



## **Surfactant-assisted synthesis and characterizations of Cadmium complex for organic light emitting diode applications**

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**Abstract :** In this paper we are discussing about the synthesise of pure and guest surfactant (TTAB) assisted cadmium [(2-(2-hydroxyphenyl) benzoxazole) (8-hydroxyquinoline)] Cd(HPB)q nanorods and their characterization. Powder X-ray diffraction analysis was used to calculate the particle size via Scherrer equation. Fourier- transform infrared (FTIR) spectroscopy was utilized to confirm the presence of all the functional groups of Cd(HPB)q. Scanning Electron Microscope (SEM) images indicated that lower temperature and a shorter reaction time would be suitable for the formation of nanorods. UV-vis-NIR spectroscopy was used to determine the band gap energies of Cd(HPB)q complexes. Photoluminescence (PL) spectra showed a prominent peak around 500 nm which indicates a strong PL emission in the green region.

**Keywords :** OLED, XRD, SEM, nanorods.

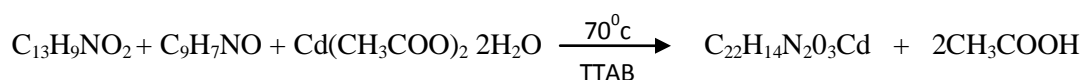
### **Introduction**

In organic light emitting diodes (OLEDs), green light emitters have attracted much attention due to their stability and brightness [1, 2]. The need for stable and efficient light emitting molecules becomes obvious due to the growing market of organic light-emitting diodes. OLEDs with low molecular weight electroluminescent materials are promising candidates for lighting applications due to their low power consumption, wide viewing angle and ease of fabrication [3, 4]. These small molecules had been synthesized by many groups by taking different metals like aluminum (Al), zinc (Zn), iridium (Ir), gadolinium (Gd), europium (Eu), platinum (Pt) etc., along with different ligands, like 2-(2-hydroxyphenyl)benzoxazole(HPB) 8-hydroxyquinoline(q), 1,10 phenanthroline (Phen), 2,2'bipyridine(Bpy) etc., [5-7]. Nowadays cadmium complexes have been synthesized by different groups for their use in OLED application due to their thermal stability, simplicity in synthesise procedure and wide spectral range. Extensive research work is presently going on to synthesise new cadmium complexes containing new ligands to produce a number of novel luminescent cadmium complexes as emitters and electron transporters in OLED applications [8-10]. The coordination numbers of cadmium complexes are variable, making it possible to synthesise new emitter materials with varying optoelectronic properties [11-13]. In this paper we have discussed about the synthesis of cadmium [(2-(2-hydroxyphenyl) benzoxazole) (8- hydroxyquinoline)] Cd (HPB)q by a simple novel method and the effects of surfactant TTAB ( 0.5 %) on its structural, morphological, optical and photoluminescence (PL) properties.

## Experimental details

### Synthesis Procedure

A solution of 2-(2-hydroxyphenyl) benzoxazole (HPB) with 0.211 g (1 mmol) (Merck, India) was prepared in 20 mL of absolute ethanol in a 100 mL three neck flask and stirred with a magnetic stirrer for one hour at a constant temperature of 70°C. Then the solution of cadmium acetate dihydrate 0.267 g (1 mmol) in 3 mL of water and 8-hydroxyquinoline (q), 0.145 g (1 mmol) in 10 mL of absolute ethanol was added drop wise in the first solution under stirring. After the addition was completed, the reaction was further carried out for 3 h. The yellow precipitate was collected by filtration by acetone and ethanol wash, and dried overnight. The above preparation procedure was followed in synthesizing the complex by adding surfactant TTAB of 0.5% before the addition of cadmium acetate and 8-hydroxyquinoline in the HPB solution. The reaction scheme for the synthesis of Cd(HPB)q is as shown below [14].



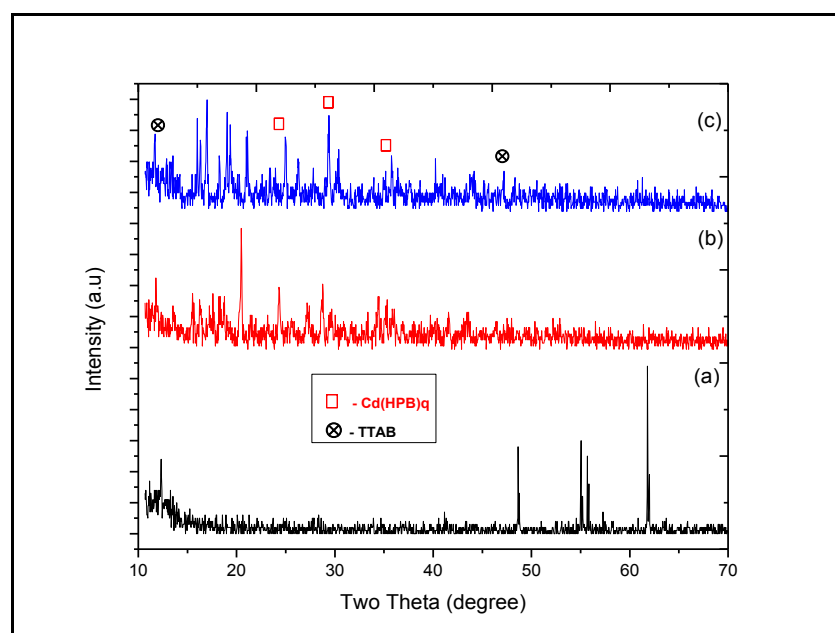
### Characterizations

The room temperature powder X-ray diffraction study for the prepared samples was carried out using RICHE SCHIFER X-ray diffractometer with monochromatic  $\text{CuK}\alpha$  ( $\lambda = 1.5418 \text{ \AA}$ ) radiation. The FTIR spectra was recorded on NICOLET 5700 FT-IR in SPECTROMETER the range  $4000 \text{ cm}^{-1}$  to  $400 \text{ cm}^{-1}$ . Scanning electron microscope (SEM) was employed for morphological study using JEOL JSM 6310 SCANNING ELECTRON MICROSCOPE operated at 15 kV with energy dispersive X-ray analyzer (EDAX). The UV-vis-NIR absorption spectral study was carried out using VARIAN CARY 5E UV-vis-NIR SPECTROPHOTOMETER in the spectral region of 200 to 800 nm.

The photoluminescence spectra of the samples were recorded with a VARIAN CARY 5E SPECTROPHOTOMETER.

## Results and Discussion

### Powder X-Ray Diffraction analysis



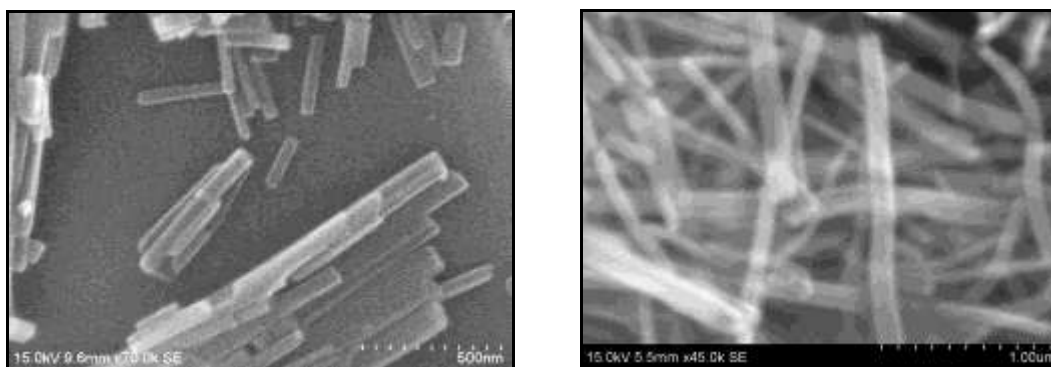
**Figure 1** PXRD spectra of (a) TTAB, (b) Cd(HPB)q and (c) TTAB assisted [Cd(HPB)q]

The recorded PXRD spectrum is shown in Figure 1. The peaks, in Figure 1(c), corresponding to  $2\theta$  values of 24.7, 28.3 and 36.4 degree ascertain the phase of cadmium complex and the  $2\theta$  values corresponding

to TTAB were observed at 11.68 and 47.33 degree [15]. From this spectrum TTAB assistance in the sample is ascertained from the two prominent peaks and explains the incorporation of the same. In powder X-ray diffraction analysis the prominent peaks observed were used to calculate the particle size via the Scherrer equation. The particle size was found out to be 68 nm, 54 nm for pure Cd[HPB]q and TTAB assisted Cd[HPB]q respectively.

### SEM analysis

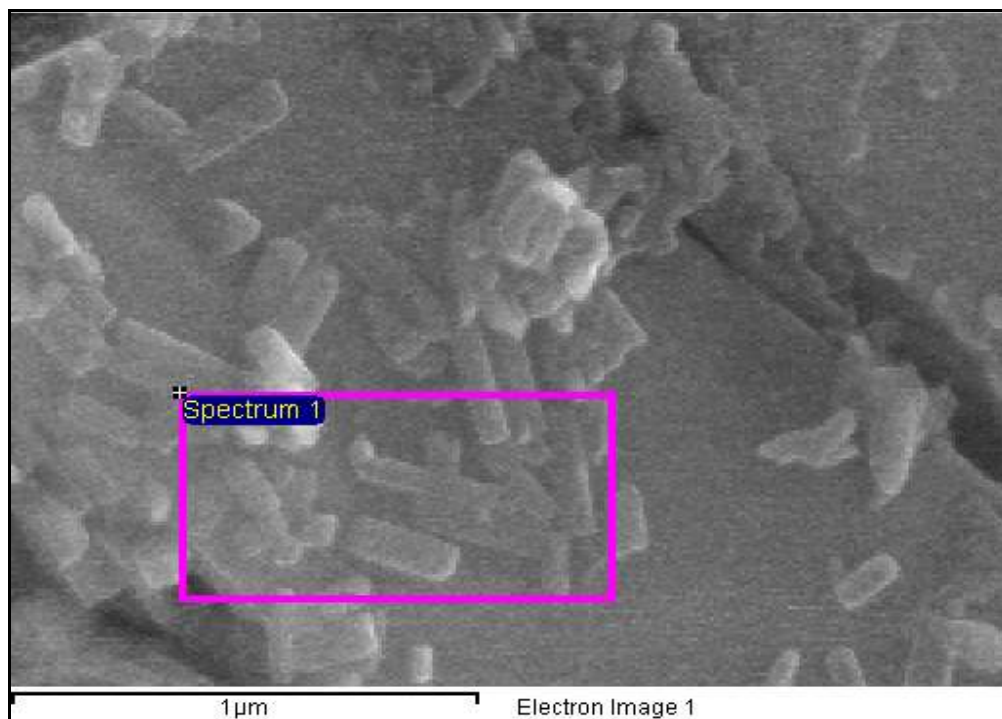
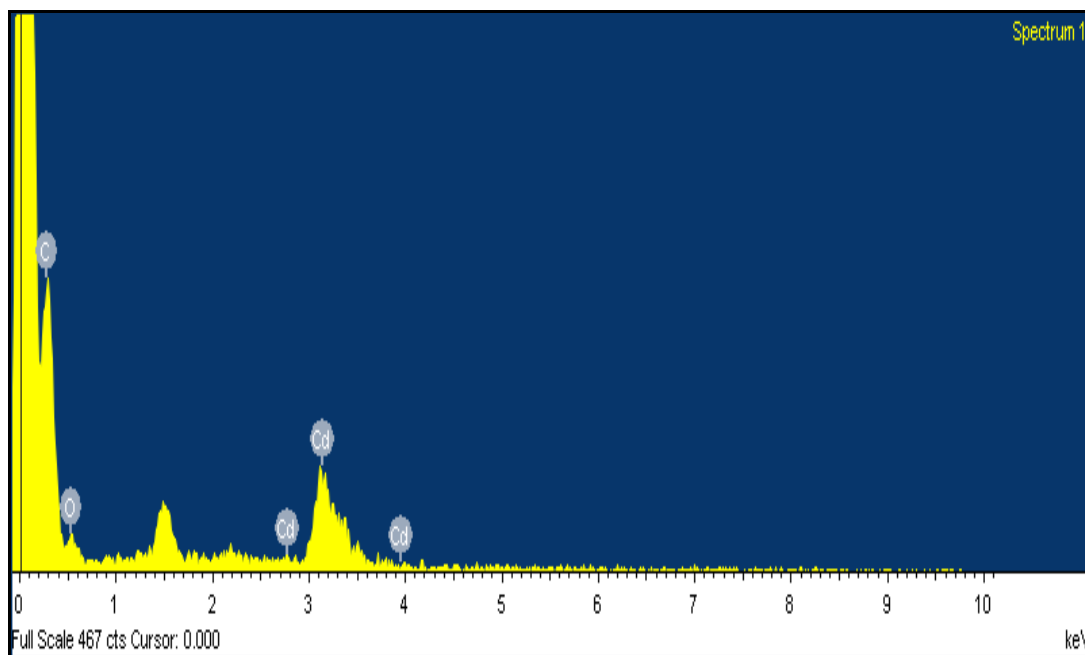
Figures 2(a, b) show the SEM micrographs of as synthesized pure and TTAB assisted cadmium complexes. The pure cadmium complex is composed of branched aggregates of nano rods and the close observation of the SEM image suggests that the surfaces of the nanorods are relatively smooth and this could be attributed to the relatively low reaction temperature employed. It unveils that in the formation process of TTAB assisted cadmium complex the cationic surfactant is well absorbed on to the different planes of the cadmium complex and prevents the agglomeration of the cadmium complex.



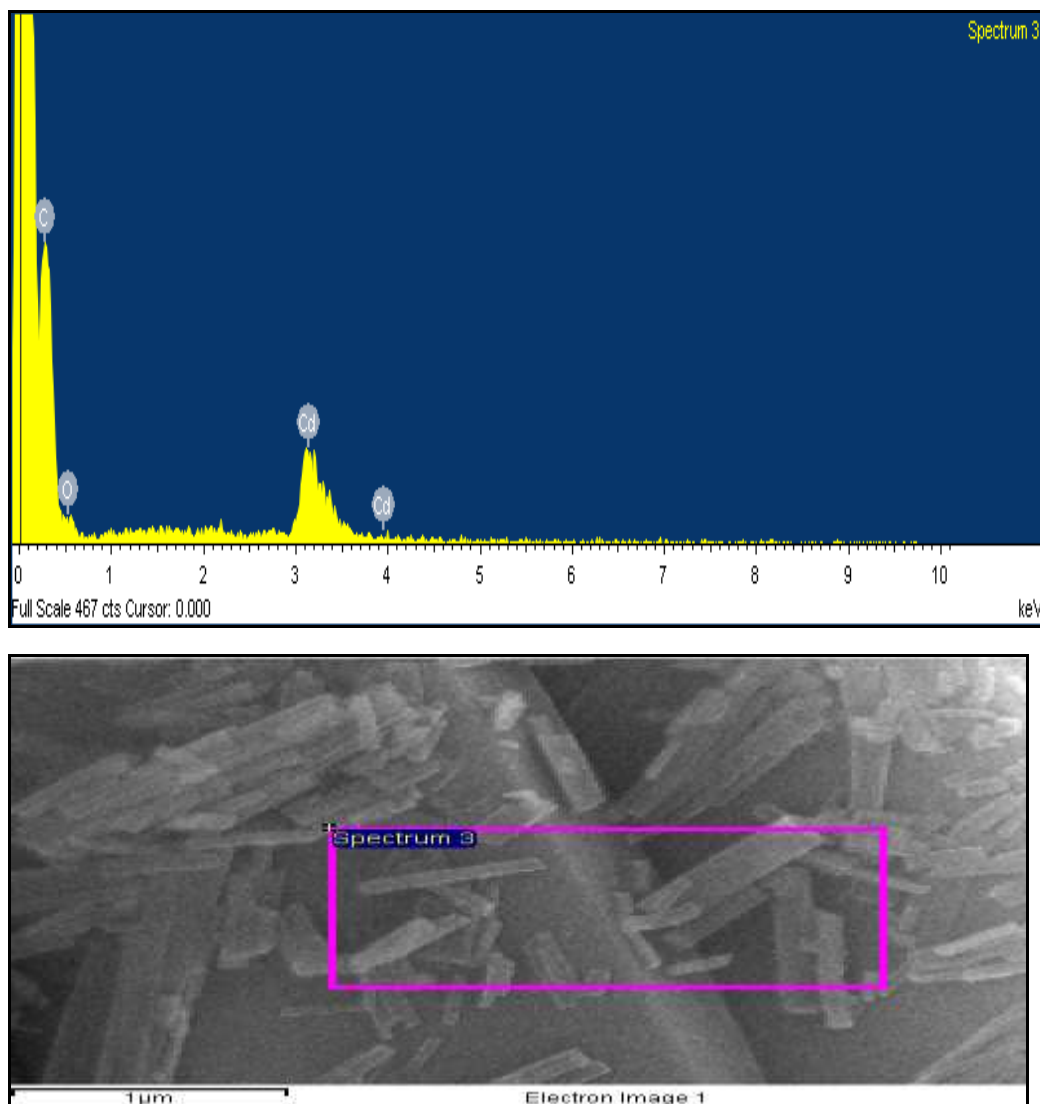
(a) (b)  
Figure 2 SEM micrographs of (a) pure, (b) TTAB assisted cadmium complexes [Cd (HPB) q]

### EDAX analysis

EDAX is an important technique to analyze the composition of elements quantitatively and solve the chemical identity of any nanomaterial. It is inferred from the result of the EDAX spectra, shown in Figure 3(a,b) that the pure sample shows the signs of carbon at 0.2 eV, oxygen at 0.6 eV and cadmium at 2.8, 3.2 and 4 eV. It also shows that in the TTAB assisted sample the presence of carbon is at 0.3 eV oxygen at 0.6 eV and cadmium at 3.2 and 3.8 eV. Both the samples are composed of only cadmium and no trace of other elements is observed. Though, two different species were employed, the peaks related to organic surface is not visible and it is clear that the samples are generally cadmium rich.



(a)



(b)

Figure 3 EDAX spectra of (a) pure and (b) TTAB assisted cadmium complexes [Cd(HPB)q]

### FTIR analysis

The FTIR spectra of the synthesized cadmium complexes, prepared as pellets containing Cd (HPB)q powder dispersed in KBr powder, are shown in Figure 4. The FTIR spectra were recorded in the range  $4000\text{ cm}^{-1}$  to  $400\text{ cm}^{-1}$ . In the case of Cd(HPB)q the main characteristic bands are observed at  $1600\text{ cm}^{-1}$ ,  $1570\text{ cm}^{-1}$ ,  $1495\text{ cm}^{-1}$  for the aromatic phenyl ring. The band at  $1321\text{ cm}^{-1}$ ,  $1279\text{ cm}^{-1}$  correspond to C-N-C. The band at  $1106\text{ cm}^{-1}$  is assigned to C-O bond stretching. The band between  $760 - 735\text{ cm}^{-1}$  represent the C-H out of plane bending vibration for the four adjacent hydrogen atoms. The bands at  $570\text{ cm}^{-1}$  and  $493\text{ cm}^{-1}$  correspond to Cd-O and Cd-N bond stretching, respectively [16]. The surfactant assisted cadmium complex has the same with some extra peaks which can be attributed to the chelates present in the complex.

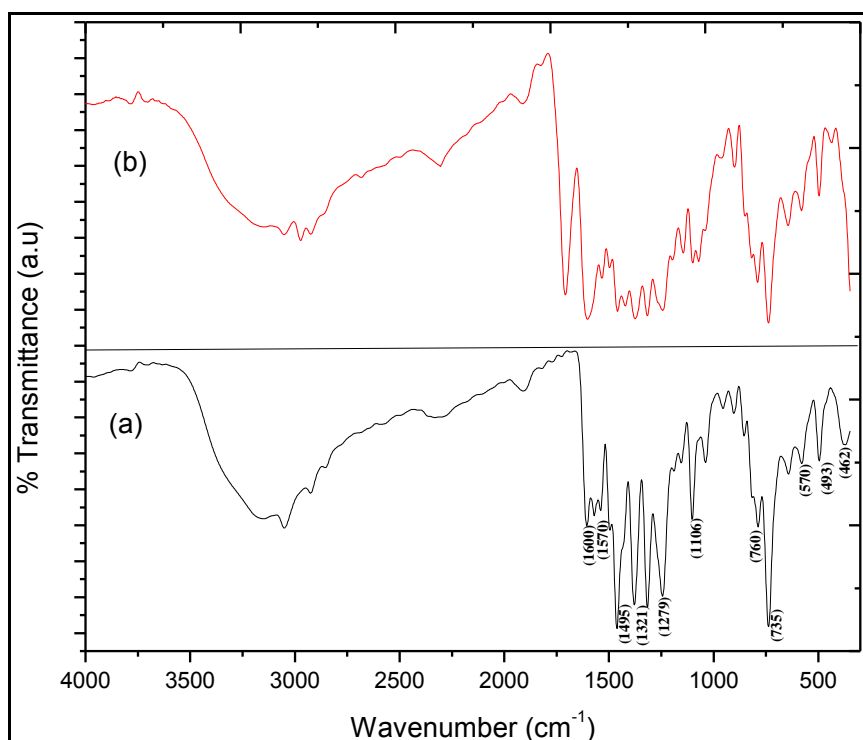


Figure 4 FTIR spectra of (a) pure, (b) TTAB assisted cadmium complexes [Cd(HPB)q]

Table 1 FTIR assignments of Cd(HPB)q

Functional group	Wave number (cm <sup>-1</sup> )
Aromatic phenyl ring	1600,1570,1495
C-N-C	1321,1279
C-O	1106
C-H	760-735
Cd-O	570
Cd-N	493, 462

### UV-vis-NIR analysis

The controlling and tuning of band edge emission and surface traps state emission of Cadmium complex are obviously very important to realize the tunable optical properties. The UV-vis-NIR spectral analysis was carried out between 200 nm and 800 nm. Figure 5 shows the absorption spectra of cadmium complex pure and surfactant assisted nanorods. The relatively sharp absorption indicates the narrow size distribution of the nanorods. The TTAB assisted Cadmium Complex, the absorption band edges are shifted to 380 nm and the corresponding band gap is 2.97 eV. Thus it is clear from the optical absorption study that the assisted Cadmium complex with organic surfactant of TTAB modifies the band gap of the complex and the sample is blue shifted in the spectrum when compared to the pure complex.

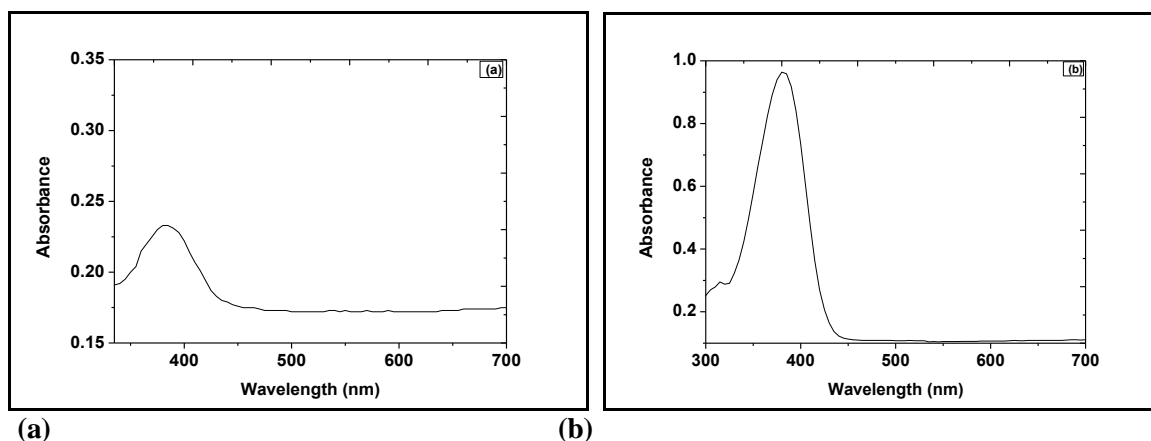


Figure 5 Absorption spectra (a) pure and (b) TTAB assisted cadmium complexes [Cd(HPB)q]

### Photoluminescence analysis

The PL studies on cadmium complexes were made and the recorded spectra are shown in Figure 6. Cd(HPB)q with TTAB showed very similar photoluminescence characteristics with that of pure cadmium complex. The yellow coloured complex with cationic surfactant exhibited a single emission band ranging from 500-540 nm respectively. It has been observed that the presence of a emission peak at  $\sim 503$  nm which indicated a strong emission in the green region.

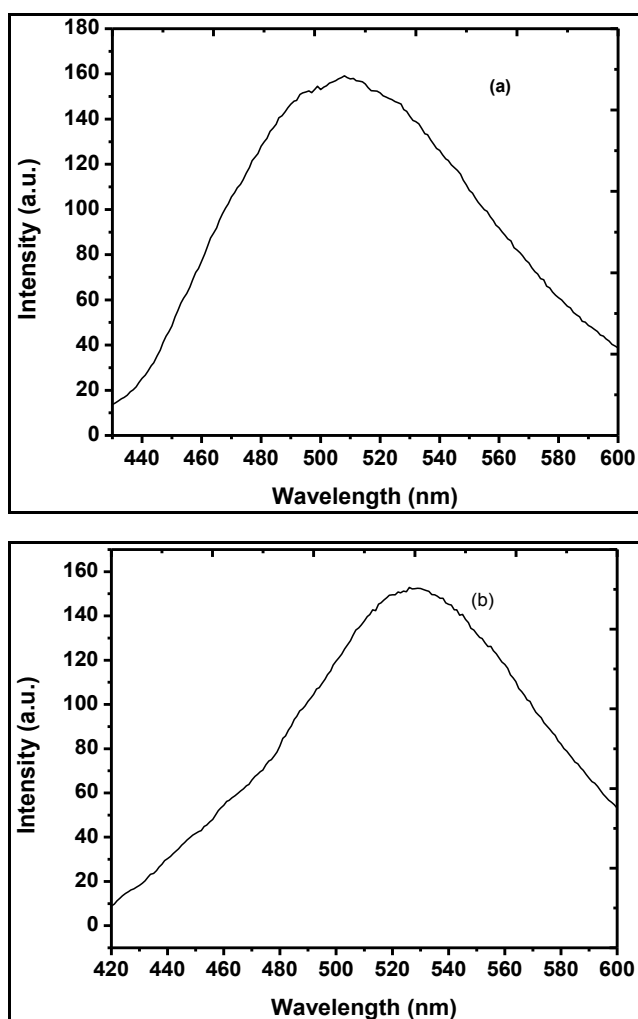


Fig. 6 PL spectra of (a) pure and (b) TTAB assisted cadmium complexes [Cd(HPB)q]

## Conclusion

A novel method has been handled in synthesizing pure and TTAB assisted cadmium complex by the reaction of 8-hydroxyquinoline and 2-(2-hydroxyphenyl) benzoxazole with cadmium acetate. The peaks corresponding to 20s 24.7, 28.3 and 36.4 ascertain the phase of cadmium complex. . A close observation of the SEM image of the present case suggests that the surfaces of the nanorods are relatively smooth and this could be attributed to the relatively shorter reaction time and lower temperature employed. Though, two different organic species were employed, the peaks related to organic surface are not visible and it is clear that the samples are generally cadmium rich. In case of Cd(HPB)q main characteristic band are observed at  $1600\text{ cm}^{-1}$ ,  $1570\text{ cm}^{-1}$ ,  $1495\text{ cm}^{-1}$  for the aromatic phenyl ring,  $1321\text{ cm}^{-1}$ ,  $1279\text{ cm}^{-1}$  for C-N-C and at  $1106\text{ cm}^{-1}$  for C-O bond stretching. The relatively sharp absorption indicates the narrow size distribution of the nanoparticles. The TTAB assisted Cadmium Complex, the absorption band edges are shifted to 380 nm and the corresponding band gap is 2.97 eV. The photoluminescent spectra showed a prominent peak around 500 nm which indicated a strong PL emission in the green region.

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