

e-ISSN:2582-7219



# INTERNATIONAL JOURNAL OF MULTIDISCIPLINARY RESEARCH IN SCIENCE, ENGINEERING AND TECHNOLOGY

Volume 6, Issue 12, December 2023



INTERNATIONAL  
STANDARD  
SERIAL  
NUMBER  
INDIA

Impact Factor: 7.54



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# Synthesis, Characterization and Application of Zinc Sulphide Nanoparticles

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**ABSTRACT:** Zinc sulphide (ZnS) nanoparticles have been synthesized by the mechanochemical route. The prepared nanoparticles have been analyzed by X-ray diffraction (XRD), Field emission scanning electron microscope (FESEM), Energy-dispersive analysis of X-rays (EDAX), Ultraviolet–visible–near infrared (UV–Vis–NIR) spectrophotometer and photoluminescences (PLs) spectrophotometer. The crystallite size of as prepared nanoparticles are found to be in the 4–7 nm range. EDAX spectrum confirmed the composition of ZnS sample. The value of optical band gap has been found to be in the range 4.04–4.6 eV. Room temperature photoluminescence (PL) spectrum of the samples exhibits a blue light emission under UV excitation wavelength of 280 nm. Thermal analysis studied with the help of Differential thermal analysis (DTA), Differential thermogravimetric (DTG) and thermogravimetric analysis (TGA).

**KEYWORDS-** Zinc sulphide, nanoparticles, characterization, application, spectrophotometer

## I. INTRODUCTION

### Synthesis

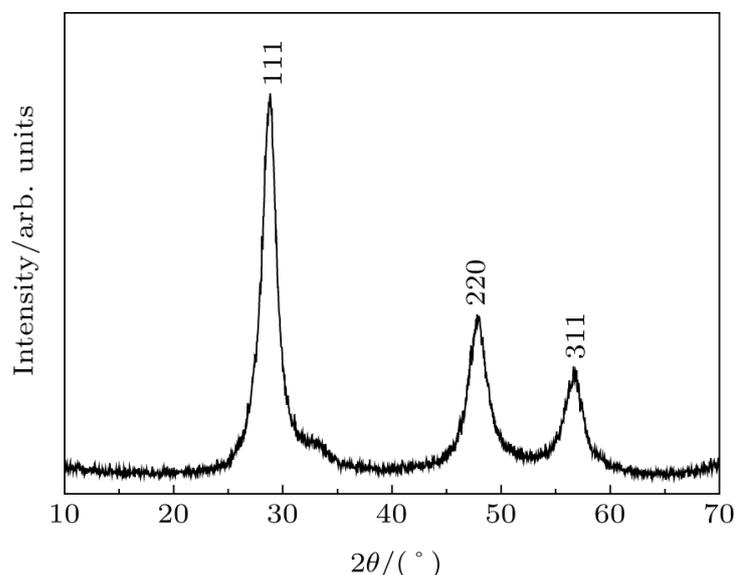
Nano-sized particles of ZnS crystals were prepared by co-precipitation reaction using zinc acetate ( $\text{Zn}(\text{Ace})_2 \cdot 2\text{H}_2\text{O}$ ), sodium sulfide ( $\text{Na}_2\text{S}$ ), and EDTA (Merck, purity > 98%) as source materials. EDTA acted as the capping ligand. All of the reagents were analytical grade in purity and used without further purification, and the deionized water was used as a solvent. The ZnS nanoparticles were prepared in the following sequence: 0.005-mol  $\text{Zn}(\text{Ace})_2 \cdot 2\text{H}_2\text{O}$  and EDTA were dissolved in 50 ml of deionized water under continuous stirring, after that the desired amount of  $\text{NH}_4\text{OH}$  was used to adjust the PH of the solution to 7 to form clear liquid. Then, 0.1-mol  $\text{Na}_2\text{S}$  was added to 50 ml of deionized water. The  $\text{Na}_2\text{S}$  solution was then poured into  $\text{Zn}(\text{Ace})_2 \cdot 2\text{H}_2\text{O}$  and EDTA solution. After the reaction was completed, a white precipitate formed. Then this mixture was centrifuged at 300 rpm for 30 min and dried at 110 °C for 24 h in air.[1,2,3]

### Characterization

Structural characterization and phase identification of nanocrystalline powder were carried out using XRD experiments with monochromatic Cu-  $K_\alpha$  radiation ( $\lambda = 1.54056 \text{ \AA}$ ) in a  $2\theta$  range of  $10^\circ - 70^\circ$  in a continuous scan mode in steps of  $0.04^\circ$  to determine the unit cell parameters of the sample under study. Sample morphology was also examined by SEM.

The FTIR analysis of the sample was recorded in a range from  $400 \text{ cm}^{-1}$  to  $4000 \text{ cm}^{-1}$ . Potassium bromide (KBr) acting as non-absorbing medium was mixed with solid sample (0.3 wt%– 0.5 wt%) by an agate mortar and pestle to prepare a disk specimen. The UV radiation was performed using a PG Instrument + T80 UV-Vis spectrometer in a wavelength range of 250 nm– 500 nm to obtain the energy of band gap. The PL spectrum of the as-synthesized ZnS nanoparticles was carried out by PL spectrophotometer to investigate the luminescence properties.

The analysis of x-ray pattern shows that ZnS sample crystallize in cubic symmetry (F-43M space group) with lattice parameter  $a = 5.368(4) \text{ \AA}$  and unit cell volume  $V = 154.7157(8) \text{ \AA}^3$ . The three different peaks of the sample correspond to the lattice planes of (111), (220), and (311), which match very well with the cubic zinc blende structure (JCPDS No. 05-0566).[4,5,6]



The significant broadening of the XRD pattern peaks reveals the small particle (crystallite) size and indicates the nanometric particle size.<sup>[17]</sup> The average particle size is approximately estimated by Hall's method<sup>[18]</sup>

## II. DISCUSSION

Semiconductor nanocrystals and nanocomposites have attracted great fundamental and technical interest because of their unique optical, mechanical and electronic properties, such as photoluminescence, continuous absorption and, microhardness, strong fluorescence, broad photo excitation [1]. Various inorganic-polymer nanocomposites composed of different combinations of two or more components have attracted progressive attention in today's world due to their interesting physical properties and applications. [2]. The inorganic/polymer nanocomposites, metal sulfides/polymer nanocomposites have been researched extensively due to their interesting optical, electrical and mechanical properties. Their excellent physical and chemical properties in various fields, such as catalysis, sensors, solar cells, photo detectors, light emitting diodes and laser communication, have made them very attractive and promising materials [3]. Polyaniline (PANI) being a conducting polymer with an extended - conjugated electron system, proved to be a promising candidate due to its high absorption coefficients in visible-light range and high mobility of charge carriers. Polymers have many applications and uses but are often limited by their inherent physical properties. Most polymers have very low thermal conductivity. To make the polymer viable for potential devices the thermal conductivity of the polymer should be high. For achieving this, fillers with higher thermal conductivity can be incorporated into polymers to form polymer composites [4,7,8,9]. ZnS is one of the important materials with wide range of applications such as light emitting devices, laser devices, cathode ray tubes, nonlinear optical devices and antireflecting coatings [5].

Zinc sulphide, a direct wide band gap transparent semiconductor, is one of the most important materials used in photonics research [6]. It is a potential candidate for variety of applications such as electroluminescent devices, solar cells and optoelectronic devices. ZnS can crystallise in two structures, the Zinc blended or the wurtzite structures. Doping on ZnS with transitional metal ions like  $Mn^{2+}$  and  $Cu^{2+}$  is an important aspect to yield different nanostructures. In polymer nanocomposites, the blends consist of organic polymer matrix and inorganic components (semiconductors). PMMA, PVA, and PVP are among the most widely used polymers in the synthesis of nanocomposites because of their availability and low cost [7]. In recent years, polyaniline (PANI) has occupied an important place in the family of conducting polymers due to its good stability in air, simplicity in doping, improved electronic properties and high conductivity in the doped state [8]. The properties of nanocomposite materials depend on the properties of the individual components and also on the microstructure and interfacial characteristics.

Polyaniline has got much importance due to its unique electrical, electrochemical properties, high environmental stability, easy polarization and low cost of monomer. Since the conductivity of PANI depends on both the oxidation states of the main polymer chain and the degree of protonation of the imine sites, any interaction with PANI that alters either of these processes will affect its conductivity. Composite of PANI with ZnS had six times higher conductivity than pure PANI. This is due to the interfacial interaction with ZnS matrix and enhancement in the crystallinity. Controlling crystallite size and doping with transition metals can tailor the optical properties of the nanocrystals, because of the stability, tenability and higher efficiency, polymer composites might present improved physical properties when compared to their polymer matrices counterparts. Polymer nanocomposites are the materials in which



nano scale inorganic particles are dispersed in an organic polymer matrix. When these particles are impregnated into a polymer matrix it enhances the stability of polymer while retaining its processing flexibility. The mixing of polymers and nanoparticles is opening pathways for engineering flexible composites that exhibit advantageous magnetic, electrical, optical, or mechanical properties [9]. Thus, new combination of materials can be manufactured to meet specific requirements, which are difficult to fulfill by using a single component [10,11,12].

The optical, electronic and thermodynamic properties of particles in the nano meter range often differ from those of bulk material due to quantum confinement [11]. Among the inorganic/polymer nanocomposites, metal sulfides/polymer nanocomposites have been researched extensively due to their interesting optical, electrical and mechanical properties when doped with iron and nickel [12].

### III. RESULTS

TEM is an important characterization tool for the direct imaging of nanomaterials to obtain quantitative measurement of particle size, size distribution, shape of nanoparticles and lattice fringes. The thickness and radius of ZnS nanoparticles are estimated using Gatan Software. Reduced FFT indicates the formation of highly crystalline nature of ZnS nanoparticles and clear occurrence of lattice fringes in ZnS nano structure. The appearance of dark and bright fringes confirms the synthesis of very fine crystalline ZnS nanoparticle via microwave radiations approach. The nanoparticles have been separated by well-defined boundaries, and are visible and uniformly distributed. TEM micrographs showed that ZnS nanoparticles were not aggregated in clusters but they were separated by equispaces which was verified by microscopy visualizing under the high resolution microscope.

Protein denaturation is a process in which proteins lose their tertiary structure and secondary structure by application of external stress or compound, such as strong acid or base, a concentrated inorganic salt, an organic solvent or heat. Most biological proteins lose their biological function when denatured. Denaturation of proteins is a well-documented cause of inflammation. • The samples studied were effective in inhibiting heat induced albumin denaturation upto certain extent. • The inhibition of zinc complex was 3.87% inhibition of albumin protein denaturation at 100 µg/ml compared to Aspirin as standard anti-inflammation drug at same concentration with maximum inhibition 70.7%. as compared to control.

ZnS nanoparticles were synthesised from microwave irradiation of thiosemicarbazone ligand as a single molecular processor : Pharmacological activities. During the interaction of Zn complex with microwave radiations colour of solution turned from an off white colour to a yellowish colour, indicating the formation of ZnS nanoparticles and this was also confirmed from UV –Visible spectra of ZnS nano particles. The absorption peaks at 340 and 378 nm in the UV-Visible spectroscopy results confirmed the synthesis of stable ZnS nano particles. Along with results of UV-Vis spectroscopy , FT-IR spectroscopy also confirmed the synthesis of ZnS NP's via microwave irradiation approach. The synthesised ZnS nanoparticles were found to be stable for up to 3 months without aggregation, due to the presence of various functional groups in Zn complex which acts as a chemical capping and reducing agents in the complex. The average particle size and crystalline nature of ZnS nanoparticles were found to be 60 nm, confirmed from TEM &XRD measurements. The microwave assisted synthesis of ZnS nanoparticles have effective anti- bacterial, antioxidant and anti-inflammatory efficacies as compared to standard substances. Finally microwave assisted ZnS nanoparticles may be consider as a stable and efficient drug candidate for the treatment of various diseases and they are nontoxic in nature.[13,14,15]

FTIR spectra of the *S. frutescens* plant and the SF-ZnS. The *S. frutescens* plant extract spectra exhibited vibrational stretching at 3400 cm<sup>-1</sup>, 2222 cm<sup>-1</sup>, 1017 cm<sup>-1</sup> and 1645 cm<sup>-1</sup>. The strong broad absorbance peak at 3400 cm<sup>-1</sup> is due to hydroxyl group (O–H) shifting, the small and sharp peaks at 1339 cm<sup>-1</sup> and 1017 cm<sup>-1</sup> are as a result of C–N and C–O stretching which might be due to amino groups [13]. The carboxylic group is responsible for the absorbance peak at 1645 cm<sup>-1</sup> which matches the C=O groups. These peaks were also observed and deposited on SF-ZnS. The Zn-S vibrational stretching was observed around 680 cm<sup>-1</sup> in the fingerprint region confirming the formation of ZnS nanostructures synthesized using *S. frutescens* plant. Since several phytochemicals have been identified from the plant extract, and these are used as reducing agents, the mechanism of formation can be proposed. The proposed mechanism for the formation of ZnS nanostructures using *S. frutescens* plant extract is as follows. Firstly, the phytochemicals such as flavonoids, alkaloids, glycosides and phenolic of *S. frutescens* plant extract bind and reduce the ZnCl<sub>2</sub> into Zn<sup>2+</sup> ions. Thereafter, the added Na<sub>2</sub>S.9H<sub>2</sub>O forms the S<sup>2-</sup> ions which bind to the zinc metal connected to the phytochemicals. Then nucleation and growth process occurs on the nanostructures thus the formation of ZnS nanostructures. The phytochemicals on the *S. frutescens* plant extract bound to the surface of the ZnS nanostructures prevented the agglomeration of the particles while stabilizing the nanostructures. Further, because the phytochemicals comprise of carbonyl groups that have the capacity to direct the shape, the capping of the ZnS nanostructures also



occurs. From the FTIR spectrum, the results reveal that the flavonol, canavanine and Gaba with the functional groups like hydroxyl, amino and carboxylic acids might have been absorbed on the surface of the ZnS nanostructures and this suggests that they played an important role during their formation. To further confirm the formation of this material, XRD analysis was conducted.

#### IV. CONCLUSION

Zinc sulphide (ZnS) nanoparticles are prepared by a simple, economic and green synthesis route. X-ray diffraction patterns confirm zinc blend structure. ZnS formation is confirmed through chemical analysis by energy dispersive analysis of X-rays. Transmission electron microscopy reveals formation of nanosize with dimension in the range of 8-22 nm. Band gap of the nanocrystals is found to lie in the range of 4.51-4.65 eV. Photoluminescence study indicate defect like vacancies. The growth mechanism of ZnS nanoparticles is discussed with the help of Fourier transform infrared spectroscopy and thermogravimetric analysis. The materials show high dielectric constant compared to its bulk counterpart. The dielectric loss of the samples shows anomalous behaviour. The frequency dependent A.C. conductivity of the samples is discussed both in high and low frequency regimes. Current-voltage (I-V) characteristic performed under dark and under illumination, shows excellent light response of the material.

A rapid, clean and simple method for preparation of zinc sulfide nanoparticles via electrodeposition without using any catalyst, surfactant, and template with possibility to control the size and shapes of the nanoparticles has been demonstrated. It was found that electrolysis voltage and sulfide ion concentration in the electrolyte solution are critical parameters for controlling morphology of the electrodeposited nanoparticles. Finally, the results of this investigation showed that electrodeposition is a simple, clean, green and relatively rapid for preparation of zinc sulfide nanoparticles. Furthermore, electrodeposition gives a better selectivity for controlling of the product morphology by changing electrolysis variables which is less tedious.[16]

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