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Synthesis and Characterization of 1-thioglycerol capped CdSe and Cu (copper) doped CdSe nanoparticles at Room Temperature

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Abstract: Copper doped cadmium selenide nanoparticles (CdSe:Cu) have been successfully synthesized by solution based chemical technique using 1-thioglycerol as surfactant. The as prepared nanoparticles were characterized by X-ray diffraction (XRD), UV-vis spectroscopy, Photoluminescence (PL), high resolution transmission electron microscopy (HRTEM), selected area diffraction (SAD) and energy dispersive spectro scopy EDS). Powder X-ray diffraction reveals the formation of cadmium selenide nanoparticles with hexagonal (wurtzite) structure. It was observed the presence of copper does not alter the structure of CdSe. EDS measurements are presented to confirm successful doping of the wurtzite CdSe. Particle size and the crystallinity of CdSe:Cu NPs were confirmed through HRTEM and SAD patterns. The UV-Visible (UV-Vis) studies show that pure and CdSe:Cu showed blue shift with respect to the bulk value. PL spectrum reveals that the PL spectra (λ_{ex} = 400 nm) of CdSe has a emission band located at 675 nm and CdSe:Cu at 694nm for (0.1M and 0.3M) concentrations. It was observed increase in the concentration of copper slightly shifts the emission towards the higher wavelength.
Keywords: CdSe, nanocrystals, Photoluminescence.

1. Introduction and Experimental

In recent two decades, research has been done in the synthesis and characterization of II-VI semiconductor nanoparticles because of their unique properties and also the growing demand in electronic industry [1-4]. Cadmium selenide is an important II–VI compound semiconductor with direct band gap of 1.74 eV. CdSe is widely used for opto-electronic devices, laser diodes, nanosensing, biomedical imaging and also used in high-efficiency solar cell. There are considerable interests in transition metal doping (Mn, Cu, Co, etc.) of colloidal nanocrystals as a means of modifying their properties. Copper (Cu)-doped CdSe bulk materials have been studied long back [5, 6], and the studies show that Cu in CdSe can act as both acceptor and donor, which have large applications in photovoltaics and some other field of electronics. Cu doped CdSe naoparticles were reported by Meulenberg et al. [7], and the results indicated a limited luminescence range in infrared region (700–800 nm wavelength range). In this work, a simple solution based chemical technique has been reported for the synthesis and characterization of copper doped cadmium selenide nanoparticles. 1-thioglycerol was used as the capping agent.

1.1. Synthesis of 1-thioglycerol capped copper doped CdSe nanoparticles

Calculated amount of sodium sulfite and the selenium was added in 50 ml of distilled water under refluxed for 3 hrs for the preparation of sodium seleno sulfate. In a typical procedure, the aqueous solution of 0.5M of cadmium acetate was added to the aqueous solution of copper acetate (0.1M), under constant stirring the capping agent 2ml of1-thioglycerol was added to the above mixture and as-prepared sodium seleno sulfate (Na_2SeSO_3) aqueous solution was introduced, and then the ammonia solution was added to adjust the pH value to 10–11. The mixture was stirred for several hours to obtain a red precipitate. The particles were washed five times with acetone using centrifugation. The washed particles were dried at room temperature.

2. Results and Discussion

2.1. XRD, HRTEM and EDAX analyses

Fig.1. shows the XRD patterns of the pure CdSe and Cu-doped CdSe nanocrystals respectively. It shows distinct diffraction peak position corresponding to the (002), (110), (103) and (112) crystalline planes of wurtzite CdSe. It is to be noted that, in all the samples the peak observed in the XRD patterns match well with the wurtzite CdSe reported in the JCPDS Powder Diffraction File no.8-459. Broadening of the peaks shows the formation of CdSe nanocrystals. The diffraction pattern of the Cu-doped CdSe is similar to that of the pure CdSe without any change in phase indicating successful synthesis of Cu-doped CdSe nanoparticles. The mean particle size was determined by using Scherer equation. Based on the full width at half maximum, the average crystalline sizes of CdSe and doped CdSe with different concentrations (1% and 3%) were estimated to be 2-4 nm, respectively.

TEM micrograph in Fig. 2 shows well-formed nanocrystallites of copper doped cadmium selinide with nearly spherical in shape with little agglomeration. The sizes of these spherical NPs were found to range from 8 to 15 nm. The particle size is greater than the average size calculated by XRD because of agglomeration of the particles during drying process or non-distinguishable interfaces of the crystallites [8]. The existence of lattice planes on HRTEM image confirms the crystallinity of the doped sample. Fig.3. shows the EDAX spectra of copper doped cadmium selenide nanoparticles. The EDAX spectra indicate the presence of Cd, Se and Cu elements.

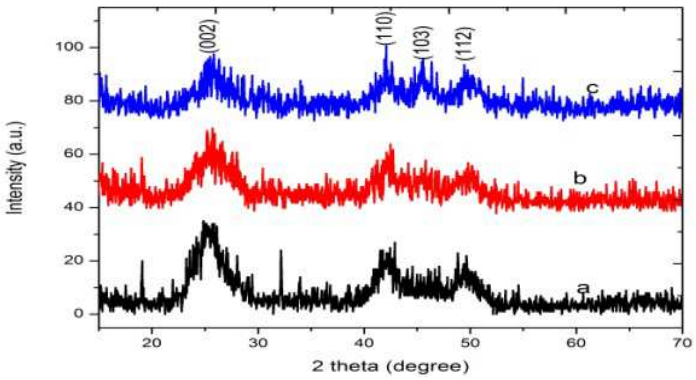

Fig. 1. XRD patterns of (a)CdSe, (b,c)CdSe:Cu nanoparticles.

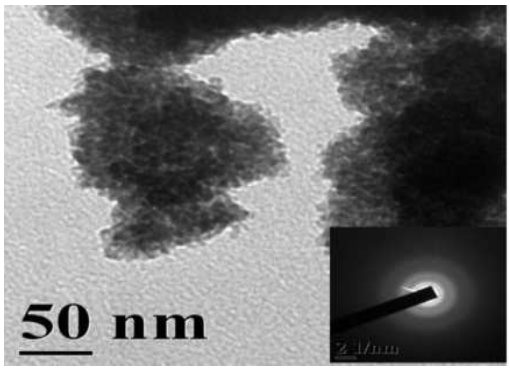

Fig. 2. TEM image

2.2 Optical Studies

Fig.4. showed the absorption spectra CdSe nanocrystals. It showed an Band to Band absorption at 601(2.06 eV), 563(2.06 eV), 561(2.211 eV) for pure, 1% and 3% CdSe:Cu nanoparticles shows a blue-shift compared with bulk CdSe 716 (1.74 eV) of bulk CdSe. These blue-shifting were caused by strong quantum confinement effect. Fig.4. shows emission spectra of pure CdSe and Cu-doped CdSe nanoparticles were recordered at excitation wavelength of 400 nm. The spectra show a broad emission peak around 600-700 nm. Sharp peak were observed at 675 nm for CdSe whereas peak at 694 nm for doped CdSe nanoparticles are due to red emission. The peak position of CdSe:Cu nanoparticles slightly shifts towards the longer wavelength compared with the undoped CdSe nanoparticles, from 675nm to 694 nm. When Cu ions were doped into CdSe nanoparticles more defect states will be introduced. Therefore, it is reasonable peaks had appeared in the longer wavelength side. Comparison of the two spectra, the impurity (Cu) doped CdSe spectra was red shifted from the undoped CdSe.

2.3 Conclusion

It can be concluded that we have successfully synthesized copper doped wurtzite CdSe nanoparticles by the chemical method with inexpensive and nontoxic precursors. The use of 1-thioglycerol as the surfactant and low processing temperature under this synthetic route aids the formation of wurtzite Cu-doped CdSe.

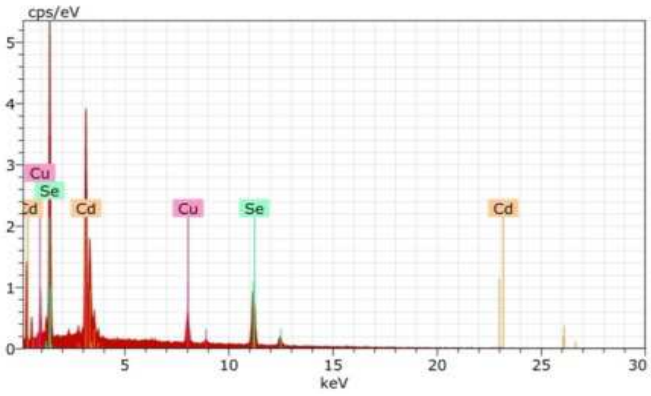

Fig.3. EDAX spectrum

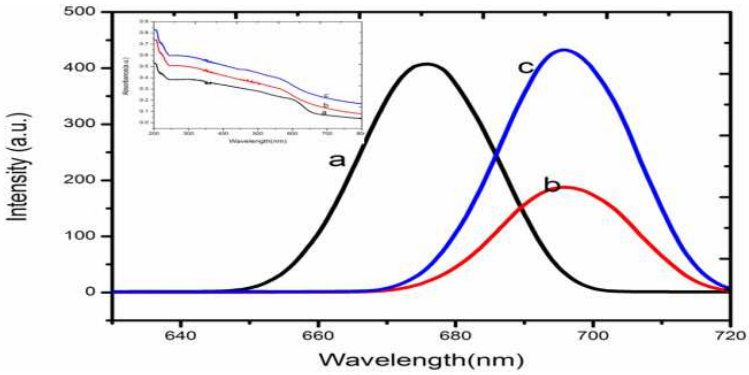

Fig. 4. UV-vis spectra and Photoluminescence spectra of (a)pure, (b ,c) CdSe:Cu nanocrystal.

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