

Synthesis and Characterization of Zinc Selenide Nanoparticles at various Reaction Time

I.R. Celine Rose ^a, A. Jeya Rajendran ^{a*}

^aDepartment of Chemistry, Loyola College, Chennai-34, India.

*jeyarajendran@yahoo.com

Abstract— Nano Particles of Zinc Selenide with different reaction time were synthesized by Solvothermal method, using Zinc acetate and Selenous acid as precursors at room temperature and at various reaction times. The composition, morphology, and optical properties of the ZnSe nanoparticles were characterised using powder XRD, SEM, UV-Vis absorption spectroscopy, PL. X-ray diffraction analysis of nanoparticles revealed the crystalline nature and the particle size as quantum dot range (3-9nm). The direct band gap energy (E_g) of ZnSe NPs was found to be 2.76 eV. The dielectric properties of ZnSe like dielectric Constant, dielectric loss, were studied at temperature range from 40 to 140°C and frequency ranges from 50Hz to 5MHz.

Keywords- X-ray diffraction, optical properties, dielectric studies.

I. Introduction

Colloidal semiconductor ZnSe nanoparticles (band gap 2.7 eV) have been intensively investigated for the past decade [1-3] because their wide direct band gap is desired for the next generation of optoelectronic communication systems. A great number of synthetic technologies have been reported in the field of II-VI chalcogenides nanoparticles in wurtzite hexagonal structure of Cd chalcogenides [4-5] and zinc blende structure of Zn chalcogenides nanocrystallites [6-7]. Nanoparticles can be prepared by a variety of methods such as molecular beam epitaxy [8] chemical vapour deposition [9], reduction by ionizing radiation [10], thermal decomposition in organic solvents [11] chemical reduction (or) photo reduction in reverse micelles [12] Solvothermal [13] and hydrothermal method [14], it has been assessed that the colloidal stability (Colloidal solutions), Particle size and properties of nanoparticles depend strongly on the specific methods of preparation and the experimental conditions applied [15].

Traditionally ZnSe has been prepared through a solid state reaction between elemental Zinc, and Selenium at relatively high temperature. Selenides which are very useful materials have been widely used as thermoelectric cooling materials, Optical filters, Optical recording materials, Solar cells, Supersonic materials and sensor and laser materials [16-20]. Solvothermal methods have also been developed for the synthesis of ZnSe and CdSe. The advantage of the solvothermal process is that they required relatively low temperatures and pressures. However, organic solvents are usually harmful to the environment, therefore the non-toxic solvents would be used for achieve the products in a large scale.

II. Experimental detail

A. Synthesis of ZnSe nanoparticles

All reagents were used of analytical grade purchased from sigma and used as received without further purification. The precursor of Zinc acetate $Zn(CH_3COO)_2 \cdot 2H_2O$, and Selenous acid (H_2SeO_3), were used for synthesis. $Zn(CH_3COO)_2 \cdot 2H_2O$, H_2SeO_3 , were dissolved in deionized water. $C_2H_6O_2$, methanol and H_6N_2O was added to the mixture, then refluxed under vigorous stirring. The White color precipitates were collected, washed with anhydrous Methanol and hot distilled water for several times, then dried in vacuum at 50°C. Maintaining the Integrity of the Specifications

B. Characterization

UV-Visible spectra of ZnSe was recorded (Shimadzu uv-vis-2700) Spectrometer and PL (Cary Eclipse-EL0808385) Spectrometer and structural information of the ZnSe nanoparticles was studied by using a X-ray diffractometer (MAC Science MO3XHF22) with $CuK\alpha$ radiation $\lambda=1.54$. SEM images of the ZnSe particles were taken using SEM working at 200kV. A HIOKI HITESTER-3532 LCR instrument was used to measure the capacitance, dielectric constant and loss of the sample as a function of frequency (50Hz-5MHz) and temperature (40°C-140°C).

III. Results and Discussion

A. X-ray diffraction

X-ray diffraction patterns of synthesized ZnSe nanoparticles at 60°C shown in (fig 1). Crystalline size was estimated from the XRD peaks of ZnSe nanoparticles using the Scherrer formula $A=0.94\lambda/\beta \cos\theta$. From different θ values, the calculated average particle size is about 9 nm. Inherent stress inside a nanocrystal could contribute to broadening of the XRD peaks.

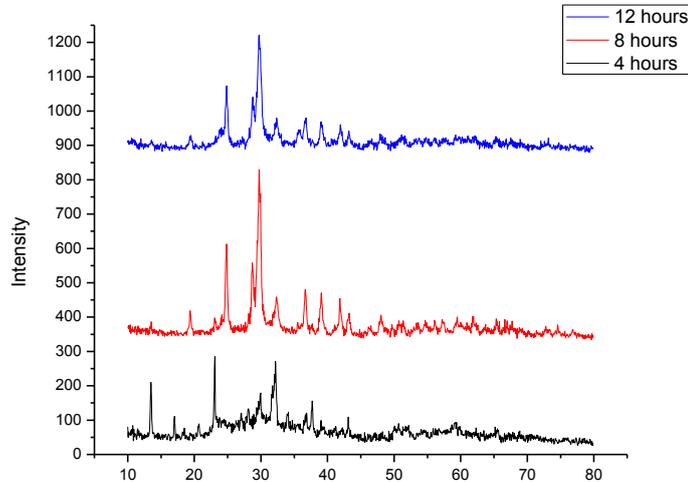


Fig-1 X-ray diffraction of ZnSe nanoparticle

The optical band gap (E_g) was calculated from the classical relationship of near edge optical absorption of semiconductors $\alpha=K(h\nu-E_g)^{n/2}/h\nu$ where k is constant, E_g is the optical band gap and n is a constant equal to 1 for direct band-gap semiconductors. The plot of $(\alpha h\nu)^2$ vs. $h\nu$. The direct band gap energy (E_g) of ZnSe NPs was found to be 2.76 eV. The band gap increases as the particle size decreases.

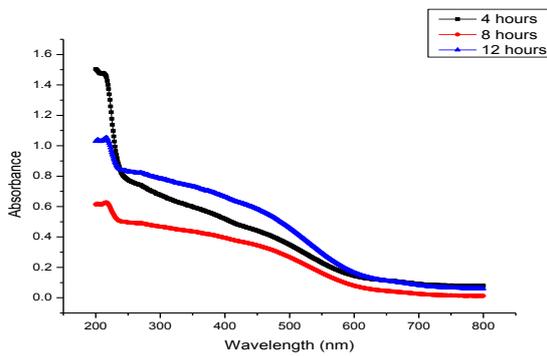


Fig-2 UV spectrum of ZnSe nanoparticle

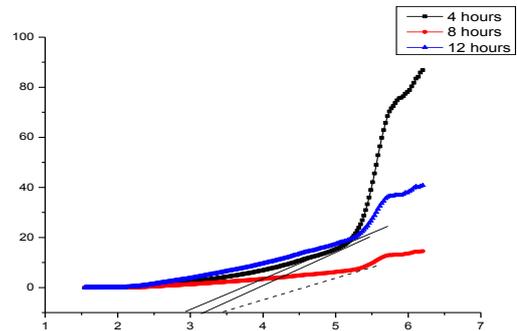


Fig-3 Band gap calculation of ZnSe nanoparticle

Fig-4 shows the photoluminescence(PL) spectra of Zinc selenide nanostructure, the near edge emission was observed at 333, 335, 337 nm at different reaction times. Good crystalline quality of ZnSe nanoparticles gives a less number of peaks. The broad emission peak which is assigned to blue emission[20].

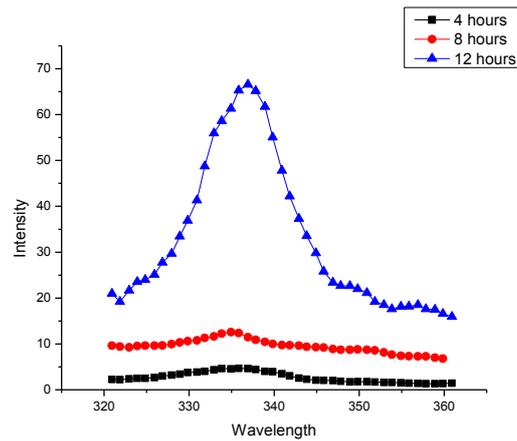
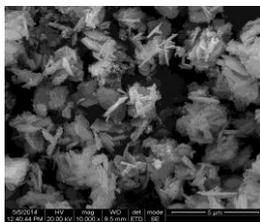
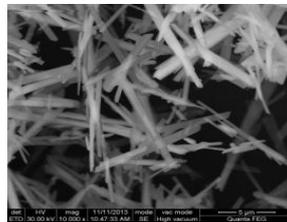


Fig-4 PL spectrum of ZnSe nanoparticles

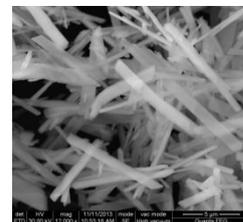
Fig-5 Shows the scanning electron micrographs (SEM) of ZnSe. The figures show high magnification image of the samples. It confirms the enhancement of well defined morphological crystallites is the sample with a vibrant needle morphology.



4 hours



6 hours



8 hours

B. Dielectric Studies

dielectric constant, and dielectric loss are important parameter in the selection of material for device application.

Temperature and Frequency dependence of dielectric constant:

Dielectric studies of the bulk material were carried out A HIOSKI HITESTER-3532 LCR meter as the function of frequency (50Hz-5MHz) at the temperature of 40oC to 140oC. The dielectric constant has been

calculated by using the eqn.
$$\epsilon = \frac{cd}{A\epsilon_0}$$

where d is the thickness of the sample, A is the area of the sample.

The relative permittivity (ϵ_r) is usually known as permittivity.

The variation of dielectric constant and dielectric loss ($\tan\delta$) of ZnSe nanoparticles with frequency is shown in figures 6 and 7. As the frequency increases the dielectric constant is found to be decreases exponentially and attain constant values. At low frequencies the dipole can easily switch alignment with the changing field. As the frequency increased, the dipole are cease to rotate and maintain phase the applied field, thus they reduce their contribution to the polarization[21]. The very low value of the dielectric constant at higher frequencies is important for the fabrication of materials towards photonic and electro-optic devices [22].

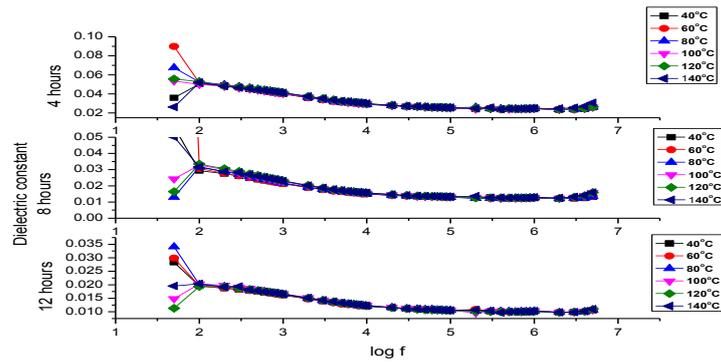


Fig-6 Dielectric constant Vs log f

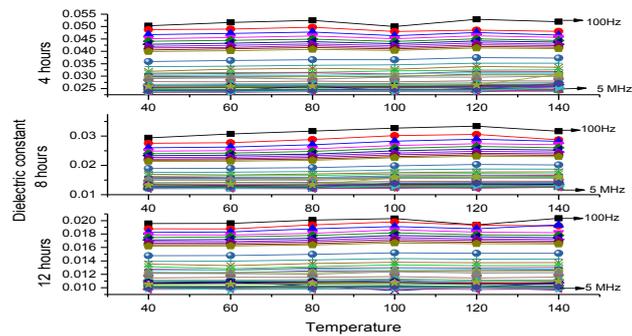


Fig-7 Dielectric constant Vs Temperature

Temperature and frequency dependence of dielectric loss

Fig 8,9 - shows the variation of $\tan\delta$ (dielectric loss) with frequency ranges from 50Hz to 5MHz at 40°C to 140°C. If the electric polarization in a dielectric is unable to follow the varying electric field, dielectric loss occurs, it is observed that the loss factor shows a definite minimum at each temperature, which shifts to higher frequency as the temperature increases. Also dielectric loss is observed to increase with increase of temperature[23] and this dependency can be explained as the average time spent by the carrier traps depend on the energy difference between two states.

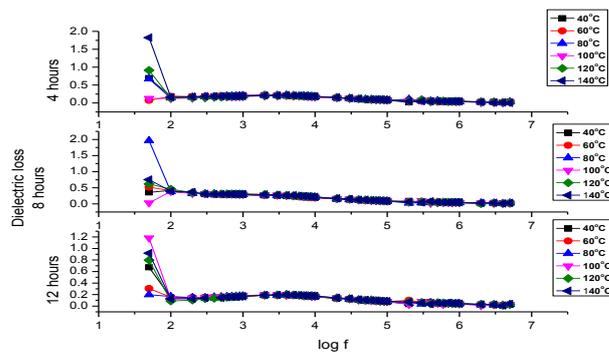


Fig-8 Dielectric loss Vs log f

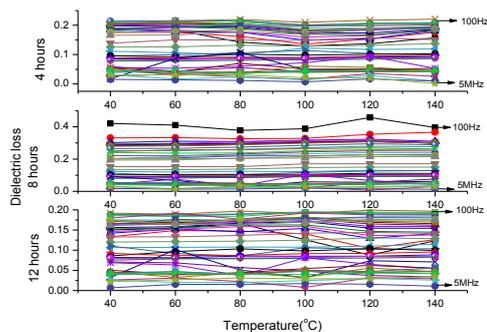


Fig-9 Dielectric loss Vs temperature

V. Conclusion

The ZnSe are successfully synthesized by Solvothermal method at 60°C. In XRD, the broadening of peaks indicates the inorganic components are in nanometer scale. The direct band gap energy (E_g) of ZnSe NPs was found to be 2.76 eV. In PL spectra, peaks were observed in at 333,335,337nm at different reaction times. Hence the rate at which progress in made on various fronts, the quantum dot will have a greater impact in the near future.

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