International Journal of Recent Technology and Engineering (IJRTE)
ISSN: 2277-3878 (Online), Volume-7 Issue-4s, November 2018

Structural and Optical Characterization of ZnS Nanoparticles Synthesized by Low Temperature Solid-State Method

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Abstract: ***The ZnS nanoparticles were synthesized by solid state reaction method. The synthesized sample was characterized by powder XRD, SEM, EDAX, UV-visible, fluorescence and FT Raman analysis. The powder XRD analysis shows the broad (111), (220) and (311) peaks which confirms the formation of cubic structure. The SEM image shows the agglomerated spherical shape of morphology. The EDAX analysis shows the composition of Zn and S are 46.47% and 53.53% respectively. The UV-Visible spectrum analysis shows the absorption wavelength as 325 nm and calculated band gap 3.81 eV. The fluorescence study reveals the near band edge emission at 363 nm and defect level peaks were observed in the higher wavelength of the photoluminescence spectrum. FT Raman analysis shows the vibration frequency at 234 cm^{-1} is due to the longitudinal optical (LO) mode of Zn-S lattice.***

Keywords: ZnS, XRD, SEM, EDAX, Optical Properties

I. INTRODUCTION

ZnS is a wide band gap II-VI compound semiconductor with energy gap of 3.54 eV at room temperature [1]. This semiconductor has received a great attention and remarkable physical properties that can exploited for versatile applications in the field of electronic devices in particular emitters, infrared windows, laser, electroluminescence and light emitting diodes [1-3]. In addition, the nanostructure ZnS is a non toxic and hence it is widely used for different biological applications especially pharmaceutics [3,4]. In order to use this semiconductor nanocrystal for these applications, it is very important to control the size of the nanoparticles [3]. The low dimensional size of the nanostructure material has the remarkable changes in their physical properties due to quantum confinement [3]. In general the structural point of view, ZnS exhibit two crystalline phases the one is cubic zinc blende and the other is hexagonal wurtzite structure, typically zinc blende is stable phase at room temperature [2]. The different physical and chemical methods have been reported for the synthesis ZnS nanoparticles [5]. Among the physical and chemical roots, in the recent years the solid state method has made great attention for synthesis of nanoparticles [6]. The advantage of this technique is simple processes, controllable size and distribution of size of the nanoparticles [6]. In this paper we report the structural and optical properties of ZnS nanoparticles.

Manuscript published on 30 November 2018.
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II. EXPERIMENTAL METHOD

The 1:1 ratio of $ZnSO_4.7H_2O$ and Na_2SO_4 chemicals were used as starting materials for synthesis of ZnS nanoparticles. The $ZnSO_4.7H_2O$ and Na_2SO_4 chemicals were mixed and grained well in a mortar pistol for about 1hr. The mixture was stirred and washed with de-ionized water then the solution was filtered out. The product was collected and heated temperature at 100˚C for 2 hrs to remove impurities in ZnS product. The synthesized ZnS sample has been characterized by structural, morphological and optical characterization studies. The structural analysis was studied using powder X-ray diffraction measurement using BRUKER D8 Advance X-ray diffractometer. The morphology and composition of ZnS was analyzed by using SEM and EDAX analysis using Carl Zeiss instrument. The optical absorption spectrum was recorded using Perkin-Elmer UV-visible spectrophotometer. The spectrum was recorded using JY Fluorolog-3-11 spectrofluorimeter at room temperature. The FT Raman analysis was performed using BRUKER FT Raman spectrometer.

III. RESULTS AND DISCUSSION

A. Structural analysis

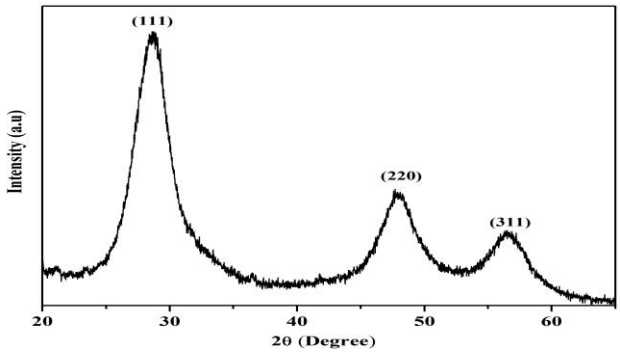

Fig.1 Powder XRD Pattern of ZnS Nanoparticles

Fig.1 shows the powder XRD pattern of ZnS nanoparticles. The XRD pattern shows the prominent peaks (2θ values) 28.51º, 47.91º and 56.56º were indexed (111), (220) and (311) planes respectively. This confirms the cubic structure and lattice parameter value found to be 5.42 Å, which is good agreement with that of existing literature value 5.439 Å [2]. Moreover, the broadening of the diffraction peak was observed due to a small size of the crystalline in the order of nanometer. The crystalline size is calculated using Debye-Scherer formula [7].

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

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where D is the crystalline size, β is the full width at half maximum (FWHM), K is the constant factor (0.89), λ is the wavelength of X-rays (λ=1.5406 Å) and θ is the diffraction angle. Based on the full width at half maximum of the diffraction peaks (111), (220) and (311), the calculated average crystalline size of ZnS is 4.63 nm.

B. SEM and EDAX analysis

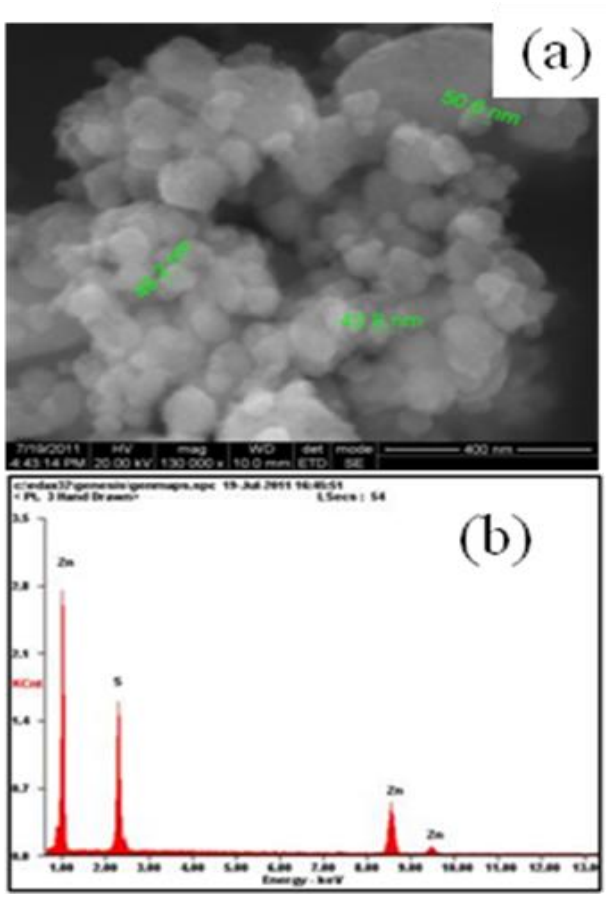

Fig.2 (a) SEM image and (b) EDAX spectrum of ZnS nanoparticles

Fig.2 (a) shows the high resolution scanning electron microscopy (HRSEM) of ZnS nanoparticles. The synthesized sample shows the agglomerated and spherical shape of morphology. The elemental analysis was performed through energy dispersive X-ray analysis (EDAX) attached with SEM instrument Fig.2 (b) shows the EDAX analysis of synthesized ZnS nanoparticles. The composition of present investigated sample shows 53.53 at.% and 46.47 at.% of Zn and S respectively.

C. Optical Absorption Analysis

UV-Visible spectroscopy is an important characterization study to analyze the optical absorption and band gap of semiconductors. The optical absorption spectrum of synthesized ZnS sample is shown in fig.3. The optical absorption cut off wavelength for the ZnS found to be 325 nm. The band gap energy was evaluated using the relation

$$E_g = hc/\lambda \quad (eV)$$

where h is plank's constant, E_g is the energy band gap of the material, c is the velocity of light and λ is the wavelength of optical absorption edge.

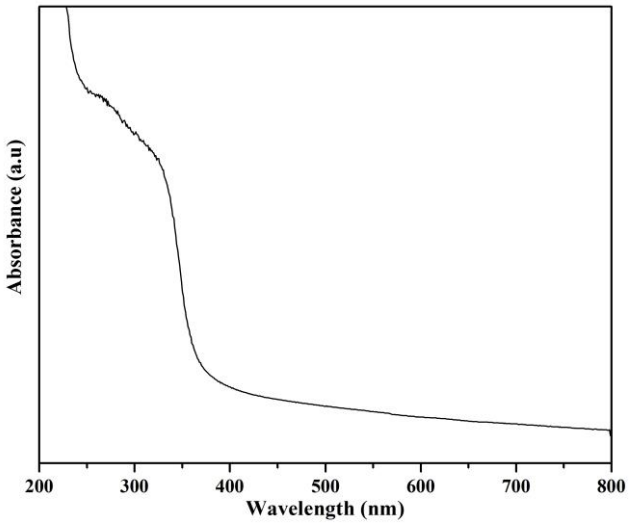

Fig.3 Optical absorption spectrum of ZnS nanoparticles

The evaluated band gap energy 3.81 eV (325 nm) which is blue shifted about ~ 0.16 eV with respect to that of bulk band gap of ZnS 340 nm (3.65 eV). The blue shifts in the absorption edge to higher energy (shorter wavelength) could be explained by the quantum confinement effect of present investigated sample [8]. The optical absorption band of ZnS is considerably broadening which indicates that the increase of the distribution of ZnS nanoparticles [9].

D. Fluorescence analysis

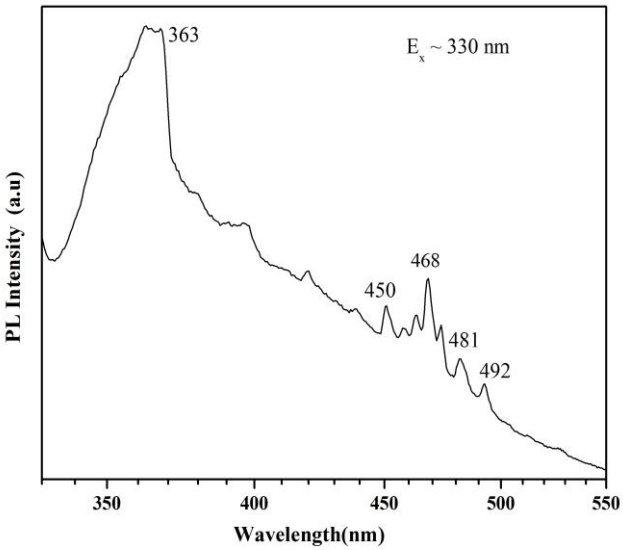

Fig.4. Fluorescence spectrum of ZnS nanoparticles

The Fluorescence characterization is used to study the optical emission property of semiconductor crystalline materials. Fig.4 shows the fluorescence spectrum of ZnS nanoparticles. The sample was excited at 330 nm by Xenon lamp source. The fluorescence emission peak at 363 nm (3.67 eV) is observed due to the near band edge (NBE) emission of ZnS [8]. In addition, the low relative intensities of other peaks 450, 468, 481and 491nm were observed in the high energy of the fluorescence spectrum which is attributed to the defect related emission of present investigated sample [8].

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E. FT Raman analysis

Raman spectroscopy is a non-destructive characterization method to analyze the molecular structure of the crystalline materials.

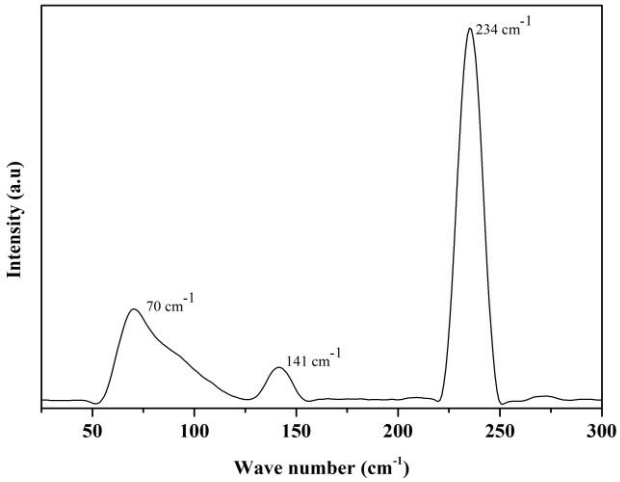

Fig.5 FT Raman spectrum of ZnS nanoparticles

Fig.5 shows the FT Raman spectrum of ZnS nanoparticles. The spectrum shows three vibration modes 234 cm^{-1}, 141 cm^{-1} and 70 cm^{-1}. The dominant and higher relative intensity of the peak at 234 cm^{-1} is assigned to the longitudinal optical (LO) mode of the Zn-S lattice [10,11]. Secondly, the low relative intensity of the peak at 141 cm^{-1} is due to the transverse acoustic (TA) mode of Zn-S lattice [11,12]. In the latter, the broad spectrum ranging between 50 -125 cm^{-1} with maximum peak intensity centered at 70 cm^{-1} is attributed to the E_2 vibration mode of ZnS [12].

IV. CONCLUSION

The zinc sulphide nanoparticles were successfully synthesized by solid state method. The powder XRD analysis confirms the cubic zinc blende structure and broadening of the peaks clearly indicates that the crystalline size in the order of nanometer. The spherical shape of morphology was observed and EDAX analysis confirms the stoichiometric ratio of Zn and S elements present in the sample. The optical absorption study reveals the absorption cut off wavelength 325 nm and evaluated band gap energy 3.81 eV. The fluorescence study shows the near band edge emission at 363 nm is due to the recombination of free excitons. Moreover, the low energy side of the fluorescence spectrum due to the defect level emission was observed. The FT Raman study reveals the three lattice vibration modes and it further confirms the tetrahedral lattice vibrations of Zn and S atom of the present investigated ZnS nanoparticles.

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