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**STRUCTURAL, MORPHOLOGICAL, COMPOSITIONAL AND ELECTRICAL  
CONDUCTIVITY STUDY OF ZNS NANOPARTICLES PREPARED BY CHEMICAL CO  
PRECIPITATION METHOD**

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**ABSTRACT**

Zinc sulphide is one of the most promising materials for solar cells and of great interest for their practical applications in up to electronics and photonics. The physical and chemical properties of zinc sulphide nano particles are found to be size depended. In the present work describes preparation and characterization of zinc sulfide using chemical co precipitation techniques. A nano structured zinc sulfide is synthesized at room temperature. The crystallite sizes (hkl) of zinc sulfide nano particles were estimated from the peaks of XRD. FT IR spectral studies reveals the presence of functional groups. The optical properties of the prepared ZnS samples were estimated by UV- visible spectroscopy. Microstructural analysis using scanning electron microscope (SEM) supplemented with EDS were carried out for the sample to find grain size as well as chemical composition. AFM study shows the above prepared nano particle will have a particle size of 70 (nm). Electrical conductivity study exhibits the semiconducting property of ZnS nanoparticles. Results will be discussed in detail.

**KEY WORDS:** Nano particle, xrd and ZnS

## INTRODUCTION

Nature has many objects and processes that function on micro to nano-scale molecular ensembles of molecules present in plants such as chlorophyll present in the nanometer scale capture light energy and produces chemical energy. Until 1959 nobody dreamt of using atoms and molecules for fabricating devices and everyday apparatus. In 1959 Richard P. Feynman talked about the problem of manipulating and controlling things on small scale during his lecture. "There is plenty of room at the bottom" he envisioned etching lines a few atoms wide with beams of electrons effectively predicting the existence of electron - beam lithography which is used today to make silicon chips.

### Classification of Nanomaterials

The nano particles are classified into

1. Clusters
2. Colloid
3. Nano particle
4. Nano crystal
5. Nano structured (or) Nano scale material
6. Quantum dots

### 1. Clusters

A collection of atoms or reactive molecules up to about 50 units.

### 2. Colloid

Colloidal particles are a stable liquid phase containing particles in 1 to 1000 nm range.

### 3. Nano particle

A solid particle in the 1 to 1000 nm range that could be no crystalline aggregate of crystallites or a single crystallite.

### 4. Nano crystal

A solid particle that is a single crystal in the size of nanometer.

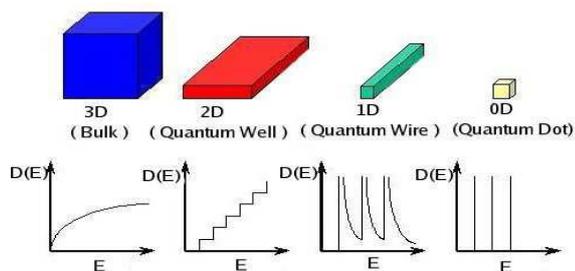
### 5. Nano structured (or) Nano scale material

Any solid materials has a dimensional value of nanometer.

Three dimension	-
Bulk Particle	
Two dimension	-
Thin films	
One dimension	-
Thin wire	

### 6. Quantum dots

A particle that exhibit a size quantization effect in at least less than one dimension.



## Importance of ZnS Nano Materials

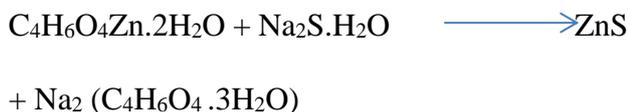
Nanosized inorganic semiconducting materials have been creating an extensive desire in recent years because of their structure, chemical and physical properties, which are discriminate from those of the bulk materials. Many techniques have been accustomed to synthesis ZnS nanoparticles such as sol-gel, electro chemical deposition, sonochemical, hydrothermal technique. ZnS can have polymorphic crystal structures, which are direct band structure. Nanoparticles attracted considerable attention in recent years because of their special properties, such as quantum size effects [1-5]. It has wide range applications including light emitting diodes, flat panel devices, solid state solar window layers, phosphors, photoconductors, catalysts, production of hydrogen, blue light diodes,

electro-luminescent displays, anti-reflection coatings for infrared devices and other non-linear optical devices[6-11]. In this paper, we report the synthesis and characteristics of zinc sulfide nanoparticles obtained by using chemical precipitation method with different temperature. The important aim of studies is finding the effect when increasing the temperature. The size, shape, crystal structure and band gap of ZnS nanoparticles was found. The simple and low cost method without any capping agent through eco-friendly path was utilized for synthesizing ZnS nanoparticles in this present work.

## Materials and Methods

ZnS nanoparticles were prepared by chemical precipitation method. The reactants used for synthesis of ZnS nanoparticles were sodium sulfide monohydrate ( $\text{Na}_2\text{S}\cdot\text{H}_2\text{O}$ ) and zinc acetate dihydrate ( $\text{C}_4\text{H}_6\text{O}_4\text{Zn}\cdot 2\text{H}_2\text{O}$ ). Using stoichiometric amounts in grams, 1 molar solution of each reactant was prepared in distilled water. The zinc acetate solution was stirred for 2 hours. Freshly prepared aqueous solutions of these chemicals were used for the synthesis of nanoparticles at room temperature.

The solution containing sodium sulfide was added drop wise in solution of zinc acetate under continuous stirring until the white precipitates were formed. Stirring was done for 1 hour to complete the reaction. The chemical reactions involved are as follows:

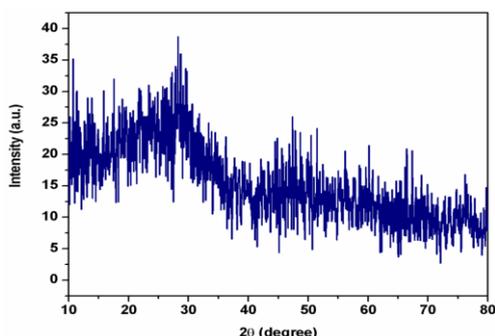


These precipitates were washed with distilled water to remove the impurities of sodium. After washing the precipitates were centrifuged at 2000 rpm and dried at room temperature for above 24 hours.

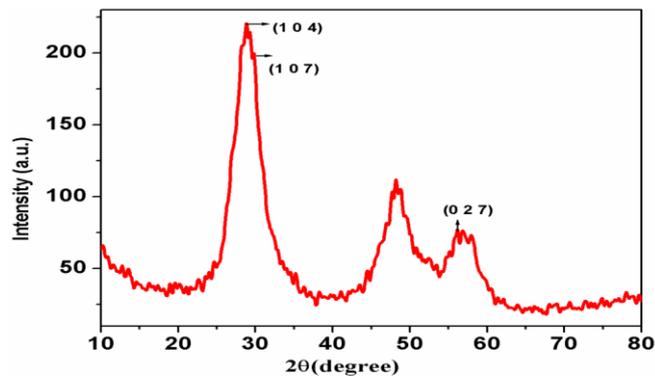
## Results and Discussion

### 1. XRD Studies

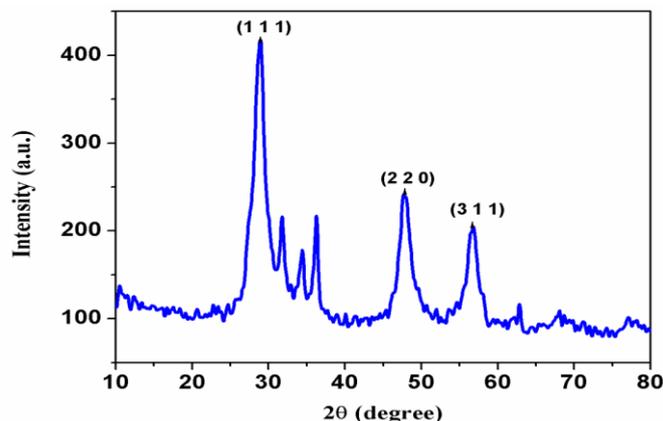
X-ray diffraction pattern of the ZnS nano particles recorded at room temperature, heated at 200°C and 400°C are shown in fig. 1a,1b and 1c.



**Fig. 1.a. X Ray diffraction spectra for ZnS nano materials at room temperature**



**Fig. 1.b. X Ray diffraction spectra for ZnS nano material at 200°C temperature**



**Fig. 1.c. X Ray diffraction spectra for ZnS nano material at 400°C Temperature**

From the X ray diffraction spectra it is observed that there is amorphous peaks appeared for the sample recorded at room temperature [12]. Also the spectrum recorded at 200°C and 400°C there is some sharp diffraction peaks obtained at  $2\theta$  values from 28° to 57° indicates the rhombohedral and cubic zinc blende structures of ZnS. This pattern was well matched with standard JCPDS data (card no: # 897386) [13,14] and sharp

reflection peaks in the XRD pattern indicates that ZnS particles were well crystallized. The broad peak indicates nanocrystalline behavior of the particles.

## 2. Surface Morphology

Atomic force microscopy (AFM) spectrum obtained by making ZnS colloidal solution dipping of on glass plate to get microscopic information on the surface structure and to plot topographies representing the surface relief. The AFM image of ZnS nanoparticles were taken at room temperature are shown in fig. 2a. From the AFM images of the ZnS, it is observed the average grain size is about 86nm.

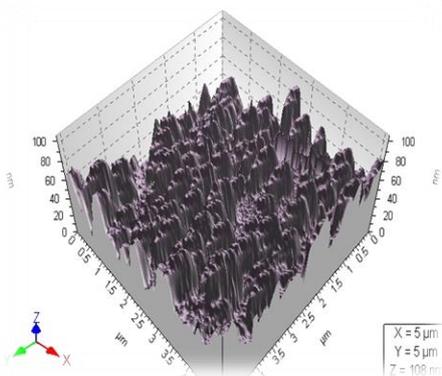
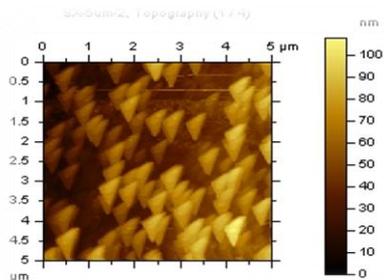


Fig.2a AFM image of ZnS nano particles

## 3. Morphological studies

The most of the SEM image of the nanoparticles in the present study have been found to be spherical shape and some of the particles are agglomerated (15). The particle sizes obtained from SEM images are found to be in the range of 73 to 80nm. Also the EDS spectrum confirms the presence of Zn and Sulphur.

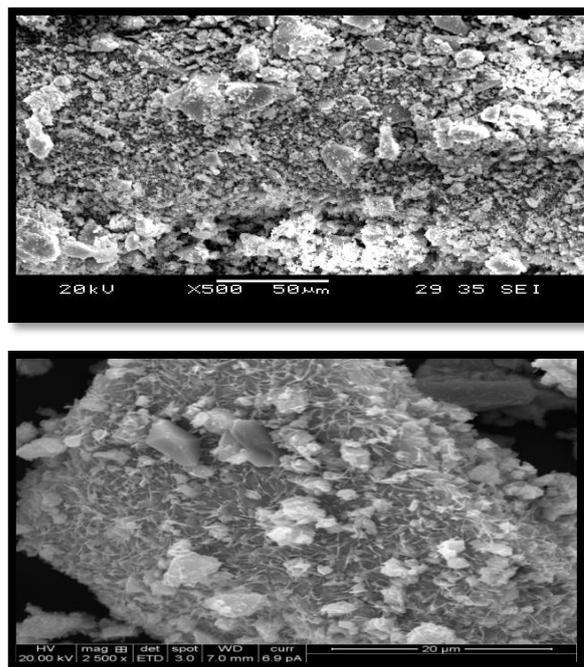


Fig. 3a. SEM images of ZnS nano particles

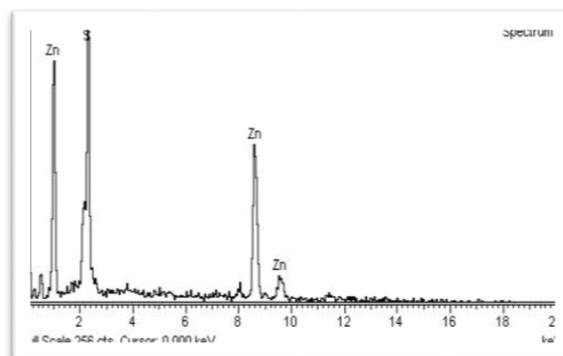
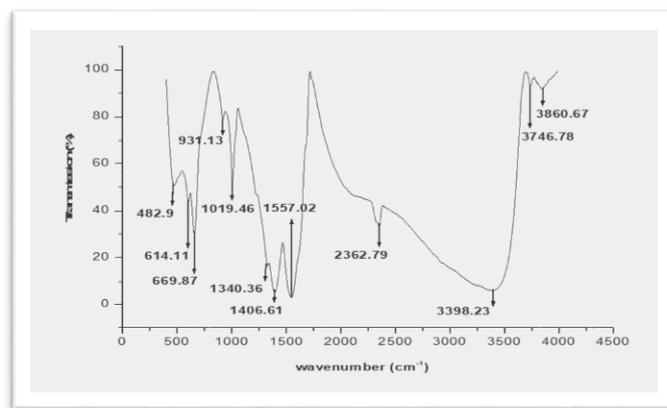


Fig.3b. EDS Spectrum of ZnS nano particles

#### 4. FTIR Studies

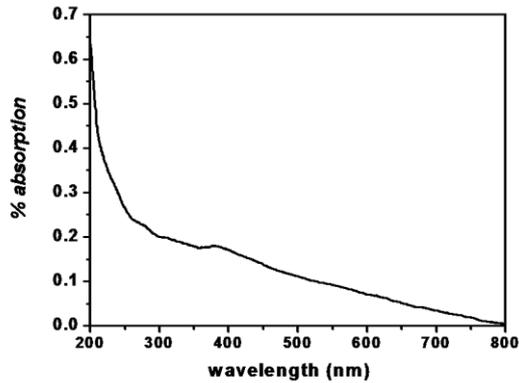
Nanoparticles of semiconductors have been investigated for a long time for their optical properties. There are few absorption peaks between 400 - 4000  $\text{cm}^{-1}$ , indicating that the prepared ZnS nano particles which was a near infrared transmittance material. The vibrational frequencies were observed at 482  $\text{cm}^{-1}$  and 614  $\text{cm}^{-1}$  which is corresponded to metal stretching mode for zinc and sulfide [15]. The transition band occurs at 3860  $\text{cm}^{-1}$ , 3746  $\text{cm}^{-1}$  and 3398  $\text{cm}^{-1}$  are described to O-H stretching of water molecule. The vibrational frequency obtained at 2362  $\text{cm}^{-1}$  is attributed to C-H stretching. The transition band occurs at 1557  $\text{cm}^{-1}$  which is ascribed to C-C stretching of carbon molecule present in zinc acetate. The vibrational frequency obtained at 1406  $\text{cm}^{-1}$  is attributed to C-H bending. C-O stretching frequencies are observed at 931  $\text{cm}^{-1}$ , 1019  $\text{cm}^{-1}$  and 1340  $\text{cm}^{-1}$ .



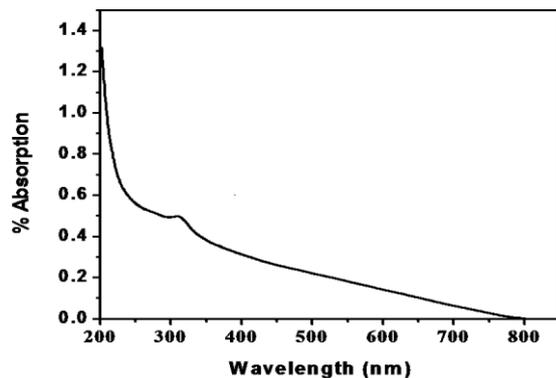
**Fig.4. FT IR Spectrum of ZnS nano materials.**

#### 5. UV Spectroscopic studies

The UV-VIS spectrum was recorded using Shimadzu spectrophotometer in the absorption mode and is shown in fig (5a and 5b). The optical absorption spectra of ZnS nanoparticles were recorded in the range of 800nm-200nm. The absorption peaks obtained in the ZnS nanoparticles at 270 nm (3.8 eV) for 400° C exhibit cubic structure related to 327 nm (4.448 eV) at room temperature gives a bulk structure. This indicates that the as prepared ZnS nanoparticles exhibit strong quantum confinement [15]. From the UV study, we can calculate the energy band gap.



**Fig 5a.**UV absorption spectrum of ZnS nano particles at room temperature



**Fig 5b.**UV absorption spectrum of ZnS nano particles at 400° C temperature

## 6. Electrical conductivity measurements



**Fig.6.** Two probe set up for measuring the electrical conductivity

**Table.1.** D.C.Electrical conductivity (resistance,capacitance,electrical conductivity and dielectrical) data of ZnS nano particles.

Temperature		Resistance (R)	Capacitance (C)	Conductivity $\sigma$ ( $\times 10^{-3}$ )
$^{\circ}\text{C}$	K	( $\text{M}\Omega$ )	(PF)	( $\Omega^{-1}\text{m}^{-1}$ )
120	393	58.2	11.2	0.021
110	383	62.3	10.5	0.019
100	373	67.3	10.3	0.018
90	363	77.9	9.6	0.015
80	353	99.1	8.8	0.012

Electrical conductivity measurements are carried out using two probe set up and LCR meter (Aplab). From the conductivity measurements, it is observed that the electrical conductivity values increases with increase of temperature. Also the temperature is increases the resistance was decreased. From these results we can assume the synthesized ZnS nanoparticles are in semiconductor nature.

## Conclusion

ZnS nanoparticles have been prepared by chemical precipitation method. X-ray diffraction was employed to study the structural properties. From the x ray diffraction spectra, the structure of the ZnS nano particles were found to be rhombohedral and cubic for the temperature varying from 200° C to 400° C. AFM and SEM images of the ZnS nano particles shows the particles size is to be 70nm and 82nm. EDS spectra is used for this study gives the elemental composition of zinc and sulphur of the synthesized nanoparticles. FTIR spectra showed the possible stretching and bending modes of the ZnS. UV absorption spectra revealed that the absorption band was present in the wave number region 270nm (blue shift) from the bulk. The electrical conductivity study exhibits the semiconducting property of ZnS nanoparticles. This chemical co precipitation method would be utilized because of its advantages of simplicity and low cost as well as the better quality of production of nanomaterials.

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