

SYNTHESIS OF ZNS NANOPARTICLES VIA CO- PRECIPITATION METHOD

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Abstract :

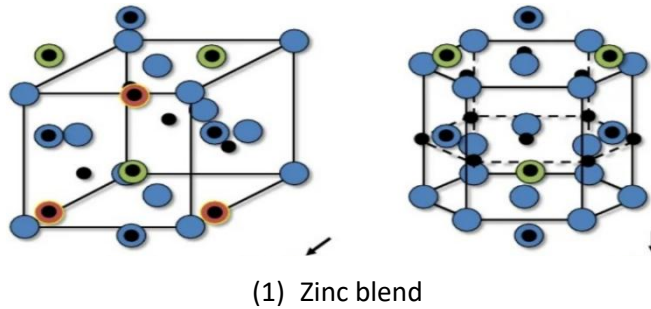
Zinc sulphide (ZnS) nanoparticles have gained considerable attention due to their wide band gap and potential application in optoelectronic and photonic devices. The objective of this work was to prepare ZnS nanoparticles by a simple co-precipitation method using zinc acetate dihydrate as precursor and sodium sulphide flakes as sulphur source. The precipitate was separated and washed by centrifuging. The synthesised ZnS nanoparticles were characterised by X-ray diffraction (XRD), the crystalline size of ZnS NPs is found to be 2.55nm and Fourier-transform infrared (FTIR) techniques.

Keywords : ZnS nanoparticles, Co-precipitation, XRD

Introduction :

Nanotechnology is one of the most revolutionary scientific fields of the 21st century. At its core, it is the science of manipulating matter at the nanoscale. The National Science and Technology Council (NCST), states that any material which measures between the range of 1 and 100 nm in at least one of its dimensions is considered a nanomaterial. Zinc sulphide nanomaterial is non-toxic material that is more stable than other semiconductors and is characteristics by a wide band gap energy of ~ 3.7 eV.[1] The nanostructured semiconductor materials having unique properties owing to large number of the surface atoms and that are three-dimensional confinement of a electrons in the crystal structure.[2]ZnS is important compound within the family of II-VI group semiconductor having a wide energy band gap (EG). ZnS has two stable crystalline structures, one is zinc blend (cubic) and another one is wurtzite (hexagonal). In both of the forms the co-ordination geometry at Zn and S is tetrahedral. The energy gap of the wurtzite ZnS (3.79 eV) is larger than that of the cubic (3.68eV).[3] Therefore, the Zinc sulphide nanoparticles is potentially useful in many applications, specifically in electronics and optoelectronics such as devices like optical sensors, electroluminescence devices, quantum dot-sensitized solar cells, and in lasers.[4]





For the synthesis of ZnS nanoparticles co-precipitation method was chosen due to its simplicity, cost-effectiveness, low-temperature processing, ability to control particle size and optical properties.

Material And Synthesis Method :

1. Material used :

To synthesize ZnS nanoparticles (NPs) we used Zinc Acetate Dihydrated [$\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$], 98% extra pure, MW:219.50 (Loba Chemie Pvt. Ltd.) and Sodium Sulphide flakes, iron free 60% extra pure [$\text{Na}_2\text{S} \cdot \text{H}_2\text{O}$], MW:78.04 (Loba Chemie Pvt. Ltd.). These all precursor using without further purification. All of this solution were prepared using double distilled water (DI water) as solvent. DI water and ethanol used for washing the sample (to remove ion impurities). All the synthesis procedure was cried out at room temperature.

Synthesis of ZnS NPs :

ZnS NPs were prepared via coprecipitation method: for ZnS nanoparticle synthesis, the appropriate amount of (10mmol) of [$\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$] was prepared in 100ml of DI water (S1) and stirred it for 30 min. Sodium Sulphide (15mmol) was dissolved separately in 100ml of double distilled water (S2) and stirred for 30 min. then (S2) is added drop wise into (S1) under vigorous magnetic stirring. That mixture was stirred continuously for 3 hours. The white precipitated formed in resulting solution (S0) that was left for 24 hours. Allowed the formed particles to settle down at bottom of the beaker. The precipitated ZnS NPs were centrifuge. The centrifuge product was washed by several times by distilled water and ethanol to remove impurities and loosely bonded ions and then collected. The collected sample was dried at the room temperature. Thus obtained material was lastly used for further characterizations.

Characterization :

The ZnS nanoparticle samples were investigated by using XRD and FTIR techniques. The Crystallite size of ZnS nanoparticle were investigated by using the X-ray diffraction method (XRD) (Rigaku miniflex 600) and Fourier Transform Infrared Radiation (FTIR) spectra were obtained using IR Affinity-1s from 400 to 4000 cm^{-2} using the KBr pellet technique.



Result and discussion :

X-ray diffraction studies :

Figure (a) shows the X-ray diffraction (XRD) pattern of the prepared nanoparticles measured in the scanning range 0° to 90°. ZnS nanoparticles show various diffraction peaks for 2θ values of 28.97°, 48.12° and 56.59° corresponding to the plane indexed as (111), (220) and (311) respectively, further validating the presence of the Wurtzite-6H ZnS structure. The Debye-Sherrer equation was used to estimate the crystallite size, The Debye-Sherrer equation is -

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

Where, k is the constant having value 0.94, wavelength of the X-ray radiation, $\lambda_{CuK\alpha}=1.5406\text{\AA}$, β is the full width at half maximum (FWHM) measured in radian (converting the degree value to radian), θ is the diffracted Bragg angle,

The calculated ZnS nanoparticle size was 2.55nm corresponding to plane (1 1 1). The broadening of the XRD peaks indicates the nanocrystalline nature of the samples.[5]

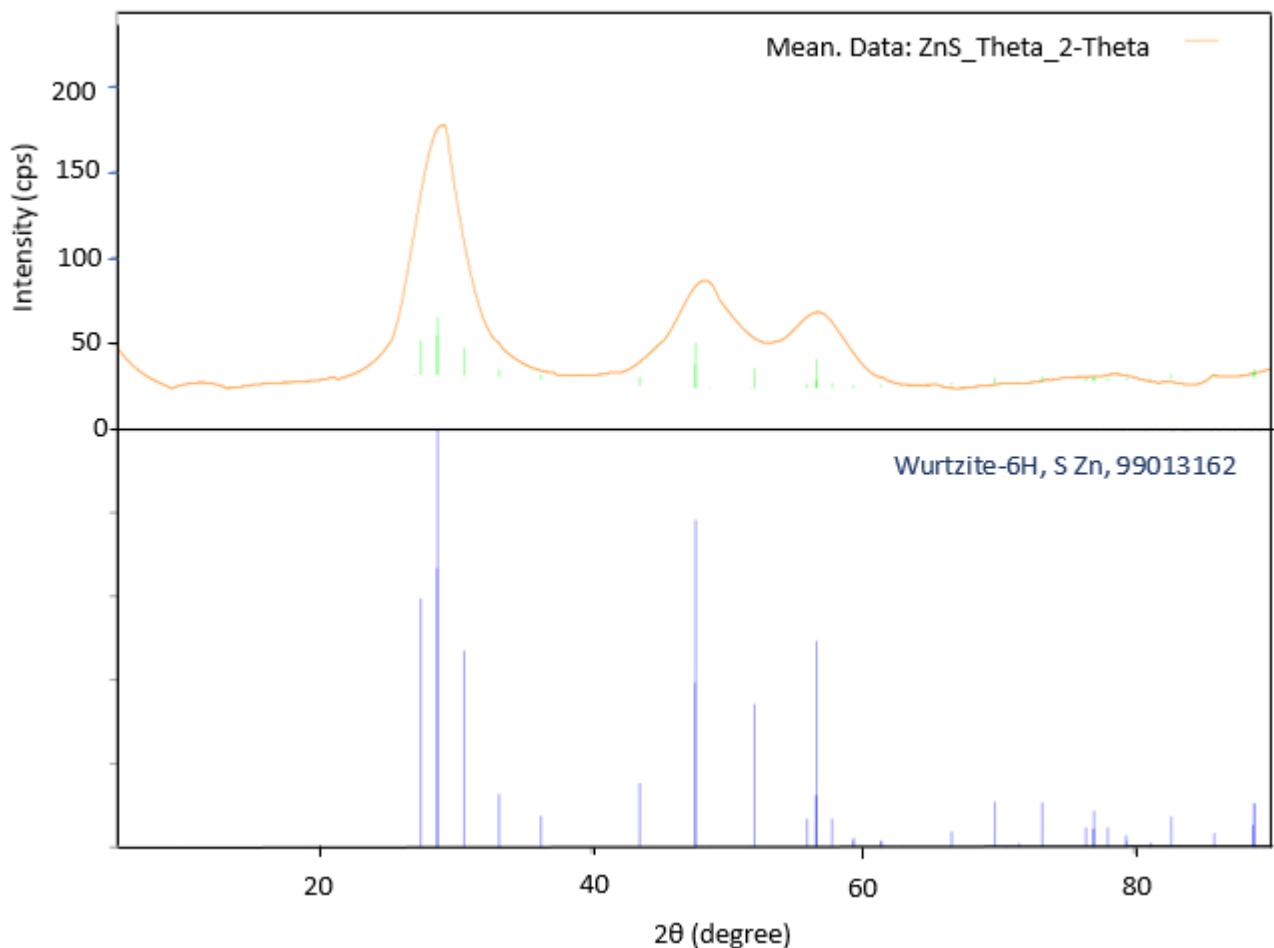


Figure (a): the X-ray diffraction (XRD)

Fourier Transform Infrared Radiation (FTIR) :

FTIR characterization helps confirm the presence of Zn–S bonds, the low-frequency region, where the peaks at 490.90 cm^{-1} and 428.22 cm^{-1} correspond to Zn–S stretching vibrations, directly confirming the presence of zinc sulphide bonds. In addition, the broad absorption around 3440 cm^{-1} is consistent with O–H stretching, which is commonly observed in ZnS nanoparticles due to adsorbed water or hydroxyl groups on their surface. The band near 1624 cm^{-1} can be attributed to H–O–H bending vibrations, further supporting the presence of surface-bound water molecules. Another peak at 658.72 cm^{-1} may also be related to lattice vibrations or mixed skeletal modes sometimes reported in ZnS nanoparticle spectra. These features together indicate that your sample does contain ZnS nanoparticles,

The peak at 2347 cm^{-1} , for example, is due to atmospheric CO_2 , the absorptions at 2255 cm^{-1} and 2093 cm^{-1} , linked to nitrile and alkyne groups, suggest the presence of organic residues or stabilizers from synthesis; these can actually act as capping agents, helping to control particle size and prevent aggregation. Similarly, the band at 1561 cm^{-1} , associated with nitro groups, and the one at 1417 cm^{-1} , related to C–H or O–H bending, point to organic molecules that may improve compatibility with solvents or other materials. The peaks at 1129 cm^{-1} and 1017 cm^{-1} , corresponding to C–O and C–N stretches, are often connected to surface modifiers that enhance dispersibility and functionalization.

Peak at 901.75 cm^{-1} , This absorption band is typically associated with Zn–S stretching vibrations in zinc sulphide. Its presence confirms the formation of Zn–S bonds and validates the crystalline ZnS phase. Peak at 3953.28 cm^{-1} , This high-frequency band lies in the region characteristic of O–H stretching vibrations. It is most likely due to adsorbed water

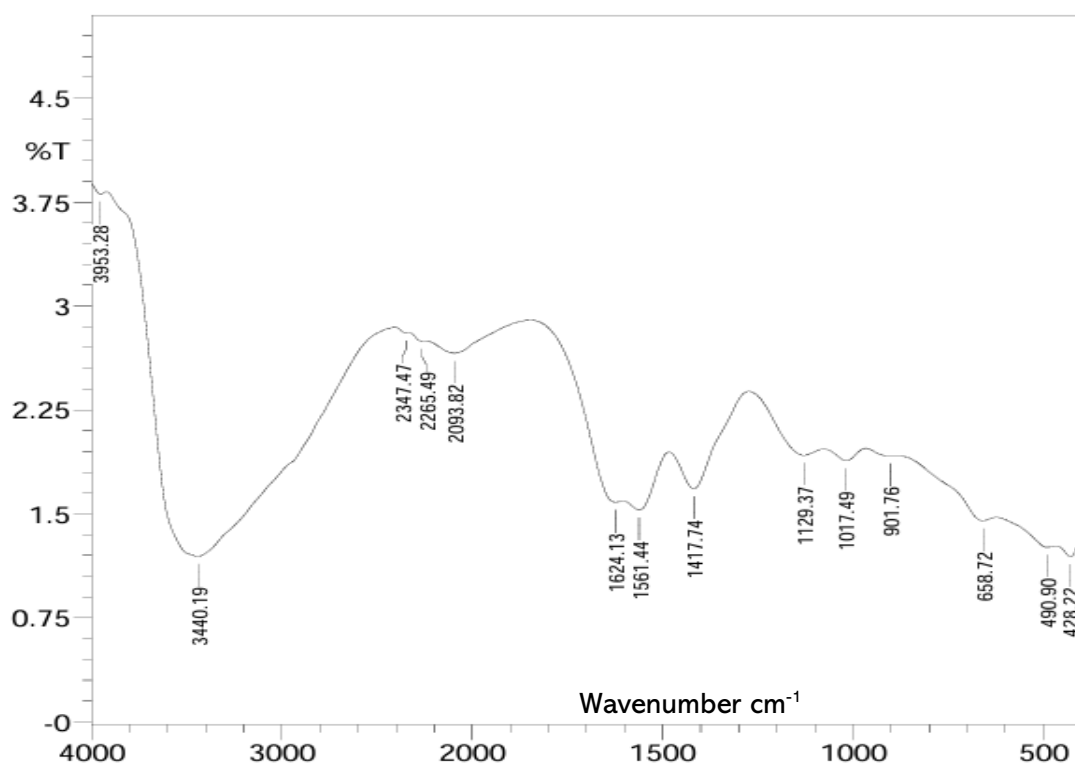


Fig (b): FTIR peak of ZnS Nanoparticles



molecules or residual hydroxyl groups on the surface of the ZnS sample.

Conclusion :

ZnS nanoparticles were successfully synthesized via chemical co-precipitation method. which proved to be a simple, cost-effective, and efficient technique for nanoparticle preparation. The X-ray diffraction (XRD) analysis confirmed the crystalline nature of the synthesized ZnS nanoparticles. The diffraction peaks matched well with the standard ZnS crystal structure, indicating the formation of a pure phase without significant impurities. The crystalline size of ZnS NPs is found to be 2.5 nm .The Fourier Transform Infrared (FTIR) spectroscopy analysis provided information about the chemical bonding and surface functional groups. The presence of characteristic absorption bands corresponding to Zn–S stretching vibrations confirmed the formation of ZnS nanoparticles. Additional peaks observed in the FTIR spectrum were attributed to residual solvents, moisture, or stabilizing agents used during synthesis.

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