iJRASET
International Journal For Research in Applied Science and Engineering Technology

INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 7 **Issue:** IV **Month of publication:** April 2019

DOI: https://doi.org/10.22214/ijraset.2019.4232

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International Journal for Research in Applied Science & Engineering Technology (IJRASET)
ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 6.887
Volume 7 Issue IV, Apr 2019- Available at www.ijraset.com

Synthesis and Photoluminescence of Zinc Sulphide Nanorods by Wet Chemical Synthesis Method

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Abstract: ***Wet Chemical Synthesis method was used to synthesis undoped Zinc Sulphide. The as-prepared products were characterized using XRD, FE-SEM, UV-VIS and PL analysis. The crystal structures, morphologies and optical properties of as-prepared samples along with the particle size have been investigated. Photoluminescence spectrum shows strong emission near 540 nm. These multilayered nanorods could be a prominent candidate for photocatalysis application***

Keywords: Wet Chemical Synthesis, XRD, FE-SEM, UV-VIS and PL

I. INTRODUCTION

Zinc sulfide is a well known photo- and electroluminescent material belongs to II-VI group of semiconductor materials having large band gap (3.75 eV) and large excitonic energy (~37 meV) along with low Bohr exciton radius (2.5 nm) makes it suitable as host material for large variety of dopants for the fabrication of various solid state devices as well as small biomolecular probes for fluorescence [1-3]. It has also remarkable chemical stability against oxidation and hydrolysis resulting in its applicability as photo-catalyst [4]. A covalently bonded solid, zinc sulfide crystallizes in two different forms: zinc blende (cubic) and wurtzite phases (hexagonal) where Zinc blende (ZnS) is generally used as luminescent materials [5]. Due to its luminescence properties it is widely used in electronic industries in flat display, solar cells, sensors, lasers and also as catalyst in pollution treatment [6]. The characteristics and concentrations of dopants are responsible for particular properties and efficiencies of semiconductor nanoparticles. Hence investigation of the role of dopant concentration on photocatalytic, optical and electrical properties of doped semiconductor nanoparticles is very important from the viewpoints of basic physics as well as applications[7].

II. EXPERIMENTAL

ZnS samples have been prepared by Wet Chemical Synthesis (WCS) method. ZnS Nanocrystalline powder was prepared by using Zinc Acetate and sodium sulfide as the precursor, Ammonia as the complexing agent and Polyvinyl Pyrroldine (PVP) as the capping agent. All the chemicals used in the experiment were of analytical grade and were used without further purification. To find a suitable reagent concentration and stoichiometry for ZnS nanocrystalline powder, it was decided to fix the concentration. The Concentration of, the molar ratio of Zinc Acetate to Sodium Sulfide was kept as 1:2. The pH of the solution is 6.8. Zinc Acetate and Polyvinyl Pyrroldine was first dissolved in de-ionized water and this mixture was stirred at room temperature for 30 minutes to get a homogenous and transparent solution. Then Ammonia was added as a complexing agent on this $Zn(O_2CCH_3)2(H_2O)_2$ solution drop by drop and the solution was stirred continuously around 30 minutes. Sodium sulfide solution was prepared by adding it with the de-ionized water and this mixture was stirred for 60 minutes. In the $Zn(O_2CCH_3)2(H_2O)_2$ prepared solution, Sodium sulfide solution was added drop by drop and this mix was continuously stirred with the magnet at 700°C for 120 minutes. A white color precipitate was obtained which was separated by centrifugation and washed several times with double distilled water. The precipitate was dried in oven at 120°C for 2 hours to get ZnS Nanocrystalline powder sample.

III.RESULTS AND DISCUSSION

A. XRD Analysis

It has been reported that ZnS may have either cubic or hexagonal structure, depending on the synthesis conditions such as deposition temperature and precursor concentration [8]. The phase purity and crystal structure of these samples were analyzed by using CuKα radiations source in the range of 20^0 to 60^o with 0.050 step size using XPERT – PRO diffractometer. Figure 1 shows the XRD Pattern of the synthesized ZnS Nanocrystalline powder. The diffraction peaks are indexed to primitive hexagonal wurtzite structure and is in accordance with the JCPDS card no. 89-2144 with unit cell parameter a = 3.823Å and c = 68.728Å. From the X-ray diffraction peaks in Figure1 the particle size are determined from at the full-width at half-maximum [FHWM] of the XRD peaks. Using the Debye – Scherrer formula:

$$D = 0.89\ \lambda / \beta \cos\theta \qquad (1)$$

Where D, λ, β and θ are the average particle size, wavelength of the CuKα radiation, full width at half maximum of the diffraction plane and diffraction angle respectively. The higher intensity and sharpness of the peaks lead to perfect crystallization. The average calculated particle size of the synthesized ZnS nanocrystal is about 2 nm.

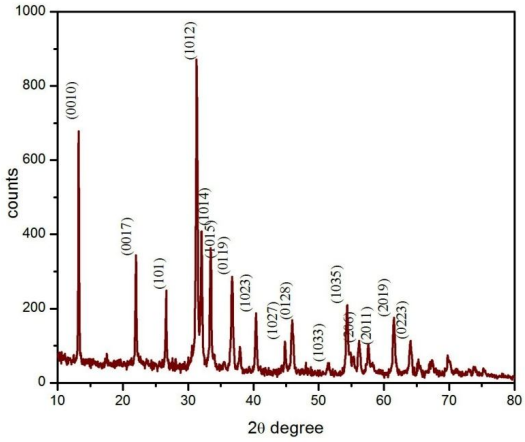

Fig.1: XRD patterns of ZnS nanocrystalline powder

B. FE-SEM Analysis

Surface morphological and Elemental compositions analysis of the prepared samples has been studied using Field Emission Scanning Electron Microscope (EIGMA –VP). Figure. 2 shows the FESEM image of undoped -ZnS Nanocrystalline powder. The high magnification FESEM image reveals that the products consist of a large quantity of flexible, various-diameter and rod like multilayered nanostructures.

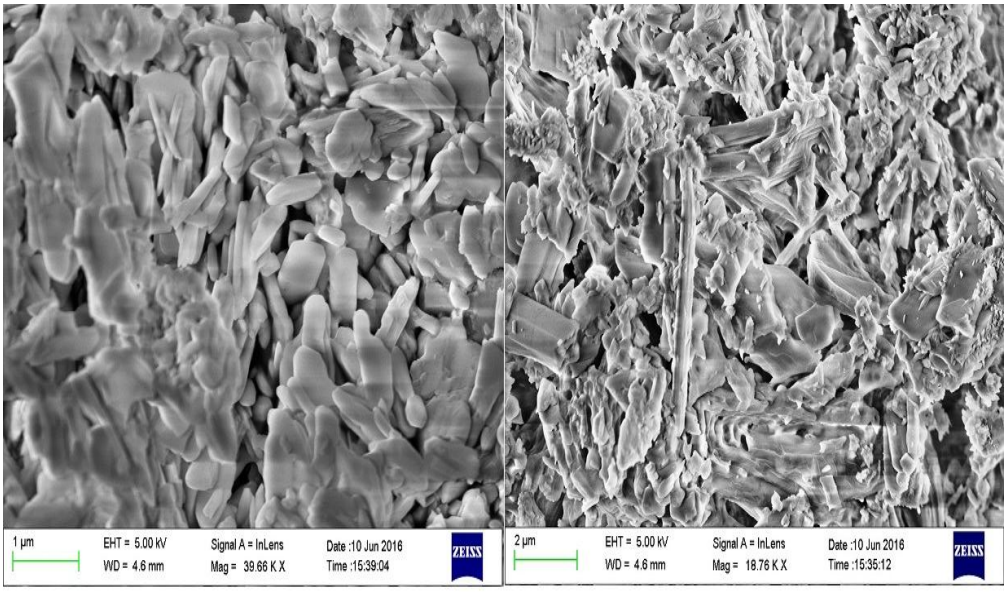

Fig. 2: FESEM image of ZnS nanocrystalline powder

C. UV-Vis Absorbance Spectra Analysis

Figure 3 shows the UV-Vis absorption spectrum of ZnS Nanocrystalline powder prepared with zinc acetate as a precursor along with PVP capping agent. It can be seen that the absorption peak appears at 280 nm. The absorption spectrum of the prepared sample covers entirely the UV, Visible and Near –IR region. The Near – IR region is not generally able to promote electron transitions and therefore it is possible to use the UV and Visible range of the sunlight for photocatalysis.

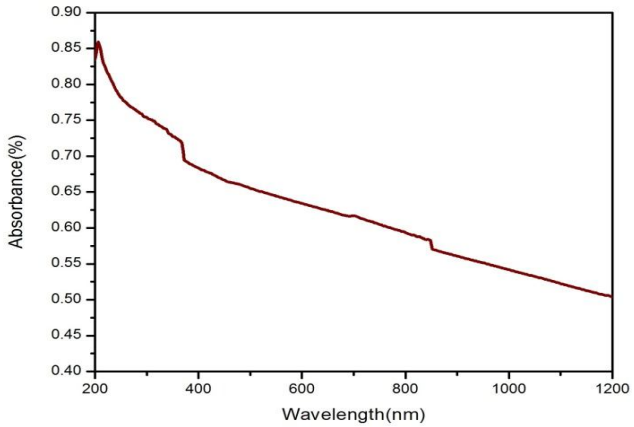

Fig. 3: UV-Vis Absorbance spectrum of ZnS Nanocrystalline powder

D. Photoluminescence Spectra Analysis

The photoluminescence properties of the samples were characterized using Horiba Jobin Yvon Fluorolog-3 Spectrofluorometer Figure.4 shows the PL spectrum of undoped ZnS Nanocrystalline powder prepared with Zinc acetate as precursor. The samples are photo excited at 350 nm. The PL peak at this range has been known due to the recombination between the sulfur-vacancy-related donor and the valence band [9]. The sample exhibits two emission bands in visible region spectra. The first emission band is at 380 nm and it is in violet region. The Green luminescence demonstrates the oxygen vacancies in the prepared samples and is a promising candidate for photocatalytic application.

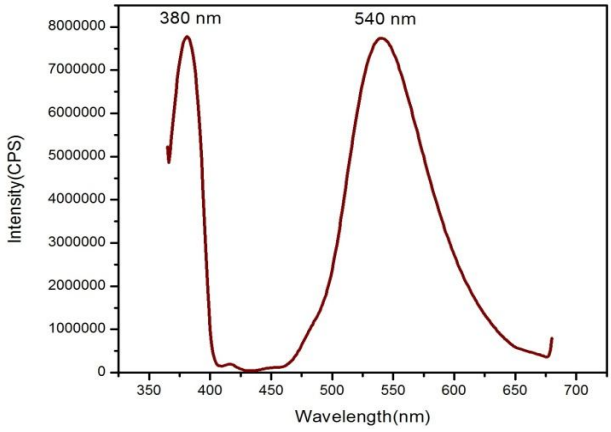

Fig. 4: Photoluminescence spectrum of undoped ZnS Nanocrystalline powder

IV.CONCLUSIONS

ZnS Nanocrystalline powders were synthesised using Wet Chemical Synthesis (WCS) method. X-ray diffraction studies exhibited hexagonal wurtzite and Rhombohedral structure for the as prepared samples. FESEM images clearly showed the formation of nanorods for samples prepared at room temperature.

The average diameters of the nanorods were found to vary between 2 – 6 nm. As the Nanorods have high surface to volume ratio, they are well suited for photocatalytic application. From the PL spectra it has concluded, the ZnS Nanocrystalline powder prepared with Zinc Acetate as precursor shows high intense broad visible emission with a peak at ~540nm (green emission). The broad emission band revealed in the visible region is due to oxygen defects which will enhance the electron hole pair separation rate in ZnS. This property is very much useful for photocatalytic application.

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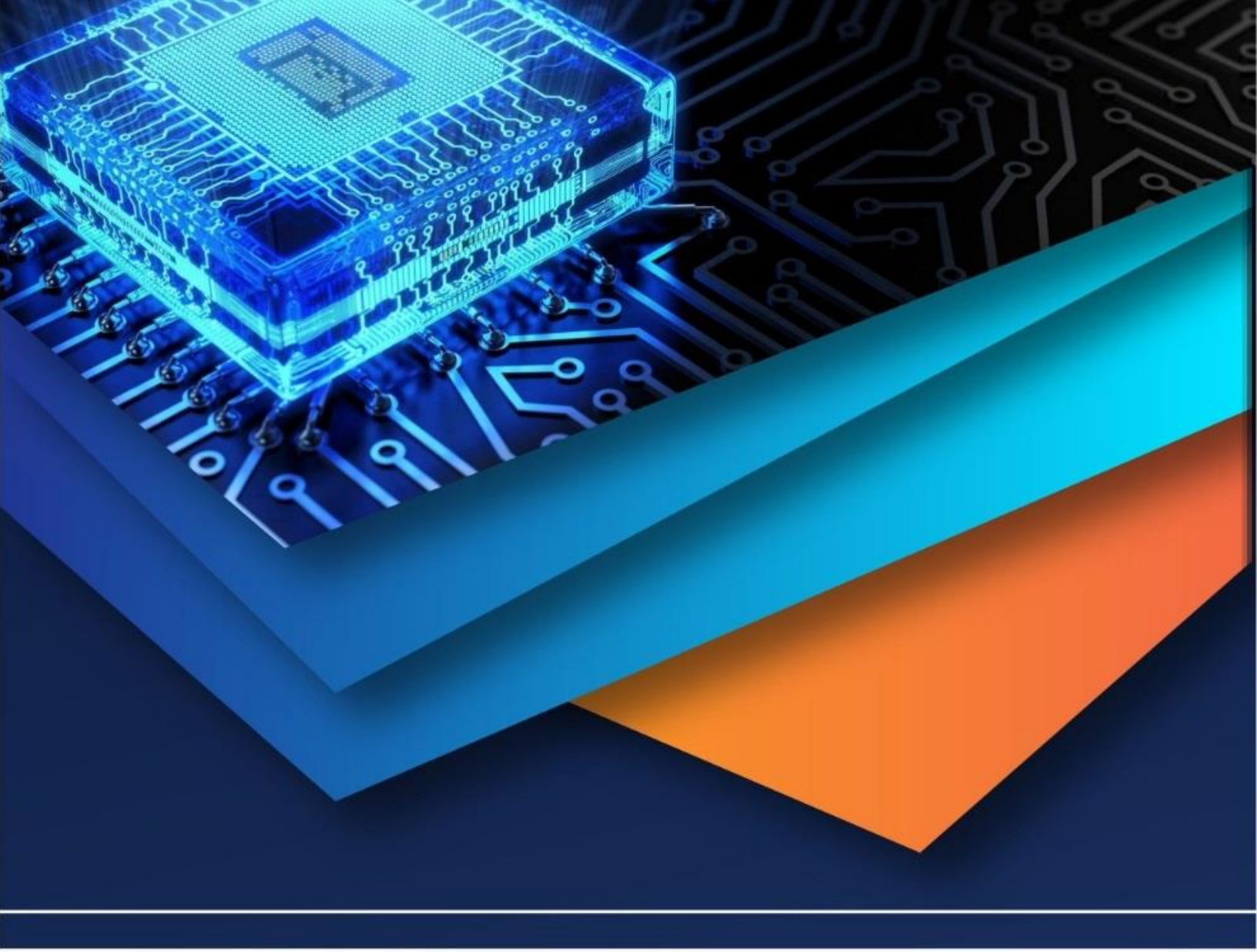

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