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Synthesis and Characterization of ZnSe Nanoparticles by Co-precipitation Method

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ABSTRACT

Zinc selenide nanoparticles (ZnSe NPs) have been synthesized by simple and inexpensive co-precipitation method using polyvinylpyrrolidone (PVP) as capping agent. The morphology and structure of ZnSe nanocrystals have been characterized by using field emission scanning electron microscopy (FE-SEM), X-ray diffraction studies (XRD), ultraviolet-visible spectroscopy (UV-vis), Fourier transform infrared spectrum (FTIR) and photoluminescence spectroscopy (PL). XRD study evident that the synthesized ZnSe nanoparticles were cubic in structure. Strong blue shift absorption (371 nm) is observed from UV–visible spectrophotometry. The presence of hydrazine monohydrate and polyvinylpyrrolidone in the ZnSe nanoparticles is confirmed by the FTIR spectrum. The enhanced luminescence property is measured from photoluminescence spectrophotometry.

1. Introduction

Nanotechnology has dynamically grown as an important field of recent research with wide spread applications in the fields of electronics and medicine. Nanotechnology can be defined as an investigation for the design, synthesis, and handling of the structure of particles with a dimension smaller than 100 nm [1]. Recently, nanostructured materials with a defined size and shape have attracted much interest due to their vast potential for fundamental research into the effect of dimensionality and size on physical and chemical properties, as well as for electronic and optoelectronic nanodevice materials. As one of the important Zn-based II-VI semiconductors, zinc selenide (ZnSe) has been considered to be a perspective material for optoelectronic devices [2], solar cells [3], photocatalytic activity [4], light emitting diodes (LEDs) [5], photoluminescence (PL) devices [6], and other biological labels [7].

Nowadays, a variety of methods has been developed to control the size and morphology of nanoparticles, such as the spray route [8], vapor-phase [9], thin films [10], hydrothermal route [11], reverse miscell method [12], wet chemical route [13], and co-precipitation method [14]. Among these techniques, the co-precipitation method is of particular interest since it is relatively simple, inexpensive, convenient and environment-friendly. Tatyana Gutul et al [15] has used polyvinylpyrrolidone (PVP) as a stabilizer in the synthesis of ZnO nanoparticles by an ultrasound-assisted sol–gel method allowed for the obtainment of stable colloidal solutions with good luminescence properties. The solid product consist of nanocomposite-encapsulated nanoparticles with sizes of 30–40 nm in a PVP matrix. Here, we report the co-precipitation route to obtain ZnSe nanoparticles. It is observed that capping ZnSe by PVP has improved the PL efficiency remarkably. The optical absorption and PL measurements were carried out at room temperature.

2. Experimental Methods

2.1 Materials

The chemicals mainly used are zinc chloride ($ZnCl_2$), sodium selenide (Na_2SeO_3), ethylene glycol ($HO\text{-}CH_2\text{-}CH_2\text{-}OH$), hydrazine monohydrate ($N_2H_4.H_2O$), polyvinyl pyrrolidone (PVP), and ethanol. All chemicals were used without further purification or treatment. Deionized water was used in all experiments.

2.2 Synthesis of ZnSe Nanoparticles

0.2 M solution of zinc chloride and sodium selenide was prepared in 70 mL of distilled water and the solutions were transferred to 250 mL round bottom flask. After that, equal ratio (1:1) of hydrazine monohydrate and ethylene glycol solution was added. Then polyvinyl pyrrolidone (PVP) solution was added dropwise to the above solution with constant stirring for 8 hours. Finally, a yellow precipitate was obtained. The obtained precipitate was washed with anhydrous ethanol for several times, then dried under vacuum at 70 °C for 2 hours.

2.3 Characterization Techniques

UV-visible spectrophotometer (Perkin Elmer -Lambda 35) was used for studying the spectral response of ZnSe NPs. Fourier-Transform Infrared Spectroscopy (FTIR) results were acquired from Jasco 6300 spectrometer (Perkin Elmer mode) in the range of 400 – 4000 cm^{-1}. The surface morphology of ZnSe NPs and binding energy of the element was inspected using FE-SEM (Hitachi SU6600, Japan) and EDAX (EMAX, Horiba 8121-H, Japan). Powder X-ray Diffraction (XRD) was carried out using X-ray diffraction meter - Cu Kα radiation. (Rigaku, Miniflex-600, Japan). The photoluminescence (PL) spectra of the collected powders were recorded using Perkin-Elmer LS-55 spectrofluorometer.

3. Results and Discussion

3.1 X-Ray Diffraction (XRD)

The X-ray diffraction pattern of synthesized ZnSe Nps is as shown in Fig. 1. The XRD peaks are obtained at angles (2θ) of 27.63, 45.15, 53.53, 65.70 and 73.25 corresponding to the indices of (111), (220), (311), (400), and (331) planes respectively. In order to confirm all the diffraction peaks are indexed as the typical cubic structure of ZnSe Nps. Their positions were in accordance with the reported pattern (JCPDS 37-1463) [16, 17]. The size of the nanoparticles was estimated by using the Debye Scherrer's formula,

$$D = (0.9\lambda)/\beta\cos\theta \qquad (1)$$

where, λ is the wavelength of X-ray used (0.15418 nm in the present case), β is the full width in radiation at half-maximum of the peak, and θ is the Bragg angle of X-ray diffraction peak. Calculation made on the (220) peak at 2θ = 45.15° gave a value of 30 nm for the average crystalline diameter of the synthesized ZnSe nanoparticles.

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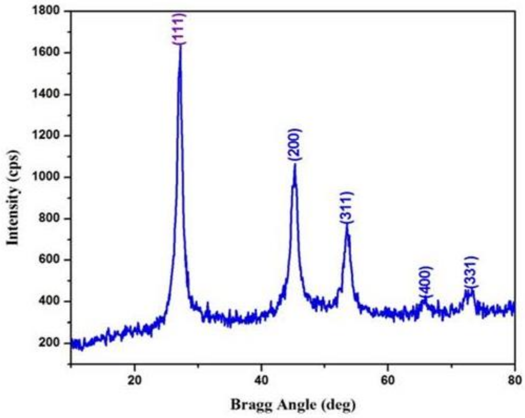

Fig. 1 XRD pattern of synthesized ZnSe NPs

3.2 UV-Visible Spectroscopy

The optical properties of ZnSe NPs are dependent on the size and the shape. The optical properties of ZnSe NPs were characterized by UV–visible absorption and the result is shown in Fig. 2(a). The UV–visible absorption spectra exhibited strong surface plasmon resonance peak at 371 nm which is characteristic of ZnSe NPs. The optical band gap of ZnSe nanoparticles is determined by plotting $(\alpha h\upsilon)^2$ versus $h\upsilon$ shown in Fig. 2(b). The band gap energy of ZnSe NPs was found to be 3.3 eV that showed the blue shift of 0.6 eV from standard bulk band gap at room temperature (Eg = 2.7 eV). It is familiar that the reaction situation such as heating time, temperature, and amount of reagents has an effect on the morphology, structure and size of the products in the processes [18].

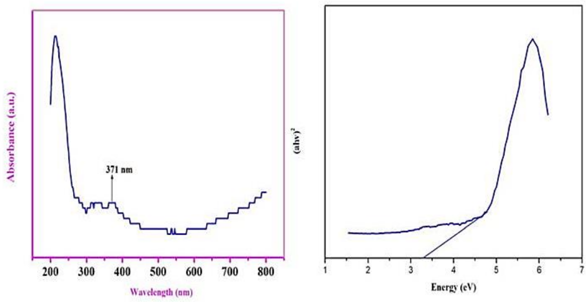

Fig. 2 (a) UV–visible absorption spectrum, (b) Band gap spectrum

3.3 Fourier Transform Infrared Spectroscopy (FTIR)

FT-IR spectrum of the corresponding sample is shown in Fig. 3. The characteristic major peaks appearing at 482, 561, 651, 671, 970 cm^{-1} belong to Zn-Se vibrations. The broad peak at 3429 cm^{-1} and the weak peak at 1595 cm^{-1} are assigned to O-H characteristic vibrations resulting from all small quantity of H_2O in the sample. The sharp peak at 3199 cm^{-1} corresponds to N-H stretching vibration band and the shift toward lower frequency compared with hydrazine may result from the interaction of N_2H_4 with zinc ion and regular periodic structure of molecular precursor. The PVP characteristic vibrations such as C=O and C–N appeared at 1656 cm^{-1} and 1327 cm^{-1} respectively [19, 20].

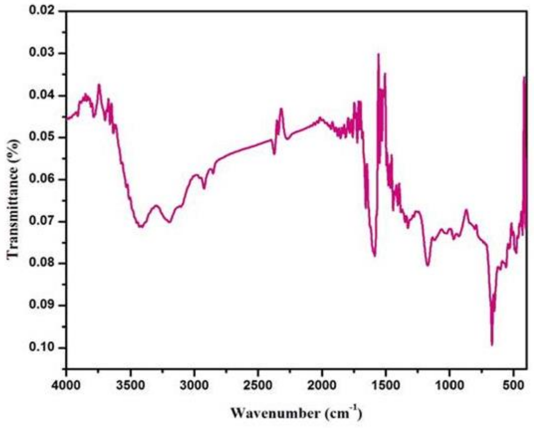

Fig. 3 FTIR spectrum formation of ZnSe NPs

3.4 Morphological and Elemental Study

Fig. 4 displays the FESEM analysis of synthesized ZnSe NP's. The image showed that the shape of the synthesized ZnSe NPs were polydispersed spherical shape. The average particle size was found to be 20-30 nm. It is in close agreement with XRD value. EDAX analysis [Fig. 4 (inset)] confirmed the presence of expected elementals such as, zinc (45.49 weight %) and selenide (54.51 weight %).

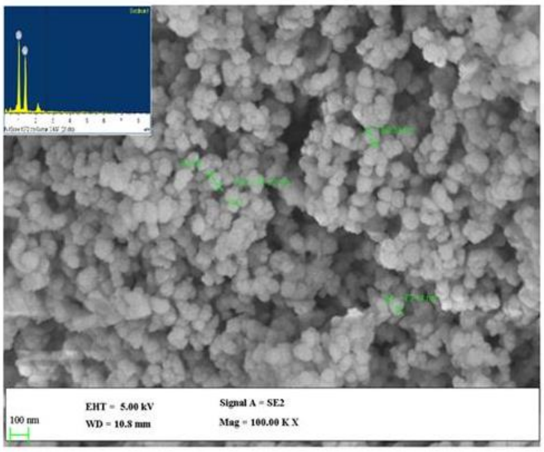

Fig. 4 FESEM and EDAX analysis of ZnSe NPs

3.5 Photoluminescence Analysis of ZnSe NPs

Fig. 5 shows the photoluminescence emission spectra of the synthesized ZnSe nanoparticles at which excitation wavelengths of 231 nm are attributed to the blue emission resulting from the recombination of a photon generated hole with a charge state of the specific defect. The absorption and PL peak of the ZnSe were shifted towards a shorter wavelength in comparison to bulk ZnSe due to the nanosized effect [21].

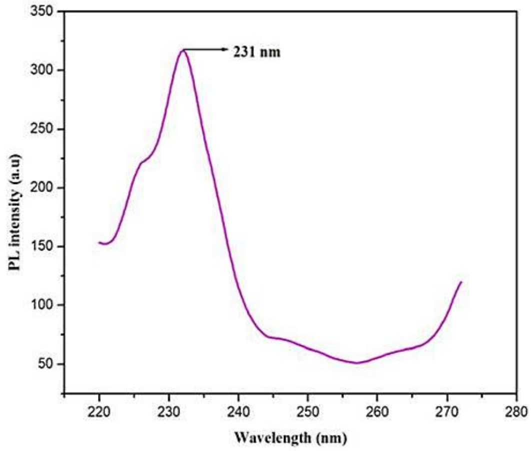

Fig. 5 Photoluminescence spectra of the ZnSe NPs at room temperature

4. Conclusion

ZnSe nanoparticles were successfully synthesized through co-precipitation route by using PVP as a capping agent. The surface morphological studies have confirmed the formation of ZnSe nanoparticles with a diameter of 20–30 nm. The high PL quantum efficiency is due to a well passivated surface of the ZnSe nanostructure. The EDAX result confirmed the expected elements present in the synthesized nanoparticles.

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