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Synthesis, Characterization of Pure ZnS Nano Particles Prepared Via Chemical Co-Precipitation Method

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Abstract: In this research pure Zinc Sulfide (ZnS) nanoparticles and nano composite powders were prepared by homogeneous chemical co precipitation method using zinc acetate and various sulfur sources. The ZnS nanoparticles were characterized by X-ray Diffraction, Scanning Electron Microscopy (SEM), and Ultraviolet-Visible absorption spectrum. The structure of nanoparticles was studied using X-ray Diffraction pattern. The crystallite size of ZnS nanoparticles was calculated by Debye-Scherrer formula. Morphology of nanoparticles was observed and investigated using the Scanning Electron Microscopy. The grain size of Zinc Sulfide nanoparticles were in suitable agreement with the crystalline size calculated by X-ray diffraction results. The optical properties of particles were studied with Ultraviolet-Visible Spectroscopy.

Keywords: Co precipitation method, Nano Particles, Zinc Sulfide, UV-VIS, SEM Analysis

I. INTRODUCTION

Nanotechnology has attracted global attention because the nano materials have properties unique from their bulk equivalents. Among the various classes of nonmaterial, semiconductor ZnS is witnessing extreme attention due to its interesting properties and applications. Zinc sulfide (ZnS) is one of the first semiconductors discovered and it has traditionally shown remarkable fundamental properties of versatility and a promise for novel diverse applications, including Light-Emitting Diodes (LEDs) [1], Electroluminescence [2], Flat Panel Displays, Infrared Windows[3], Sensors, Lasers, and Bio Devices[4]. ZnS crystallizes in the zinc-blende (ZB or sphalerite) or wurtzite (WZ) structures at room conditions with a band gap of 3.68 eV or 3.77 eV respectively. ZnS nano particles have been synthesized using several methods like Homogeneous Precipitation[5-8], Microwave Methods, Thermal Evaporation, Pulsed Laser Deposition[9], Spray Paralysis[10,11] and Solution- Gelation (sol-gel) [12]. So far, extensive efforts have been made on the synthesis of low dimensional ZnS nanostructures, including nanoparticles, nanowires, nano belts, nanocables, and nanotubes.

II. EXPERIMENTAL DETAILS

A. Materials

Nano sized particles of ZnS crystals were prepared by chemical co precipitation reaction using analytical reagent grade chemicals, Zinc Acetate Dehydrate ($\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$), Sodium Sulfide ($\text{Na}_2\text{S} \cdot \text{H}_2\text{O}$), Polyvinyl Pyrrolidone (PVP ($\text{C}_2\text{H}_5\text{NO}$)_n) as source materials. Deionized water is used throughout the entire preparation process.

B. Synthesis

For the synthesis of pure zinc sulfide nanoparticles, the following procedure was used. 0.3 mol of zinc acetate and 0.032 gms of PVP (capping ligand)[13] were dissolved in 50 ml of de ionized water under continuous stirring. After that 0.6 mol of sodium sulfide is dissolved in 50 ml of de ionized water. The Na_2S solution was then poured drop by drop in to zinc acetate and PVP solution[14] under continuous stirring. After the reaction was completed, a white precipitate was formed. The mixture was centrifuged at 300 rpm for 30 minutes and dried at 120 °C for about 1 hour in hot air oven.

III. CHARACTERIZATION

The phase purity and crystal structure of as prepared pure ZnS nanoparticles were carried out by analyzing the X-Ray Diffraction (XRD) patterns using monochromatic Cu- $K\alpha$ radiation in 2θ range of 10° to 80° with a X' Pert PRO Diffractometer (PANalytical, Netherlands) running under the continuous scanning mode in steps of 0.050° [15-17]. A JEOL-Scanning Electron Microscopy (SEM) (Model JSM – 6390, Made in JAPAN) was used to record the micrograph for the samples of pure ZnS nanoparticles. Optical absorption studies were carried out using a UV-Visible Spectrometer (JASCO V- 570, CANADA Make) in the range of 200 - 1200 nm [18]. The elemental compositions of pure ZnS nanoparticles were analyzed and the characteristic elements were identified by using EDAX.

IV. RESULTS & DISCUSSION

A. XRD Analysis

XRD analysis were performed to determine the crystalline structure and phase formation of zinc sulphide nanoparticles. . Figure 1. represents the XRD pattern for the sample (1:2 molar ratio of Zn and sulfur source). From the XRD results it is confirmed that the pure ZnS material has a Rhombohedral structure. The XRD pattern of pure ZnS nanoparticles show 3 distinct peaks at $2\theta = 13.14^\circ$, 31.38° , and 54.50° which corresponds to the (0039), (0147) and 0273 planes and in good agreement with standard JCPDS Card No. 89-2176.

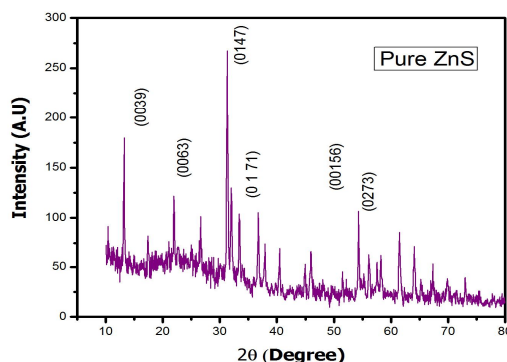


Figure 1. XRD Patterns of Pure ZnS

Average crystallite size of pure ZnS nanoparticles are calculated using the Debye-Scherrer's equation

$$D = K \lambda / \beta \cos \theta \quad (1)$$

where, D is the mean grain size, K is constant, λ is the X-ray's wavelength (1.5406 \AA), β is the full width at half maximum and θ is the Bragg's diffraction angle. The crystalline size of ZnS nanoparticles calculated about 36 nanometers by Debye-Scherrer equation.

B. SEM Analysis

The surface morphology of synthesized pure ZnS nanoparticles has been studied by using SEM. The morphologies are noticeably dependent on the preparation procedure and crystal composition. SEM images of pure ZnS nanoparticles are shown in Fig. 2. Microscopic images reveals the rose flower petals like ZnS nanoparticles.

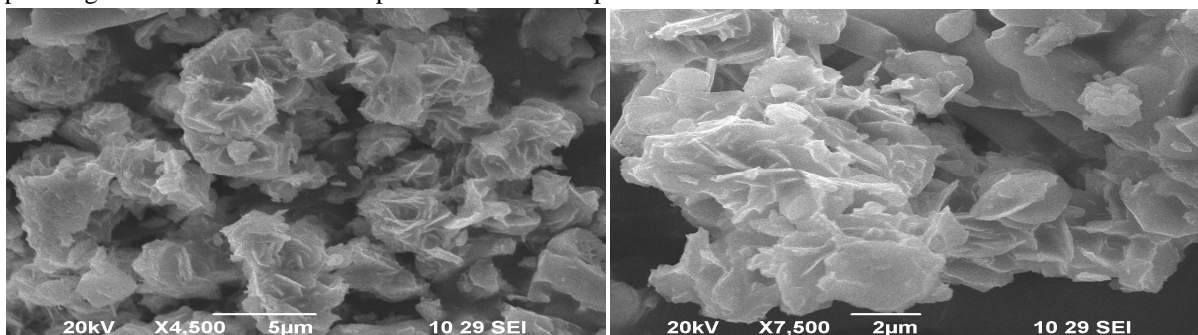


Figure2. SEM Images of Pure ZnS

C. EDAX Analysis

Chemical composition analysis of all samples was done by EDAX technique. The Energy Dispersive X-ray (EDAX) analysis was used to determine the percentage of zinc and sulfur present in the ZnS sample. Energy dispersive X-ray analysis gives both qualitative and quantitative information about the elemental composition of the materials. From the EDX spectra, we can conclude that there are other materials, such as impurities or adducts, present in the samples. These impurities occur either accidentally with the reagent molecules or are added for modification of the basic materials. Fig.3 shows EDAX spectra of Pure ZnS. From the EDAX spectra of the samples it is confirmed that the amount of Zn, S were close to the nominal (target) values and it is intervened that the as prepared ZnS nano particles are highly pure.

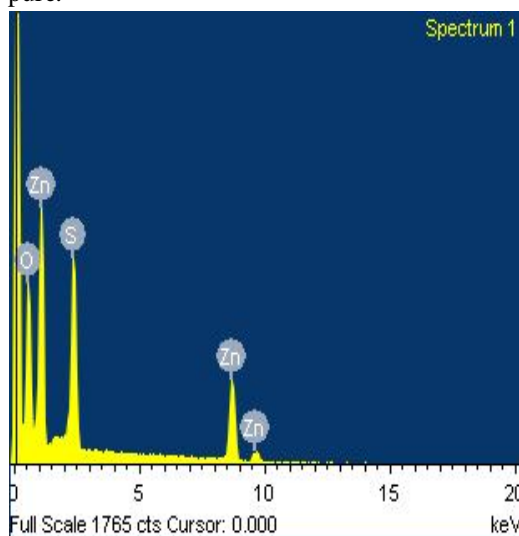


Figure 3. EDAX of Pure ZnS

D. UV-Visible Optical absorbance and Band gap

The UV-Visible optical absorption spectrum of the samples recorded in room temperature. The optical band gap E_g can be estimated from the Tauc plot [17]. For crystalline semiconductors, n can take values 1/2, 3/2, 2 or 3 depending on whether the transitions are direct allowed, direct forbidden, indirect allowed and indirect forbidden transitions respectively [18]. Optical absorption spectra of samples using UV Vis are indicated in Fig.4. The optical band gap of pure ZnS nano particle is found to be 1.72 eV [19-20].

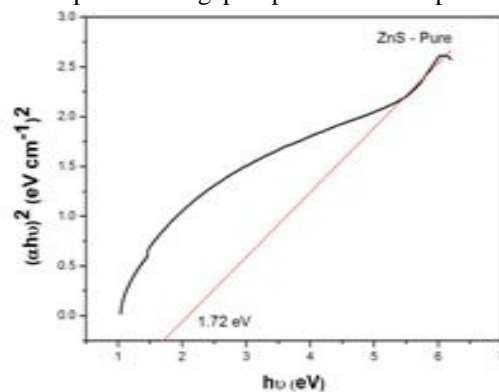


Figure4. Tauc Plot of UV Vis absorption spectra and Optical Band gap of Pure ZnS Nanoparticles

V. CONCLUSION

In this research study, ZnS nanoparticles were successfully synthesized via the chemical co-precipitation method using zinc acetate. The structural properties of pure ZnS nanoparticles were characterized by X-ray diffraction (XRD) to identify crystal structure. Crystallographic and morphological studies reveal the zinc blende nanoparticles having average crystallite size of approx 36nm. The SEM image reveals that the ZnS nanoparticles have regular shapes. EDAX analysis confirms the presence of Zinc and Sulphur. From the UV-Visible absorbance spectrum the optical band gap of pure ZnS nanoparticles is found to be 1.72 eV

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