

Synthesis, Structure and Magnetic Characterization of Mn-Doped CdS Nanoparticles

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Abstract

Manganese doped cadmium sulfide (CdS:Mn) nanoparticles with varying weight percentage 2, and 10% have been synthesized by wet chemical reaction route under argon atmosphere using mercaptoethanol as a capping agent. The obtained nanoparticles are characterized by X-ray diffraction, energy dispersive X-ray (EDX) and vibrating sample magnetometer (VSM) technique. The crystal size estimated from (XRD) of the prepared NPs was about 4-6 nm with cubic zinc blende structure. VSM data of Mn doped CdS nanoparticles show weak hysteresis loops at room temperature indicating the superparamagnetic behavior that can be change to ferromagnetic by cooling the samples to 20°K.

Keywords: CdS:Mn nanoparticle; Diluted Magnetic Semiconductors

Introduction

Nanomaterials can be defined as materials whose structural have dimensions ranged from 1 to 100 nm. Diluted magnetic semiconductors are a semiconductor which doped by magnetic ions. These materials have many optical and magnetic properties. The most commonly used DMS are II–VI semiconductors (CdSe, ZnS, CdS, etc.), doped with elements (e.g. Gd, Tb, Eu, Mn, Fe) substituting their host cations. The unique properties of these particles results from the interaction of the spin induced magnetic moments of the transition metal ions with the electronic bands of the host semiconductor lattice^[1]. In the present paper, wet chemical method has been used to synthesis Mn doped cadmium sulfide nanoparticles caped with mercaptoethanol. Effect Mn doping percentage on structure and magnetic properties has been studied.

Experimental Procedures

Materials: Cadmium Chloride (CdCl₂) 99.5%, Sodium Sulfide Nonohydrate (Na₂S.9H₂O) 99%, Merceptoethanol (ME); (C₂H₅OSH) and Manganese Chloride (MnCl₂) 99% were obtained from Aldrich. All chemicals and solvents were used as received without additional purification. Mn doped CdS nanocrystals prepared by wet chemical route method^[2]. Mercaptoethanol was used as capping agent for the synthesized nanoparticles.

Preparation of Mn Doped CdS Nanoparticle: The water used in the preparation was distilled, deoxygenated, and deionized. The deoxygenating was done by bubbling with argon gas. For doping, an aqueous solution of MnCl₂ was injected directly to the solution of CdCl₂ on the reaction flask and refluxed under Ar atmosphere at 100°C. There after 0.01 M solution of Na₂S.9H₂O was injected drop wise into the solution. The reaction can be determined in the following equation.



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The weight percentage of Mn was fixed by varying x value to get Mn doped CdS NPs (2 and 10 wt %). Samples were prepared by varying the volume of Mn precursor and keeping volumes sodium sulfide constant. The solution of the product samples was precipitated then washed several times with distilled water and dried in vacuum oven at 70°C. Finally, the dried powder was grounded. Ar gas and glove bag are very important and used to prevent Mn oxidation during the experimental. The as prepared samples; Mn doped CdS NPs (2 & 10 wt %) were coded as S1, S2, respectively.

Results and Discussion

Energy Dispersive X-ray Spectroscopy (EDX) Analysis: The typical EDX spectra of Mn doped CdS NPs (2 and 10 wt %) are shown in Figure 1. The quantitative weight percentage of the compositional elements Cd, S and Mn in the prepared samples are given in (Table 1).

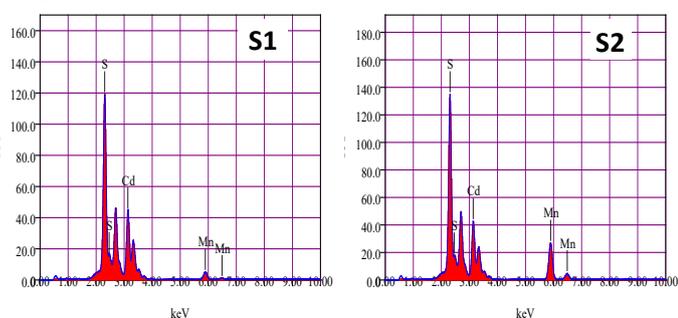


Figure 1: Energy dispersive x-ray spectra (EDX) of (S1) CdS:Mn (2 wt %), (S2) CdS:Mn (10 wt %).

Table 1: EDX compositional analysis of the prepared samples.

S.Code	As prepared samples	Elements from EDX analysis in wt %		
		S	Cd	Mn
22.58	75.55	1.87	CdS:Mn (2 wt %)	S2
21.83	68.71	9.46	CdS:Mn (10 wt %)	S6

The EDX spectrum of Mn doped CdS NPs indicates chemical purity of the samples. The strong peaks related to Cd, S and Mn are obtained in the spectrum. It clearly shows that the intensity of Mn peaks increases with the more addition of Mn concentrations. The deviation of the estimated elements from the target elements were within acceptable limits and attributed to variations in compositional homogeneity. Therefore, the EDX spectra shows good agreement with the experimental Mn concentration used in synthesized NPs.

Structural Characterization: (Figure 2) displays diffraction pattern of Mn doped CdS NPs. X-ray diffraction (XRD) spectra were recorded at room temperature using Philips PW 1710 diffractometer with Cu-K α radiation with $\lambda = 1.5406 \text{ \AA}$ and diffraction angle 2θ from 10° to 90° . The three diffraction peaks were noticed on all samples at 2θ values close to 26.45° , 43.88° and 51.97° degrees. The peaks are appearing due to reflection from the (111), (220) and (311) planes of the cubic phase of the CdS. The XRD pattern of the prepared NPs is well matched with the standard cubic CdS (ICCD card No. 04-006-3897). No other

phases have been observed. The observed broadening of all diffraction peaks confirms that, the products are in nanosized scale. The calculations of samples are presented in (Table 2).

The crystallite size of the samples was calculated from the three obtained diffraction peaks, by using the Sherrerequation^[3];

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

where λ is the x-ray wavelength, θ the Bragg diffraction angle and β the full width at half maximum of the peak. The particle size for Mn doped CdS NPs (2 and 10 wt %) were found to be 6 and 4 nm respectively. XRD patterns show that there is no change in the phase upon the incorporation of Mn in the host CdS lattice. That can be explained according to Vegard's law; the dopant alone cannot produce an individual peak by the side of host peak but it can generate shift in the position of host peak. In addition, the 2θ position peaks is found at 26.43° and 26.68° degree showing slightly increasing shifts to the larger angles with the increment of the Mn concentration. The small shifts in the position of peaks confirm the incorporation of Mn²⁺ into the CdS lattice. The lattice parameter (a) of cubic zinc blende are calculated for the prepared NPs by using the Bragg's law ($2d_{hkl} \sin\theta = n\lambda$), where (θ) is the peak position, (λ) is the wavelength of X rays, (n) is the order of diffraction (usually $n = 1$) and (d_{hkl}) is the inter planer separation according to Miller indices hkl which defined from the next two equations:

$$\frac{1}{d_{hkl}^2} = \frac{h^2 + k^2 + l^2}{a^2} \quad (2)$$

$$a = \frac{\lambda}{2 \sin \theta} \sqrt{h^2 + k^2 + l^2} \quad (3)$$

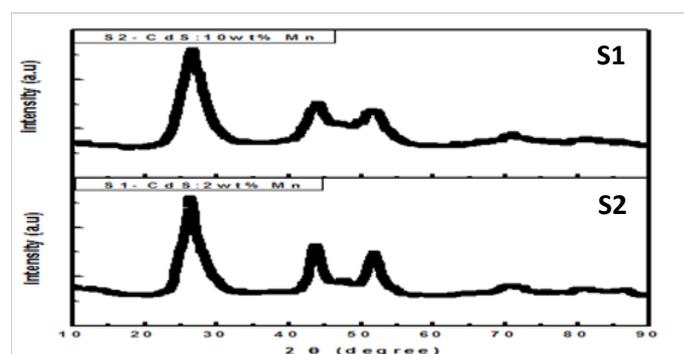


Figure 2: XRD pattern of (S1) CdS:Mn (2 wt %) and (S2) CdS:Mn (10 wt %).

Table 2: XRD results for synthesized NPs.

Sa mp. code	Samp. type	$2\theta^\circ$	FW HM	(hkl)	d spacing	Av. Particle size (nm)	Lattice Parameter (a) (nm)	Cell volume (nm) ³
S1	CdS:Mn (2 wt %)	26.43	2.96	(111)	0.3369	6	0.583	0.1981
S2	CdS:Mn (10wt%)	26.68	3.40	(111)	0.3338	4	0.578	0.1931

It is important to notice that the unit cell lattice volume is decreasing considerably by increasing Mn concentration; (Table 2). This can be explained by the incorporation of smaller Mn^{+2} ions (0.66 Å) replacing larger Cd^{+2} ions (0.97 Å) in cubic host semiconducting CdS lattice structure^[4].

Magnetic Characterization

(Figure 3) shows the magnetic hysteresis (M–H) loops at RT for Mn doped CdS NPs (S1&S2). For S1 (2 wt % Mn), superparamagnetic (paramagnetic with weak ferromagnetic) behavior is observed at room temperature.

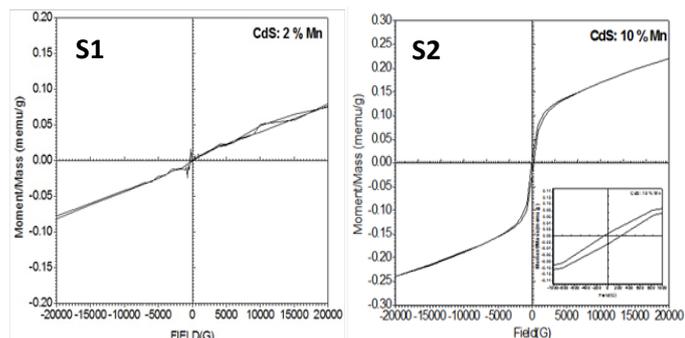


Figure 3: (M–H) curves measured at RT for CdS:Mn NPs: S1) 2 wt % Mn and S2) 10 wt % Mn

At figure (S2) when the amount of Mn concentration reaches 10%, the M–H curve transits into S-type ferromagnetic hysteresis with saturation magnetization M_s equal to 0.021 memu/g and a coercive field of 580 G, indicating that the Mn doped CdS nanocrystals do become ferromagnetic even at room temperature. (Figure 4) shows the (M–H) curves measured at 20 K for Mn doped CdS NPs (2&10 wt %). The field dependent magnetization (M–H) curves for CdS:Mn nanoparticles show clear hysteresis loops at 20K temperature. These obvious hysteresis loops, indicating ferromagnetic ordering at low temperature.

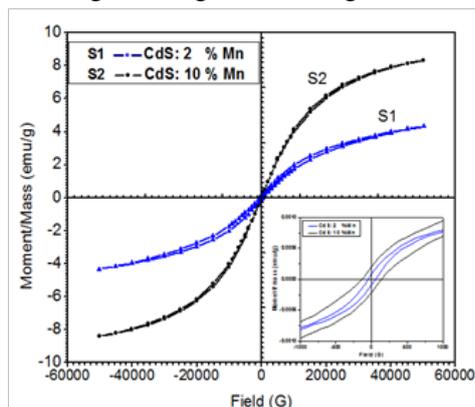


Figure 4: (M–H) Hysteresis loops measured at 20 K for CdS:Mn (2 & 10%) NPs. Inset, expanded curve.

The magnetic properties of the prepared CdS:Mn samples depend on the magnitude of the transition metal ion exchange coupling with the electronic levels. The cubic zinc blende structure for CdS:Mn differ significantly with the bond structures between the magnetic ion and its Next-Nearest-Neighbor (NNN) cations.

Conclusion

Nanoparticles of manganese doped cadmium sulfide (CdS:Mn) with (2 and 10) weight percentage have been synthesized under argon by wet chemical reaction route using mercaptoethanol (ME) as capping agent. XRD revealed the cubic phase structure of Mn doped CdS nanoparticles with average crystallite size ranged from ~ 4 to ~ 6 nm. VSM data reveals superparamagnetic behavior (weak hysteresis loop) in Mn doped CdS samples at room temperature and ferromagnetic at 20 °K. The origin of magnetization has been discussed in this paper. All the prepared samples have semiconducting behavior and can be used in dilute magnetic semiconductor applications and fabrication of solar cells and light emitting diodes^[5-10].

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