



Luminescence of Cadmium Sulphide Nanoparticles and Nanocomposites

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Abstract:

Cadmium sulphide (CdS) is an excellent material for optoelectronic applications. It is interesting to investigate the optical properties of CdS at nanometer regime where the properties become size dependent. Presently photo- and electro-luminescence of undoped and manganese (Mn) doped cadmium sulphide nanoparticles as well as their nanocomposite in poly vinyl carbazole (PVK) and poly vinyl alcohol (PVA) matrices have been reported. The CdS and CdS:Mn nanoparticles of different sizes and CdS/PVK and CdS/PVA nanocomposites with different loadings have been synthesized by various chemical methods like chemical bath deposition, chemical encapsulation, organometallic precursor route, embedding in polymer matrix etc. The samples were characterized by TEM, SEM, AFM, XRD, UV-Vis absorption, photoluminescence and electroluminescence analysis. It is observed from AFM, SEM and TEM micrographs that the CdS nanoparticles are irregular in shape. The size is found to increase with reaction time and decrease with increasing capping agent concentration. In case of nanocomposites the particle size of the embedded crystals is found to decrease with increasing the CdS loading. From the XRD analysis of mercaptoethanol capped CdS nanocrystals the structure has been found to be zinc blende with three peaks corresponding to (111), (220) and (311) planes. Mn doping up to 5% has not been found to affect the structure. The CdS prepared by organometallic precursor shows hexagonal structure. The crystal size computed by Debye Scherrer formula is in the range of a few nm. The UV-Vis absorption shows blue shift in the absorption edge and in some cases stepwise absorption which is indicative of size quantization. In case of nanocomposites the absorption edge shifts towards red by increasing CdS loading. The photoluminescence (PL) of pure CdS excited by UV light shows single Stokes shifted peak attributed to second ionization states of sulphur vacancy. The peak becomes intense for smaller CdS nanocrystals. The CdS:Mn gives two peaks. The second peak is due to Mn and its intensity is found to increase with Mn doping. The composites of CdS/PVA give single peak due to CdS states only. The peak intensity is found to increase with increasing CdS loading in the composite. The electroluminescence (EL) studies have shown lower threshold and higher brightness for small CdS nanoparticles. In case of Mn doping EL brightness increases up to 1% Mn and then decreases due to concentration quenching. Mn does not affect the threshold voltage. The EL intensity is found to increase with CdS loading in nanocomposites and then become constant for loading above 30%. In CdS/PVA polymer luminescence is absent but CdS/PVK gives emission due to CdS as well as PVK. The linear current-voltage characteristic indicates ohmic nature and lower impedance in case of smaller nanoparticles. The correspondence between EL and PL spectra shows that these are originated from same states. It is observed that the nanocomposites show better electroluminescence and are suitable materials for large area display devices.

Keywords: Cadmium sulphide, nanocrystals, Nanocomposites, XRD, Absorption spectra, photoluminescence, Electroluminescence

1. INTRODUCTION

Nanostructured materials have been the subject of intense study for last several years, due to their size dependence physical and chemical properties below a critical size- characteristic of the material. Surface atoms have different environment and therefore exhibit different characteristics. Nanoparticles have a large number of surface atoms compared to the bulk, therefore, the characteristics of surface atoms have more contribution towards their properties. Due to finite size of the nanocrystals the continuous energy band of the bulk crystal transforms into a series of

discrete states resulting in widening of the effective band gap. Blue shift in the optical absorption spectra, size dependent luminescence, enhanced oscillator strength and nonlinear optical effect are some of the interesting properties exhibited by these nanocrystals.

Semiconductor nanoparticles have been extensively investigated during the last decade, due to their unique properties and application in diverse areas such as solar cell, display panel and new devices like single electron transistor and so on. Among these, CdS is a widely used substance with many advanced

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technological application. It is a direct band gap material of energy band gap 2.42 eV at 300 K. For nanoparticles, the size and surface effect are both important. By controlling these, it is possible to design materials of required optical, magnetic, elastic and chemical properties. The synthesis of highly monodispersed CdS nanocrystals via size restricting growth modes, eg: by adding surface capping organic materials to the solution has opened the way towards tunable light emitting devices and low voltage display.

The hybridization of organic and inorganic semiconductor is expected not only to permit wide range selection of emitter and carrier transport material, but also provide a new approach to construct high performance electroluminescence (EL) devices taking advantage of high photoluminescence efficiency of organic material and high carrier density and low resistivity of inorganic semiconductors. The incorporation of nanocrystals in polymer is expected to increase the life of the device and enhance the brightness of emission. An attempt has been made here to investigate the photo- as well as electroluminescence of doped and undoped nanocrystalline CdS in powder form as well as in polymer matrix.

2. EXPERIMENTAL

Nanostructures of cadmium sulphide were synthesised by chemical route. Four different types of samples were prepared-

1. CdS nanocrystalline powder
2. Mn doped CdS nanocrystalline powder
3. CdS nanocrystals in PVK polymer matrix
4. CdS nanocrystals in PVA polymer matrix

The samples were characterized by XRD and UV-Vis absorption and their photoluminescence as well as electroluminescence were investigated.

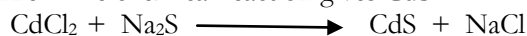
2.1 Synthesis of samples

2.1.1 CdS nanocrystalline powder: Single source organometallic precursor technique was used to prepare nanocrystalline CdS particles. The precursor solution A was prepared by dissolving 36 mg cadmium chloride ($\text{CdCl}_2 \cdot 2\text{H}_2\text{O}$) and 12 mg of thiourea $[(\text{NH}_2)_2\text{CS}]$ in 30 ml ethanol at 60°C in water bath under magnetic stirring. Another solution B was prepared by dissolving 10 mg sodium hydroxide (NaOH) in 10 ml ethanol. Now the solution B was added into solution A. The mixed solution was kept at 60°C under magnetic stirring. In the beginning a white solution was obtained which gradually become transparent and the colour changed from white to green yellow. The

solution samples were extracted from the mixture solution at different reaction times, centrifuged and washed with ethanol and then with acetone, and subsequently spread over glass slides and dried to obtain nanocrystalline CdS powder. Five different samples CdS I, CdS II, CdS III, CdS IV and CdS V were prepared with 20, 30, 60, 80 and 90 min reaction time, respectively.

2.1.2 Mn doped CdS nanocrystalline powder:

The nanoparticles of manganese doped cadmium sulphide have been prepared by chemical route. For synthesis, 10^{-2} mole aqueous solutions of CdCl_2 , mercaptoethanol ($\text{C}_2\text{H}_5\text{SH}$) and Na_2S each, were taken. For doping of Mn, MnCl_2 solution was simply added to CdCl_2 solution. First mercaptoethanol solution was added drop wise to CdCl_2 (+ MnCl_2 for doping) solution at the rate of 1ml per minute, while stirring it continuously so that solutions are properly mixed. Then Na_2S solution was also added in a similar manner. The chemical reaction gives CdS.



CdS is precipitated, being insoluble in water. The presence of mercaptoethanol does not allow the particles size to grow. Four different samples were prepared with Mn concentrations 0.0%, 0.1%, 0.2% and 0.3%. The precipitate thus obtained was thoroughly washed in double distilled water, centrifuged and then air-dried.

2.1.3 CdS nanocrystals in PVK polymer matrix:

For Preparing CdS/PVK composites 400 g of PVK was dissolved in 20 ml DMF by heating the solution. After adding appropriate quantity of cadmium acetate (and MnCl_2 for Mn doping) to this solution so that the CdS loading in polymer could be 1, 2, 5 or 20%, the resulting solution was stirred for about 30 min and then concentrated to about 10 mL. The solution was taken in a flask and degassed several times by supplying nitrogen. To this clear solution of cadmium acetate and PVK, H_2S gas was passed for a few seconds and then flushed out with nitrogen. The dense liquid was then transferred on plane glass slides and also on glass slides coated with SnO_2 and was dried in an oven for several hours. The Mn doping was varied from 0 to 10000 ppm.

2.1.4 CdS nanocrystals in PVA polymer matrix:

For the synthesis of CdS/PVA composite films, 400 mg PVA was dissolved in dimethylformamide (DMF) by heating at 80°C . After proper solution was obtained, cadmium acetate was added to it in appropriate quantity, so that CdS loading in polymer is

2, 5, 10, 20, 30 and 40 % by weight. The resulting solution was stirred for 30 minutes and concentrated to 10 ml. The solution was degassed many times by applying nitrogen and then H₂S gas was passed for 30 second to this clear solution of cadmium acetate and PVA. The solution immediately turned almost yellow. Now again the solution was stirred for a few seconds, and then spread over the plane glass slide and also on SnO₂ coated conducting glass slides and dried in oven to obtain uniform film of CdS/PVA nanocomposite. Here the wt % of cadmium acetate represents the loading of CdS.

2.2 Characterization

The samples were characterized by Transmission electron microscopy (TEM) and X-Ray diffraction (XRD) techniques. In some cases SEM and AFM micrographs were also obtained. The XRD was done by Rigaku Rotating Anode (H-3R) diffractometer with irradiation from K α line of copper ($\lambda=1.5418\text{\AA}$). Optical absorption spectra of the samples were recorded by using Perkin Elmer Lambda-12 spectrometer in the UV/VIS region ranging from 700 nm to 400 nm.

2.3 Photoluminescence

The photoluminescence spectra of CdS nanoparticles and CdS/PVA nanocomposites were recorded by a Perkin Elmer LS-55 fluorescence spectrometer in nanophosphor laboratory, Allahabad. The photoluminescence (PL) of nanoparticles doped with Mn was investigated using grating monochromator HM-104 and photomultiplier tube (PMT- RCA-931). The nanoparticles were placed on a glass slide using toluene and it was fixed onto the entrance slit of the monochromator. Mercury lamp was used for PL excitation. The PL emission intensity in the range of 400-1000 nm was measure with the help of PMT placed at the other slit of monochromator. The photoluminescence spectra of CdS/PVK nanocomposite were obtained using Cary Eclipse Fluorescence Spectrophotometer at delhi University Delhi.

2.4 Electroluminescence

The cells for electroluminescence (EL) investigations were fabricated by depositing emissive layer of CdS nanoparticles dispersed in DMSO or the nanocomposite on SnO₂ coated conducting glass plate. The conducting glass plates were prepared by depositing a layer of SnO₂ over heated glass plates using chemical vapour deposition technique. A thin mica sheet having small window of 2x2 mm² was fixed over emissive layer of CdS nanoparticles. An

aluminium strip was attached over the emissive layer, so as to have a good contact with it. For electroluminescence studies A.C.voltage of various frequencies was applied at conducting glass plate and aluminium electrodes using EL power supply along with audio frequency generator. EL brightness at different voltages and frequencies was measured by a photomultiplier tube connected to a picoammeter.

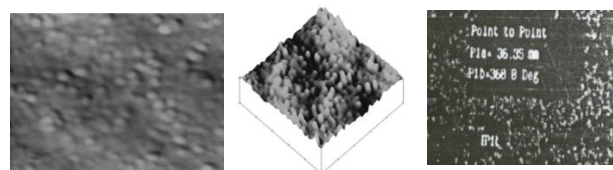


Fig.1 AFM 2–d and 3 –d images of 0.5 % Mn in CdS:Mn nanoparticle, and SEM of CdS nanoparticles

3 RESULTS AND DISCUSSION

It is observed from AFM, SEM and TEM micrograph (Fig 1) that the CdS nanoparticles are irregular in shape. The size is found to increase with reaction time and decrease with increasing capping agent concentration. In case of nanocomposite the particle size of embedded crystals is found to decrease with increasing the CdS loading in polymers.

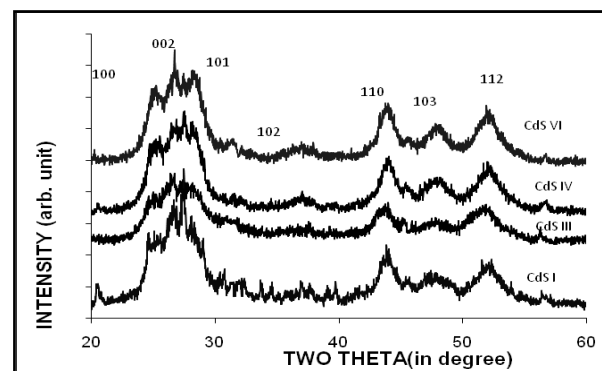


Fig. 2: X-Ray Diffraction patterns of CdS nanoparticles.

3.1 X-Ray Diffraction

The XRD patterns of CdS nanocrystalline samples are shown in Fig 2. From the figure it can be seen that the XRD pattern of CdS V can be consistently indexed on the basis of the hexagonal, wurtzite structure, in which the six prominent peaks at 2θ values of 24.4°, 26.7°, 28.4°, 44°, 47.8° and 51.9° angles corresponds to the reflections at (100), (002), (101), (220) (103) and (112) planes. The weak peak due to (102) plane was also observed. The XRD pattern of CdS III also exhibit hexagonal structure with similar peaks, but the peaks are smaller and broader as compared to CdS V sample. In case of CdS I two extra peaks at $2\theta=27^\circ$ and $2\theta=52.1^\circ$ are

also observed. These respectively correspond to reflections from (111) and (311) planes of cubic structure, but the peaks corresponding to the hexagonal structure are also present. Hence it can be said that the phase of CdS I is mixture of hexagonal and cubic structure.

XRD also indicated that the particle size is reduced with decreasing reaction time period. It is known that the XRD peaks intensity decreases and width increases with decreasing crystalline size.

TABLE 1: Size of CdS nanoparticles by various techniques

Sl. No.	Reaction time (in min)	Size by TEM	Size by XRD	Size by abs. Edge
1	20	5 nm	7 nm	5.7 nm
2	30	-	-	6.6 nm
3	60	7 nm	9 nm	8 nm
4	80	8 nm	-	-
5	90	-	10 nm	10.8 nm

The broadening of XRD peaks can be qualitatively expressed by Debye Scherrer’s formula-

$$D = \frac{K\lambda}{\beta \cos\theta} \quad \text{-----(1)}$$

where D is average crystal size, λ is wavelength of X-rays used. β is the full width at half maxima (FWHM) of the XRD peak, θ is Bragg angle and K is the shape factor, which has the value close to unity. The average crystal size was computed from the broadening of peak using equation (1) and it is given in column IV of Table 1. The structure transform from cubic to hexagonal phase as the crystal size increases.

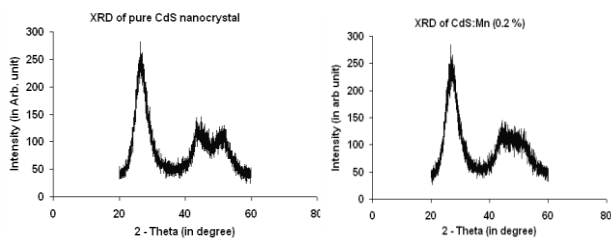


Fig.3: X-Ray Diffraction pattern of CdS:Mn nanoparticle

Figure 3 shows the XRD pattern of CdS:Mn nanocrystals. The analysis of XRD reveals that CdS:Mn nanocrystals have cubic zinc-blende crystal structure. For all the samples three peaks are observed

corresponding to diffraction from (111), (220) and (311) planes. For lower concentration of Mn, three peaks are seen separately, but as the Mn concentration increases second and third peaks corresponding to (220) and (311) planes get merged.

Due to the size effect, the XRD peak tends to broaden and their width increases as the size of crystals decreases. The crystalline size has been estimated from the broadening of the first diffraction peak using equation (1). The average particle size of CdS:Mn nanocrystals has been obtained as 2 nm. XRD gives the regularity in the atomic arrangement in the samples where as TEM gives the full size of the particle including non-crystalline part also.

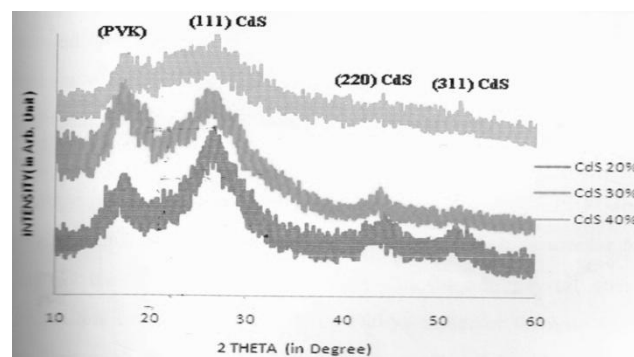


Fig. 4: X-Ray Diffraction pattern of CdS/PVK nanocomposites

The XRD pattern of CdS/PVK nanocomposite is shown in Fig. 4. The peaks at 2θ values of 26.54°, 43.94°, and 52°, matching the (111), (220) and (311) planes of cubic, indicate the formation of CdS. This shows that CdS particles formed in pyridine and polymer media under the microwave irradiation still retained good crystallinity. The broad nature of the XRD peaks could be attributed to the nano-crystalline nature of the CdS particle.

The XRD patterns of CdS/PVA nanocomposite samples are shown in Figure 5. It shows a broad amorphous peak around 2θ = 20° due to PVA. Apart from the broad peak due to PVA, there are three sharper peaks corresponding to (111), (220) and (311) lattice planes of cubic CdS phase (JCPDS card no. 80-0019). The grain sizes of the films corresponding to (111) plane for cubic reflection have been found to decrease with increasing loading percent of CdS in CdS/PVA nanocomposite film.

3.2 UV-Vis absorption

CdS is a direct band gap material, which has the band gap energy ~ 2.4 eV with absorption edge at about

515 nm. The optical absorption spectra CdS nanocrystalline powder samples are shown in Figure 6. From the figure it is clear that the absorption edge for all the samples is blue shifted as compared to that of bulk CdS. For CdS V sample, the absorption edge is nearly equal to that of the bulk CdS. The blue-shift in

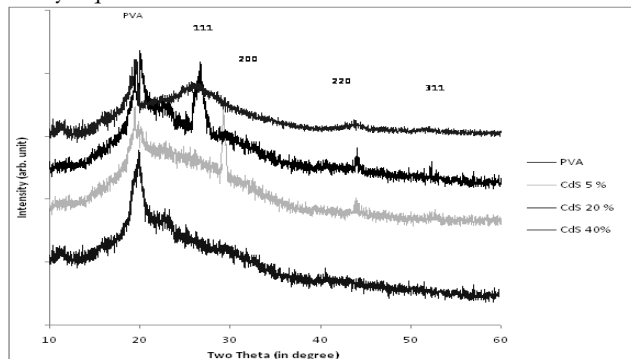


Fig.5 X-Ray Diffraction pattern of CdS/PVA nanocomposites

the absorption edge indicates increase in effective band of the samples. The increase in band gap in nanoparticles due to quantum confinement has the quantitative form:

$$\Delta E_g \equiv E_g(nano) - E_g(bulk) = \frac{h^2}{8MR^2} \quad \text{-----(2)}$$

where h is Planck’s constant, R is the radius of the particle and M is the effective mass of the system. Equation (2) can be used to estimate size of the particle taking proper value of M and increase in band gap obtained from blue shift in absorption edge. For hexagonal CdS, $M=1.919 \times 10^{-31} \text{Kg}$. The absorption edge for samples and their particle size from equation (2) are shown in column V and VI of Table 1. The red shifting in absorption edge by increasing reaction time reveal that the size of the CdS nanoparticles was increased; this is in good agreement with XRD and TEM, observation. Mn doping does not affect the absorption spectra of CdS nanoparticles

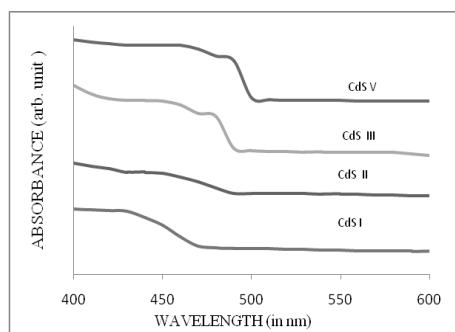


Fig. 6: Absorption spectra of CdS nanocrystals

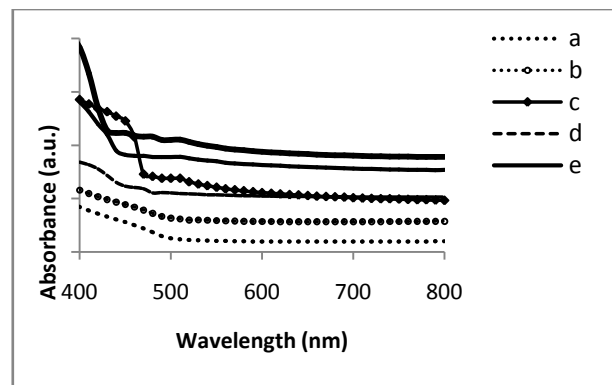


Fig.7: Absorption spectra of CdS/PVA nanocomposites

The absorption spectra of the CdS/PVA nanocomposite are shown in Figure 7. It is seen that the absorption edge wavelength is blue - shifted with higher loading of CdS in PVA polymer. The decrease in the particle size with higher loading of Cd in PVA polymer can be understood as higher the cadmium content a greater proportion of Cd atoms are available for surface reaction with the PVA - a situation resulting in the stabilization of smaller particles.

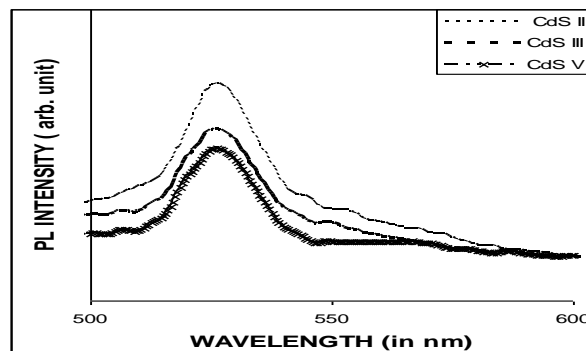


Fig. 8: Photoluminescence Spectra of CdS nanocrystals

3.3 Photoluminescence

Photoluminescence (PL) studies provide information of different energy states available between valence and conduction bands responsible for radiative recombination. Figure 8 shows PL spectra of nanocrystalline CdS samples. Photoluminescence was excited by 390 light. In the PL spectra of CdS nanocrystals, band edge luminescence was not detected. One PL peak has been observed at 528 nm. This peak can be attributed to the second ionization state of the sulphur vacancy. The peak becomes intense for smaller particles. In case of large CdS clusters the Stoke shifted luminescence is attributed to mid-band gap states. These indicate larger Frank-Condon displacement in the excited states. As the cluster size decreases, the increased band gap makes

certain defect states energetically accessible, i.e. the number of surface states increases, thus increasing luminescence intensity with smaller particles.

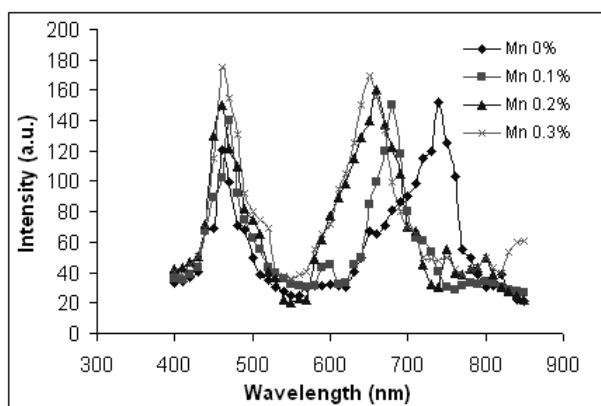


Fig. 9: Photoluminescence of CdS:Mn nanocrystals

Photoluminescence of CdS:Mn shows two prominent peaks. The PL spectra for various concentrations of Mn are shown in Figure 9. It is seen that first peak (A) is obtained at 465 nm and the other peak (B) is found between 750 to 650 nm for different Mn concentrations. Both PL peaks exhibit significant Stoke's shift with respect to absorption edge (3eV). Hence the luminescence bands can be identified with transitions involving donors, acceptors and surface states; and photoexcited electrons decay to these levels by some nonradiative processes.

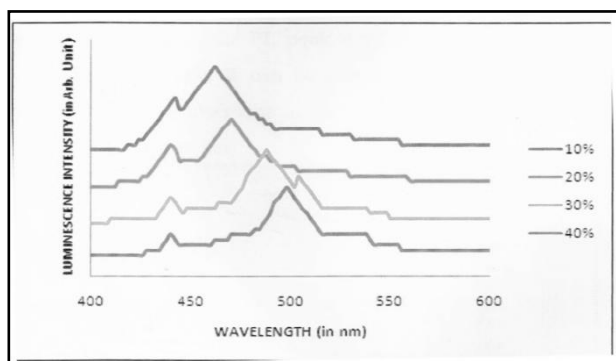


Fig. 10: PL Spectra of CdS/PVK nanocomposites

In case of CdS/PVK nanocomposites, the photoluminescence was excited by 400 nm light. Single PL peak was obtained which shifts towards higher wavelength by increasing CdS loading in PVK as shown in Figure 10. The PL peak occurs at about 465, 470, 488 and 498 nm for 10, 20, 30 and 40% CdS/PVK nanocomposite samples, respectively. The other smaller peak obtained at 440 nm is due to PVK.

In case of CdS/PVA nanocomposites photoluminescence was excited by 450 nm. The

variation of the fluorescence spectra with different loading of CdS is shown in figure 11. The spectral behaviour of the luminescence is characterized by broad band with a maximum at 535 nm and a long tail towards higher wavelength. A very efficient luminescence has been attributed to Cd²⁺ ion excess for CdS nanoclusters. Here, the cadmium rich CdS/PVA composite films only affect the intensity of luminescence. Variation in excitation wavelength in PL studies of composite film has no effect on the emission maxima.

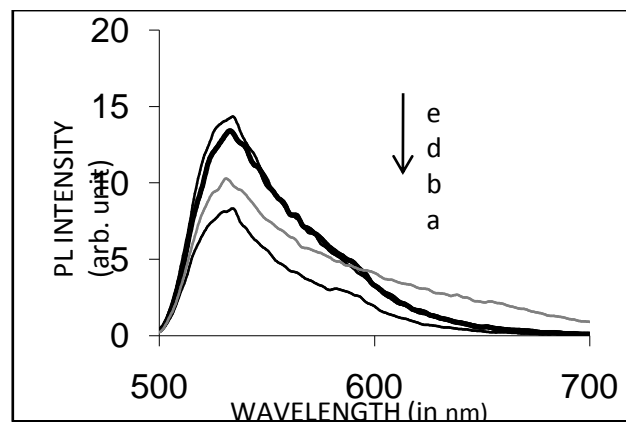


Fig. 11: PL spectra of CdS/PVA nanocomposite films (a -2%, b -5%, d -20% and e -30%)

3.4 Electroluminescence

In the electroluminescence, a high electric field enables injection of charge carriers at the interface of emission layer and electrodes. The injected carriers say electrons are accelerated and cause impact ionization and excitation under the influence of the strong interfacial electric field. They move to the other end of the crystalline particle and recombine with opposite sign carriers at the luminescence centres giving rise to the light emission. The set up for measurements of electroluminescence is shown in figure 12.

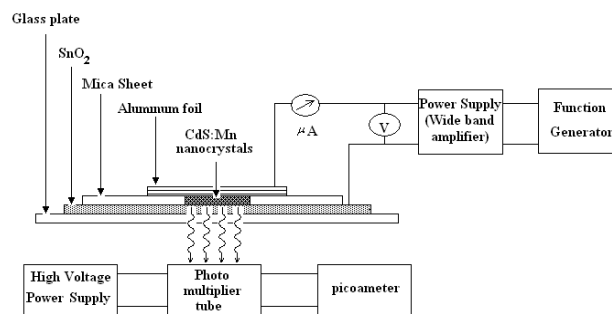


Fig.12 Set up for Electroluminescence

Figure 13 shows EL brightness versus voltage curve for nanocrystalline CdS powder samples. It is

observed that EL starts at a threshold voltage and then increases first slowly and then rapidly with increasing voltage. The lower threshold and higher brightness have been observed for samples prepared from shorter reaction time. As voltage is increased, more electrons and holes are injected into the emission layer and their subsequent recombination increases the EL brightness. For shorter reaction time smaller CdS nanoparticles are obtained having increased oscillator strength, which improves the electron-hole radiative recombination and enhances the electroluminescence. It is observed that at higher frequencies, light emission starts at lower threshold voltages, and electroluminescence brightness increases with increasing frequency. The nature of frequency dependence of EL brightness can be understood on the basis that the emptying and detrapping of EL centres more rapidly with increase in frequency. The linear relation between current and voltage indicates the ohmic nature. Similar results are obtained in the samples presently investigated. Such an ohmic behaviour can be attributed to hopping conductivity of electrons through nano-granules

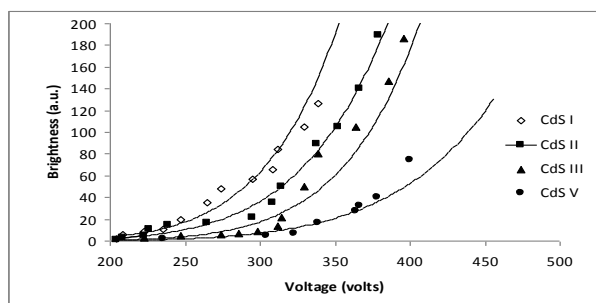


Fig. 13: Brightness- Voltage curve of CdS Nanocrystals

The EL brightness is increased by Mn doping. The variation of EL brightness with Mn concentration at various frequencies is shown in figure 14. It is clear from figure 10 that on increasing the percentage of Mn doping, the EL brightness increases up to 1.0 % but for higher percentage of Mn, there is concentration quenching and the EL brightness is reduced. The maximum brightness is observed for the sample of 1.0 % Mn doping. It is also seen that Mn does not influence the threshold voltage, but it affects only brightness.

Figure 15 shows the brightness vs voltage characteristics at a particular frequency for CdS/PVK nanocomposites. It is seen that the emission starts at a particular threshold voltage and then the brightness increases rapidly with the applied voltage. Mn doping increases EL brightness up to 3000 ppm (i.e. 0.3%)

and for higher doping it is reduced due to concentration quenching. Perhaps interaction between Mn ions is more due to presence of PVK and

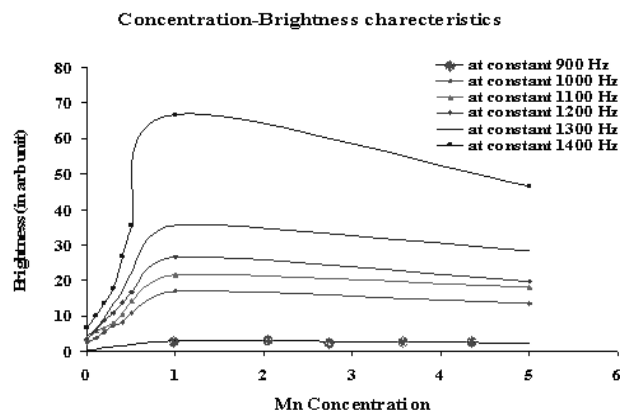


Fig. 14: Variation of EL brightness with Mn Concentration in CdS nanoparticles

luminescence is reduced at lower Mn concentration as compared to CdS:Mn nanoparticles. In case of CdS/polymer nanocomposites, EL brightness increases very fast with the voltage. It is observed that at a particular frequency and voltage, the EL brightness first increases with increasing concentration of the CdS nanocrystals in PVK matrix and then attains saturation value for more than 10 % CdS.

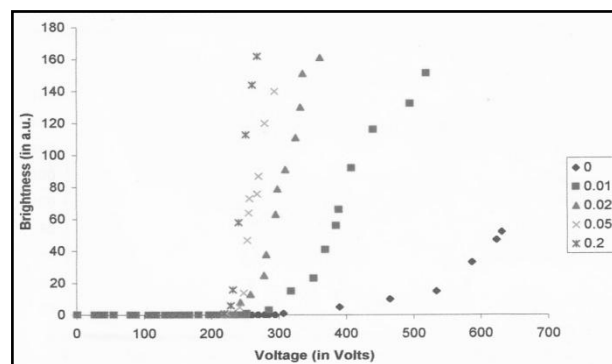


Fig. 15: Brightness- Voltage curve of CdS/PVK Nanocomposites

In case of CdS/PVA, the variation of EL brightness with voltage is shown in Figure 16. Higher concentration of CdS in polymer shows intense emission due to recombination process of excited states on the impurity centres. Figure 17 shows electroluminescence spectra of CdS/PVA nanocomposite film of CdS loading of 30 and 40%. The emission peak is observed at 535 nm for 30 % loading CdS in PVA, same as in case of photoluminescence, suggesting a multimodal particle size distribution with higher loading of CdS in

polymer, which indicate smaller particle size with wider size distribution and could be full of defects. The enhanced emission from CdS in PVA could be due to a possible chemical bonding between CdS surface and hydroxyl group of PVA. This cures the defects at the surface and improves electron hole recombination. The PVA polymer luminescence is absent in EL studies.

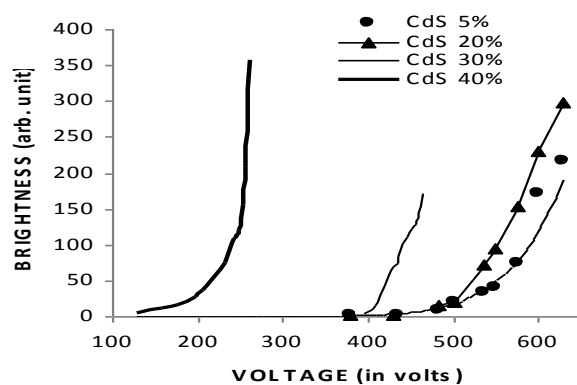


Fig.16: Brightness- Voltage curve of CdS/PVA

In fact, we have observed the high field EL in CdS nanoparticles. In this case bending of bands takes place and release of electrons from traps to the conduction bands and their subsequent recombination with the hole centres give rise to the light emission. If electron moving in the conduction band recombines with holes in valence band, then edge emission will be observed. On the other hand, if the electron moving in the conduction band recombines with the holes trapped in impurity centres, then the light emission characteristics of the defect centres will be observed.

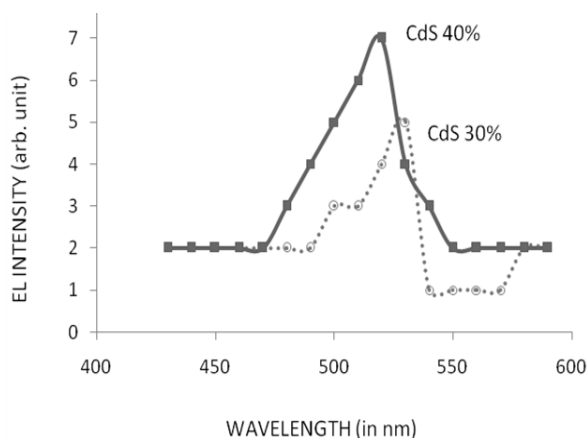


Fig.17: Electroluminescence spectra of CdS/PVA nanocomposite

4 CONCLUSIONS

The investigations have shown that properly passivated cadmium sulphide nanocrystals can be used in luminescent devices. The correspondence between photoluminescence and electroluminescence indicates that these are originated from same states. It is found that nanocrystalline powder shows better photoluminescence but for electroluminescence, nanocomposites are more suitable for large area devices.

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