Analysis of the Influence of Mn$^{2+}$ Incorporation on Structural and Spectroscopic Features of CdS Nanoparticles

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Abstract--- Nanocrystalline bare and Mn$^{2+}$ doped CdS with different concentration have been synthesized by employing a simple chemical co-precipitation method. In order to gain crystalline nanoparticles of CdS and CdS:Mn; cadmium acetate, sodium sulphide and manganese (II) acetate have been utilised as the supply of cadmium, sulphur and manganese (Mn) respectively. DMF has been used as the stabilizing agent and also the reaction catalyst. The structural analysis of the prepared samples by X-ray diffraction (XRD) exhibits the Mn$^{2+}$ dopant atoms are incorporated at the Cd sites and authenticate the cubic phase, without affecting the original cubic phase of CdS and the particle size estimated to be around 3 to 5 nm. The scanning electron microscopy has been employed to study the morphology of the prepared samples. UV-Vis spectral analysis validates the significant blue shift in the optical band gap of CdS nanoparticles on account of the quantum confinement impact. The optical band gap of the samples is found to be decreasing with the accumulation of dopant content. The presence of functional groups in the prepared samples has been detected by making use of the FTIR technique. The PL emission spectrum shows two major emission peaks one in the blue region and second in the red region along with one broad lower intense peak in the IR region.

Keywords---CdS:Mn nanoparticles, XRD, SEM, FTIR, optical band gap and PL.

I. INTRODUCTION

Nanoparticles behaves substantially different from their bulk materials by the reason of their fascinating optical, electronic, electrical, magnetic, structural, chemical features and their plenty of potential applications. Among the variety of II-VI nanosized semiconductor, synthesis of CdS nanoparticles with a direct band gap of 2.42 eV and doped with transition metal and rare earth ions motivated many researchers in the recent years, owing to its excellent physical and chemical features, nonlinear optical response, photocatalytic activity and its suitability for the broad technological applications in LEDs, solar cell, optoelectronic devices, optical data storage and as a excellent luminescent material [1-5]. Research on CdS nanostructures has a great deal of interest owing to their unbelievable size dependent features and the quantum confinement impact in metal nanoparticles. These nanoparticles offer the new approach for designing next generation energy conversion devices. Mn$^{2+}$ doped CdS has been the most comprehensively studied luminescence activator in II-VI group semiconductor nanoparticles. Mn$^{2+}$ doped CdS nanoparticles are motivating because of the fact that Mn$^{2+}$ ions gives good traps for the excited electrons, which gives rise to their potential use in nonlinear optics, electronic and optoelectronic devices [6-7]. The impurity states play a precise job in affecting the electronic energy structure and transient probabilities by doping the nanoparticles. In doped CdS nanoparticles, the luminescence quantum efficiency is expected to increase as a result of greater interaction between electrons and holes of the host CdS nanoparticles with localized dopant levels [8-9]. The several synthesis approaches have been employed by numerous researchers and scientific groups for growing and also controlling the size and shape of CdS and CdS:Mn nanoparticles. These techniques include hot injection method, sol-gel technique, reverse micelles techniques, photochemical growth, chemical colloidal techniques, hydrothermal synthesis, and chemical precipitation route and so on [10-14]. Among these synthesis techniques, chemical precipitation route is simple, cheapest, high yield producing and low-priced equipments are needed, and therefore, an attempt would have been made to employ this simple
synthesis technique for the preparation of CdS and CdS:Mn nanoparticles in this work. This analysis is focused on the preparation, structural and spectroscopic characterization of Mn$^{2+}$ doped CdS nanoparticles by the cost effective simple chemical precipitation technique. The as-prepared samples were analyzed by employing XRD, SEM, UV-Visible Spectrometer, FTIR and PL spectrometer to recognize the structure, morphology, optical band gap energy, presence of templates and photoluminescence performance respectively. Organization of this research paper has been done into five distinct sections: Introduction, Related work, Materials and Methods, Results with Discussion and Conclusions. Introduction section provides the brief review of literature related to our present investigation. Related work section elaborates on some work done by the various researchers on structural and optical features of CdS:Mn nanoparticles. Materials and methods section contains the materials used for the synthesis of CdS:Mn nanoparticles, details of experimental procedure as well as the characterizations techniques employed for the analysis of prepared CdS:Mn nanoparticles by chemical co-precipitation route. Results and Discussion section provides a brief details on the various characterization of CdS:Mn nanoparticles and the analysis. Conclusion section provides the major conclusions drawn from the results.

II. RELATED WORK

There are some reports on the influence of Mn$^{2+}$ incorporation on the structural and spectroscopic features of CdS Nanoparticles. Some of the related works reported earlier by various workers are Joseph R. Lakowiez et al. 1999 [15] reported the luminescence spectral properties of CdS nanoparticles. M. V. Artemyev et al. 2001 [16] studied the luminescence properties of Mn doped CdS nanoparticles, Ageeth A. Bol et al. 2003 [17] reported temperature dependent luminescence and luminescence measurements for the nanocrystalline CdS/Mn$^{2+}$, V. Sivasubramanian et al. 2006 [18] studied the annealing effect on the optical properties of CdS nanoparticles, Muhammad Arshad Kumaran et al. 2014 [19] reported the microphotoluminescence band red shifts with individual and multi- Mn (II) ion emissions within CdS microwires, Xiaqin Gao et al. 2015 [20] successfully obtained industrial fabrication of high quality Mn-doped CdS core/shell nanocrystals for white light emitting diodes with a simple and scalable approach, A. Gadalla et al. 2018 [21] reported the effect of Mn doping on the structural, optical and magnetic properties of CdS doped magnetic semiconductor nanoparticles.

III. MATERIALS AND METHODS

A. Experimental

All the required precursors and chemicals such as cadmium acetate [(CH$_3$COO)$_2$Cd, 2H$_2$O] (extra pure Loba Chemie), manganese (II) acetate [(CH$_3$COO)$_2$Mn, H$_2$O] (Merck) and sodium sulphide [Na$_2$S, H$_2$O] (extra pure Loba Chemie) and DMF (CH$_3$NCO)H (Merck) for synthesizing the bare CdS and CdS: Mn nanoparticles, by employing the cost effective chemical precipitation technique were purchased from the market with the highest purity. All the reagents used in the present analysis were of analytically pure (AR) grade and used as received with no further refinement. The deionised water was used throughout the experiment as the solvent for all the solutions referred in this analysis.

CdS:Mn powder samples were synthesized by chemical route using their acetate salts. 0.1 M cadmium acetate [Cd$^{2+}$] solution was made by dissolving appropriate weight of cadmium acetate in 100 ml double distilled water in a beaker and 0.1 M sodium sulphide [S$^{2-}$] was also made freshly by dissolving appropriate weight of Na$_2$S in 100 ml double distilled water in a conical flask. Then 2, 5, 10 and 15 at % manganese (II) acetate [(CH$_3$COO)$_2$Mn, H$_2$O] was simply added to cadmium acetate solution and stirred continuously to mix the solution. Then specific amount of DMF was added in the mixture and stirred for 20 minutes. 100 ml freshly prepared sodium sulphide solution was then added in the mixture drop wise with constant stirring for 3 hours. A change in colour of the solution was observed and the yellow colour solution was formed. This solution was kept overnight to form the precipitate. Later on, it was centrifuged and washed several times with the deionised water and acetone to get rid of the un-reacted molecules. The obtained precipitate was filtered, dried in vacuum oven at 60$^0$ for 8 hours and then converted into the fine powder using mortar and then collected in a sample bottle for the analysis and the characterizations.

B. Characterization of CdS:Mn nanoparticles

The structural examination of CdS and CdS:Mn nanoparticles has been performed using X-ray powder diffractometer (Model: D-8 Advance) with CuKα radiation ($\lambda = 0.15406$ nm) scanning 20 in the range 10$^0$-80$^0$. The X-rays were produced using the sealed tube and the wavelength of X-ray was 0.1541 nm. The X-rays were detected using a fast counting detector based on Silicon strip technology (Bruker Lynx Eye detector). The morphology of the nanoparticles was observed through scanning electron microscopy (SEM) using the model JEOL JSM 5600. UV-Vis absorption spectrum was recorded using Jasco spectrometer, (Model name: V-770, Serial No. A013161801) for the wavelength range 200-800 nm. FTIR spectra were recorded using Bruker, Germany. Model: Vertex 70 with resolution 0.5 cm$^{-1}$. The PL measurements were carried out using the model: FP-8200 FL Spectrophotometer, Serial number: C026661448 using the Xe lamp as the light source at the scan speed of 200 nm/min.
IV. RESULTS AND DISCUSSION

A. Structural studies of CdS and CdS:Mn nanoparticles

Figure 1 demonstrates the XRD blueprint of all the prepared samples of pristine and Mn$^{2+}$ doped CdS. The percentage of Mn doping is (2, 5, 10 & 15 at %). The XRD blueprint of pristine and CdS:Mn samples exhibits three prominent, very broad remarkable diffraction peaks appearing around 20 values of 26.68$^0$, 43.57$^0$ and 52.06$^0$, which might be referring to scattering from (111), (220) and (311) crystal planes respectively which reveals a face-centred cubic crystal structure of CdS which are in consistent with the standard JCPDS database (89-0440).

![Figure 1: XRD pattern of CdS and CdS:Mn nanoparticles](image)

Any significant phase transformation to CdS host due to incorporation of Mn$^{2+}$ doping is not detected. The peak broadening is fairly large due to Mn$^{2+}$ incorporation, as obtained in the XRD spectra. It gives the significance of the reduced particle size with increased Mn$^{2+}$ concentration. The peak broadening in the diffraction pattern is a sign of the presence of the nanosized particles. The intense and the sharp peaks suggest that the nanoparticles are well crystallized. Also, it is seen that the relative intensities of (111), (220) and (311) diffraction are varying in Mn doped CdS nanoparticles. This might occur due to replacement of Cd$^{2+}$ by Mn$^{2+}$ at different lattice sites of CdS. The average particle size for all the samples has been estimated by applying Debye-Scherer’s principle [22, 23], taking into account the most intense peak.

\[
D = \frac{0.9\lambda}{\beta \cos \theta}
\]

Where \( \lambda = 0.1541 \) nm is the wavelength of X-ray diffraction, \( \beta \) is the FWHM in radian of the most intense XRD peak and \( \theta \) is the angle of diffraction. The grain size of the as-prepared samples determined using XRD found to be in the range of 3 - 5 nm.

The lattice parameter ‘a’ for CdS and CdS: Mn nanoparticles is determined using equation

\[
a = \frac{\lambda}{2 \sin \theta} \times \sqrt{h^2 + k^2 + l^2} \ \text{Å}
\]

The d-spacing for cubic system for 20(111) is calculated by using equation

\[
d = \frac{a}{\sqrt{h^2 + k^2 + l^2}} \ \text{Å}
\]

The microstrain calculated using equation

\[
\varepsilon = \frac{8 \cos \theta}{4}
\]

are listed in table-1.

![Table-1 : The structural parameters of undoped and Mn doped CdS samples](image)

| Samples        | Peak position (2θ) (degrees) | FWHM (β) (degrees) | Lattice parameter (a) in Å | Microstrain | Interplanar spacing (d) in Å for 2θ(111) |
|----------------|-----------------------------|-------------------|---------------------------|-------------|----------------------------------------|
| CdS (Pure)     | 26.6849                     | 1.59735           | 5.8077                    | 0.3885      | 3.3530                                 |
| CdS:Mn (2%)    | 26.6494                     | 2.4006            | 5.7939                    | 0.5839      | 3.2699                                 |
| CdS:Mn (5%)    | 26.5563                     | 2.3996            | 5.8141                    | 0.5838      | 3.3656                                 |
| CdS:Mn (10%)   | 26.6835                     | 2.47268           | 5.7889                    | 0.6014      | 3.2659                                 |

B. Morphological study (SEM)

The TEM morphological studies of pure CdS are reported in our earlier work [25]. The surface morphology of pure and Mn$^{2+}$ doped CdS samples have been noticed using Scanning Electron Microscope (SEM). The SEM micrographs for Mn$^{2+}$ (10% & 15%) doped CdS samples are depicted in figure-2(a) and 2(b). The SEM images show that the surface morphologies are in the form of assemblies of nanoparticles and seen aggregation of the particles. The synthesized particles are nearly spherical shaped and the grain size is decreased with the increasing of Mn$^{2+}$ concentration.

![Figure 2 (a): SEM image of CdS:Mn (10%)](image)
C. FTIR Analysis

Figure-3 represents the Fourier Transform Infrared spectra of CdS and CdS: Mn nanoparticles recorded in the range 400-4000 cm\(^{-1}\) to obtain the details about the presence of chemical bonding in the prepared samples. The KBr pellets technique has been employed to trace the FTIR spectra of prepared CdS and CdS:Mn samples at room temperature. A broad absorption band observed in the higher energy region at 3415 cm\(^{-1}\) has been attributed to O-H stretching vibration of H\(_2\)O molecule indicating the existence of water absorbed on the surface of the nanoparticles. The peak appeared at 1550 cm\(^{-1}\) is attributed to very weak bending vibration of water molecule [26]. The peak at 1406 cm\(^{-1}\) is due to C-H stretching mode vibration [27]. A strong peak at 1113 cm\(^{-1}\) occurs due to C-O stretching vibration [28]. The peak at 611 cm\(^{-1}\) and 677 cm\(^{-1}\) which have been attributed to vibrations of Cd-S bond [29].

D. Optical analysis of CdS and CdS:Mn nanoparticles

It is widely known that semiconductor nanoparticles made from metal chalcogenides (II-VI) exhibit a remarkable change in their optical absorption features when their sizes are reduced below a certain threshold dimension. Hence, the influence of Mn\(^{2+}\) addition on the optical band gaps of nano dimensional CdS particles has been investigated by employing UV-Visible absorption spectroscopy. The optical absorption behaviour of all the prepared CdS and CdS: Mn samples at room temperature have been depicted in figure 4(a). The material of the present study is of the direct band gap temperament. The bulk band gap of CdS is 2.4 eV as reported by earlier workers [30, 31]. The optical band gap is related to the absorbance and the photon energy by the following Tauc relation [32].

\[
(\alpha h\nu)^n = A (h\nu - E_g)
\]

Where ‘\(\alpha\)’ is absorption coefficient, ‘A’ is constant related to the effective masses associated with the bands, ‘h\nu’ is the energy of photon and ‘\(E_g\)’ is the band gap of the material. The exponent n depends on the type of transition.

For the direct allowed transition n = ½, indirect allowed transition n = 2, direct forbidden transition n = 3/2, indirect forbidden transition n = 3 [32]. To determine the possible transitions, \((\alpha h\nu)^2\) against hv is drawn and the corresponding band gaps were evaluated by extrapolating the linear section of the curve on the energy axis. The direct band gap value of CdS sample have been estimated from \((\alpha h\nu)^2\) vs hv plot as displayed in figure-4(b).
The absorption edge shifts towards the lower wavelength (higher energy) side with the addition of Mn$^{2+}$ doping concentration. It is observed that the band gap reduces slightly with the insertion of doping concentration which results in the increased conductivity of the material [33]. According to the effective mass approximations (EMA), particle radius of CdS and CdS: Mn samples were estimated using following Brus equation [34, 35], which describes the energy band gap of nanoparticles as a function of particle radius

$$E_{g(nano)} = E_{g(bulk)} + \frac{\hbar^2}{8R^2} \left( \frac{1}{m_e} + \frac{1}{m_h} \right) - \frac{1.9 \epsilon^2}{4\pi\epsilon_0 \epsilon_r R}$$

where $E_{g(nano)}$ = band gap of nanoparticles, $E_{g(bulk)} = 2.4$ eV, $m_e^* = 0.21 m_e$ is the effective mass of electron, $m_h^* = 0.8 m_e$ is the effective mass of hole, $m_e$ is the free electron mass and $R$ is the particle radius, $\epsilon_r$ is the dielectric constant and $\epsilon_0$ is the permittivity of free space. The first term in above equation indicates the confinement effect and the second term is the coulomb term. The second term is small due to strong confinement and can be ignored [32]. The crystalline radius and the optical band gap estimated from UV-Visible absorption spectra for bare and Mn$^{2+}$ doped CdS samples are presented in table-2.

Table-2: Optical band gap and crystalline size of CdS and CdS:Mn samples.

| Samples          | Band gap (eV) | Crystalline size (nm) |
|------------------|---------------|-----------------------|
| CdS              | 4.4           | 2.7                   |
| CdS: Mn (5%)     | 4.3           | 2.78                  |
| CdS: Mn (10%)    | 4.22          | 2.80                  |
| CdS: Mn (15%)    | 4.17          | 2.88                  |

The values of the band gap shown in the above table-2 are larger than the band gap of bulk CdS (2.42 eV) and it is as a result of quantum confinement impact. However, it has been noticed that the optical band gap shrinks with the accumulation of doping quantity. The earlier researchers [36-39] have also been reported similar shrink in the band gap.

E. Photoluminescence Studies:
Photoluminescence (PL) is the process in which an electron excited by monochromatic photon beam of certain energy undergoes radiative recombination either at valence band (band edge luminescence) or at traps/surface state (normally red shifted luminescence) within the forbidden gap. Photoluminescence originates from the recombination of surface state [40, 41]. PL emission measurements of CdS and CdS:Mn (5%, 10% & 15%) nanoparticles carried out at room temperature using Xe lamp as the light source are displayed in figure 5.

The PL spectrum of pure CdS and Mn doped CdS have been obtained at the exciting wavelength of 426 and 436 nm respectively. The PL spectrum exhibits two emission peaks in the visible region. A high intense strong emission peak is found centred at wavelength 492 nm along with two shoulder peaks at the wavelength 482 nm and 468 nm. It found in the blue region and originating from the recombination of electron-hole pair and is attributed to transition from conduction band to valence band. The second peak is at around the wavelength 650 nm which shows well known red emission band in CdS and CdS:Mn nanoparticles. From the PL spectrum it is observed that the intensity increases with doping of Mn (5%). However, the intensity decreases with further increase in doping % of Mn. It has also been observed a broad peak in the infrared region at the wavelength around 826 nm.

V. CONCLUSIONS AND FUTURE SCOPE
CdS nanoparticles doped with various concentration of Mn$^{2+}$ have been synthesized successfully by chemical precipitation route in presence of the organic solvent. The structural characterization done through XRD reveals that
the prepared samples are nanocrystalline in nature which exhibits cubic structure and the average grain size of the particles determined are in the range of 2.5–5 nm. The particle size of the prepared samples is found to be decreasing with increasing Mn doping concentration which is also confirmed by SEM micrograph. The SEM micrograph shows particles are almost spherical, which form aggregation. FTIR analysis reveals the Cd–S stretching at 615 cm\(^{-1}\). The UV-Visible absorption spectrum shows that the band gap of the prepared samples decreases with increasing Mn doping concentration. The blue shift in absorption maxima clearly indicates the quantum confinement of charged particles. The PL emission spectrum shows two emission peaks in visible region, one in the blue region and second in the red region along with one broad and lower intensity peak in the IR region. Thus the prepared material has a good potential to be used in optoelectronics, solar cell applications. We plan to study the effect of another doping material Fe on structural and luminescence properties of CdS nanoparticles as our future work.

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