparticles

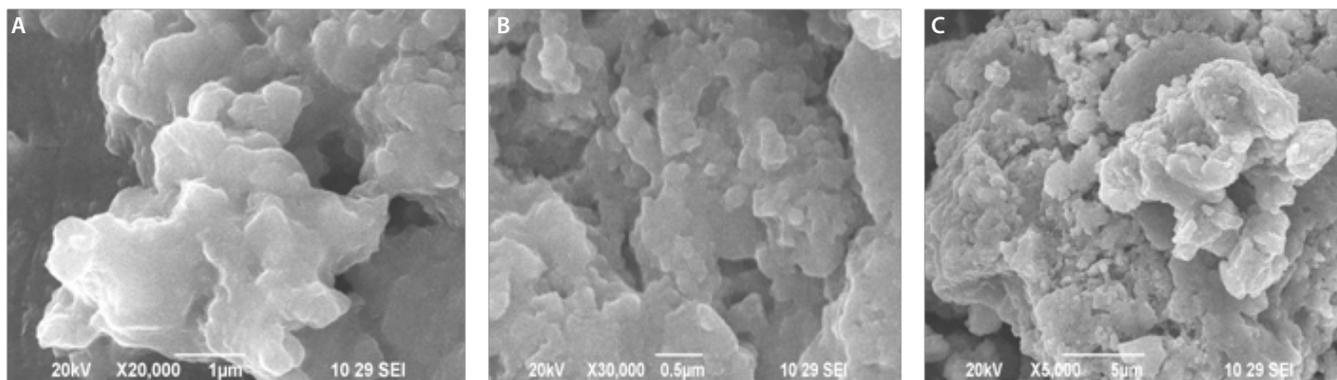


Fig. 4.1 (A to C): SEM Images of Undoped ZnS Nanoparticles

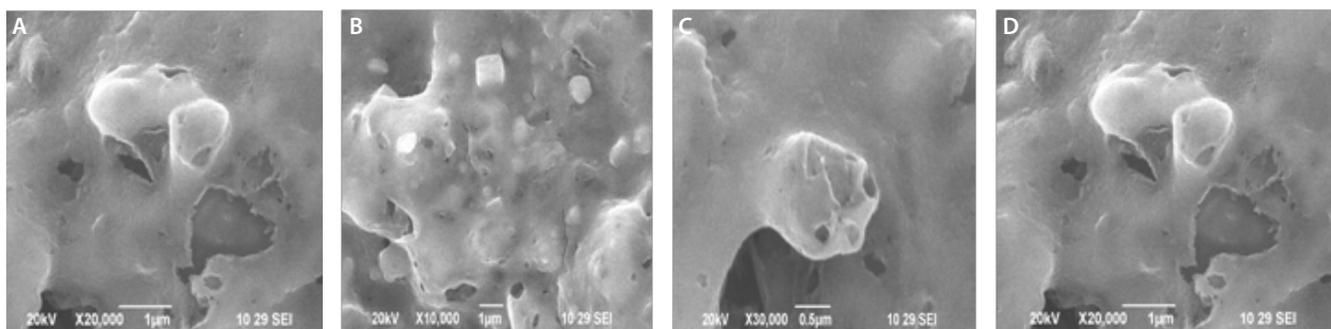


Fig. 4.2 (to D): SEM Images FeCl₃ Doped ZnS Of Nanoparticles

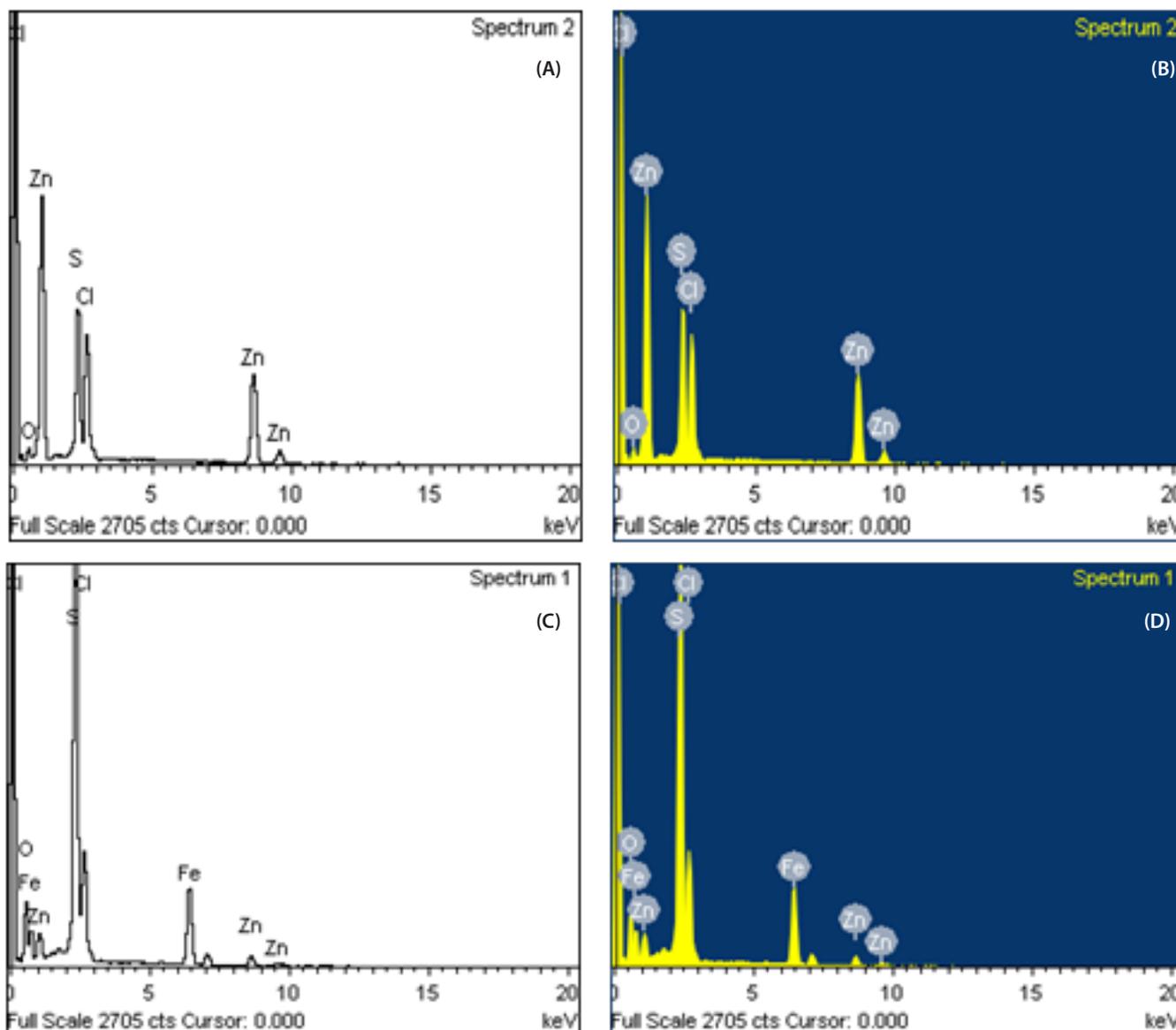


Fig. 5 (A and B): Undoped ZnS Nanoparticles; (C and D) FeCl₃ Doped ZnS Nanoparticles

3.5 EDAX analysis

The composition of elements presents in the nanoparticles is examined using EDAX. Nano-sized ZnS compositions of a specimen are observed and the images

are shown in the figure. The closure examination of FeCl₃ doped ZnS and undoped ZnS nanoparticles confirm the composition, distribution and elemental mapping of the sample.

4. CONCLUSION

The undoped ZnS and FeCl₃ doped ZnS have been prepared by chemical precipitation method. XRD analysis suggests that the average particle size is in the nano range. The FTIR spectroscopy examines the functional group analysis present in the sample. The band width of the absorption peaks are calculated by using the UV-Spectrum. SEM images confirm the cauliflower structure present in the nanoparticles. EDAX shows the composition of elements present in the nanoparticles. ZnS is used in the applications of white pigment, X-ray screens and cathode ray tubes.

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