

Synthesis of ZnO-CdO Spherical Nanocomposites Via Co-Precipitation Process

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Abstract

A spherical like morphology of ZnO/CdO nanocomposites were prepared by co-precipitation method at 400°C. The as-synthesized samples were analyzed by using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), Ultraviolet spectroscopy (UV-vis), Photoluminescence spectroscopy (PL), Scanning electron microscopy (SEM) and Transmission electron spectroscopy (TEM). The XRD and FTIR results are showed that the formation of the ZnO/CdO nanocomposite. The SEM and TEM images are showed that the ZnO/CdO nanocomposites comprise of spherical particles in the size range of 20-30 nm. The UV-vis and PL spectra revealed that the optical properties of prepared ZnO/CdO nanocomposites. In particular, the PL emission spectra showed an enhanced higher emission peak of the ZnO/CdO nanocomposites.

Keywords: Semiconductor; Composites; ZnO/CdO; Precipitation route; Optical properties

1. Introduction

In recent years, the semiconducting metal oxide nanomaterials are used for photocatalytic activity and antibacterial applications owing to its unique physicochemical properties and environmental stability when compared than bulk. Among the metal oxides, Zinc oxide (ZnO) is the suitable photocatalyst material compared with other semiconductor materials, because of its wide band gap ~ 3.37 eV with large exciton binding energy of 60 meV at room temperature, environmental stability and low cost. Further, it has been widely used for the photocatalyst in the treatment of waste water and gas sensing [1-3] and also it is used for many industrial applications due to its optical property, and magnetic property [4]. There are numerous reports on the synthesis of various nanocomposites of ZnO with different

compounds such as Co_3O_4 , CuO , CeO_2 , TiO_2 are the different coupled semiconductors that have been successfully fabricated [5-9]. Further, cadmium oxide (CdO) is an important semiconductor with band gap of $\sim 2.2\text{eV}$ with many applications like photocatalytic activity, gas sensing, optical property, storage of lithium batteries, etc [10-13]. The coupling of ZnO nanoparticles with CdO nanoparticles to observed enhanced photocatalytic activity [14]. There are very few reports on the coupling of ZnO with CdO [15, 16]. In general, the synthesis method of ZnO/CdO nanocomposites is adopted by thermal decomposition, sol gel, simple thermal treatment, thermal vapour transport deposition method etc.

In the present work deals with the synthesis of ZnO coupled with CdO nanoparticles by using co-precipitation method. The well-crystalline and uniformly spherical shaped nanoparticles are observed from the current synthesis method. Moreover, the optical properties of prepared ZnO-CdO nanocomposites are analyzed by UV and PL studies.

2. Experimental procedure

A typical procedure, zinc acetate dihydrate (Rankem) and cadmium acetate (Rankem) used in the present study were of analytical reagent grade. The precipitation method was employed for the synthesis of the ZnO/CdO nanocomposites and EDTA was used as the stabilizer (capping agent). The reagents containing suitable mole percentage of the metal ions and a dilute solution of EDTA were mixed slowly in a beaker with vigorous stirring. All the preparations were carried out at ambient temperatures. For the present preparations aqueous solutions of zinc acetate (0.1 M), cadmium acetate (0.1 M), ammonium carbonate (0.1 M) and EDTA (0.05 M) were used. The resulting precipitates were centrifuged and washed thoroughly many times with distilled water and dried at 80°C . Finally the sample calcined at 400°C for 2h.

The X-ray diffraction (XRD) analysis was done on a Rich Siefert 3000 diffractometer using $\text{Cu K}\alpha$ radiation ($\lambda = 0.15405\text{ nm}$). The FTIR spectrum was observed on a Bruker-Tensor 27 Fourier transform infra red spectrophotometer. The UV-vis absorption spectrum was obtained on a CARY 5E UV-VIS-NIR spectrophotometer. Field emission-scanning electron microscopy (FE-SEM) and energy dispersive X-ray spectroscopy analysis were carried out using HITACHI-SU6600. The TEM image was carried out on the sample with a Philips model CM-20 by using accelerated voltage of 300 kV.

3. Results and Discussions

The structural property of the ZnO/CdO sample was studied and the corresponding X-ray diffraction pattern is shown in Fig.1. It showed that the presence of wide diffraction peaks of hexagonal wurtzite structure ZnO, along with the low intensity peaks corresponding to the CdO phase. The strong intensity and narrow width of the ZnO diffraction peaks indicate that the resulting products are of high crystallinity. Two phases are identified in ZnO/CdO sample, One is the hexagonal structure of ZnO and other is CdO with peaks (1 1 1), (2 0 0) and (2 2 0) indexed to cubic structure

(card no.: 73-2245) and the lattice constants values are determined as $a = b = c = 0.470$ nm [7]. The average crystallite size of ZnO/CdO sample was calculated to be about 25-36 nm using Debye-Scherrer's equation.

The FTIR spectrum of as-prepared ZnO/CdO sample is shown in Fig.2. The broad absorption band at $\sim 3426\text{cm}^{-1}$ corresponds to the -OH stretching vibrations of water present in ZnO and the other absorbance bands at ~ 2923 and $\sim 2853\text{cm}^{-1}$ are assigned to the residual organic components of EDTA molecules. The band at $\sim 1631\text{cm}^{-1}$ can be associated with the bending vibrations of H_2O molecules. The absorption bands at ~ 1570 and $\sim 1431\text{cm}^{-1}$ in both the samples are due to the carbonyl groups of the carboxylate ions which might remain adsorbed on the surface of ZnO. The stretching vibrations of ZnO and CdO were appeared at ~ 503 and 722cm^{-1} , respectively. The ZnO/CdO nanocomposites cause slight changes in the intensities of absorption bands at $\sim 1631-1431\text{cm}^{-1}$ [14].

Fig.3(a) shows the SEM image of ZnO/CdO nanocomposites. It clearly revealed that the well-shaped spherical-like particles of size 20-30 nm in diameter. It can be believed that the presence of well-dispersed particles due to the capping agent i.e. EDTA did the spacing of individual particles since it prevents from the aggregated particles. The EDX analysis confirms the stoichiometry of the prepared sample as shown in Fig.3 (b). No other impurity is seen in the EDX pattern, indicating that the formed ZnO/CdO nanocomposites were 100% pure.

The TEM analysis was carried out to obtain further information about the morphology and crystallinity of the samples. Fig.4 displays the TEM image of the ZnO/CdO nanocomposites. The ZnO/CdO nanostructures showed a predominant spherical shaped particle, which confirms the crystalline nature of the samples. The average particle size is calculated as 25nm, which is in accordance with the XRD data analysis.

Fig.5 shows the UV-absorption spectrum of ZnO/CdO nanoparticles. From the UV-absorption spectrum, the band gap energy is calculated as 3.23eV, which is in good agreement with the previously reported values of ZnO/CdO nanocomposite [15]. In the present investigation, the band gap energy of the nanocomposites is slightly blue-shifted with the CdO content compared to that of ZnO due to their smaller crystallite size. The slight blue-shift in the emission peak could be attributed to the existence of some Cd phase at the interface of the nanocomposite.

The room temperature PL emission spectrum of ZnO/CdO nanocomposite with an excitation wavelength of 300nm is displayed in Fig.6. The peak observed at 390 nm corresponds to the band-edge emission of the CdO nanocrystals [7, 15]. However, the ZnO and ZnO/CdO samples exhibit a strong UV emission peak at 487nm (2.54 eV) and 424 nm (2.92 eV) accompanied by other peak of relatively lower intensity positioned at 532nm (2.33 eV). The origin of the violet emission at 424 and 487 nm could be ascribed due to the transition occurring from Zn interstitials to the valence band, and the peak of 424 nm may be the result of the singly ionized oxygen vacancy (V_o) [14]. Therefore, the PL peaks of the nanocomposites were observed close to ZnO crystalline phase.

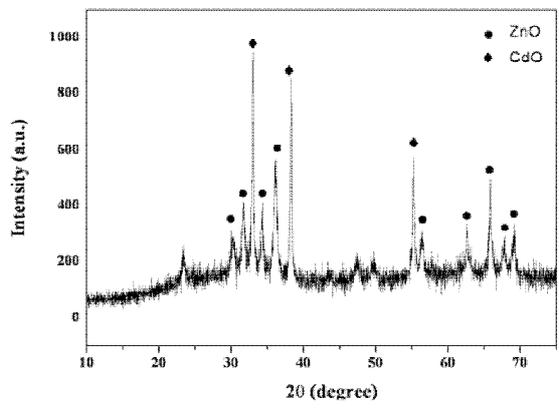


Fig.1 XRD pattern of ZnO/CdO naocomposites

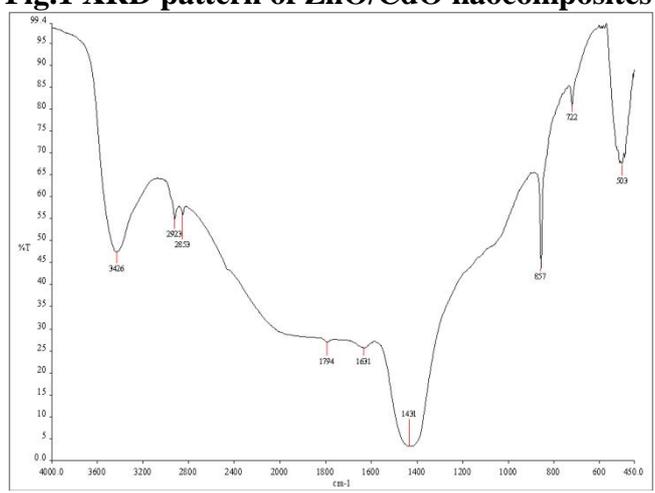


Fig.2 FTIR spectrum of ZnO/CdO as-prepared sample

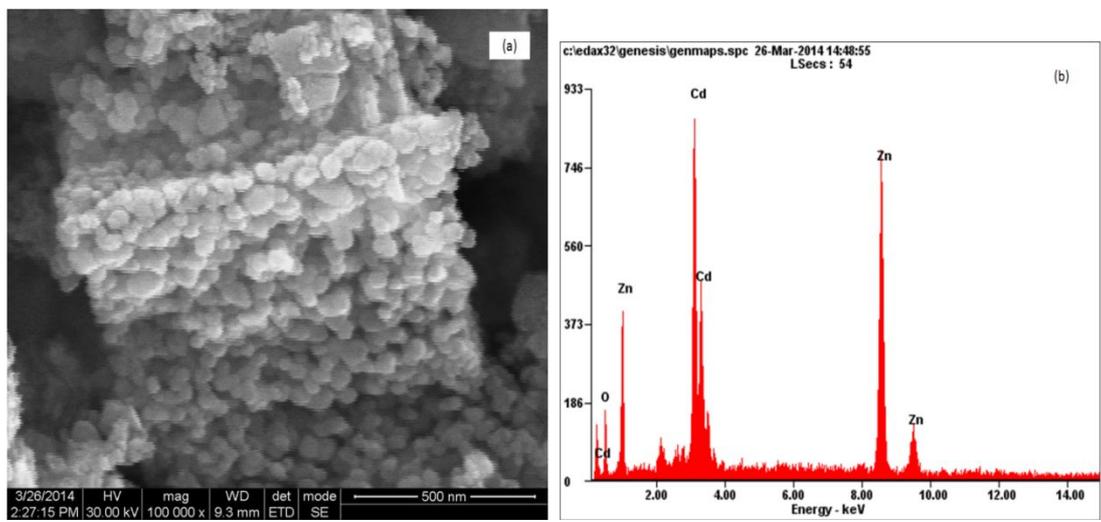


Fig.3(a-b) SEM image and EDX spectrum of ZnO/CdO nanocomposites

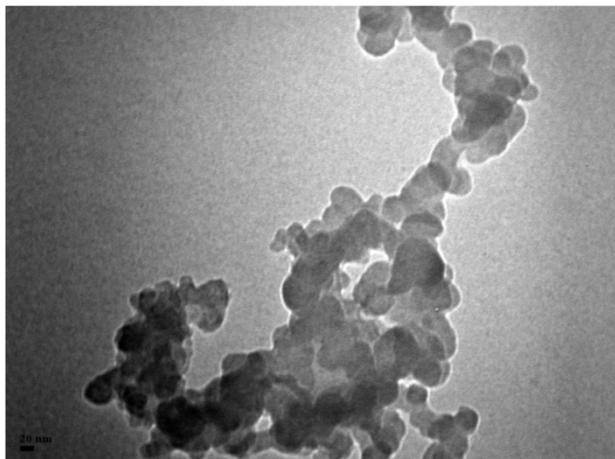


Fig.4 TEM image of ZnO/CdO nanocomposites

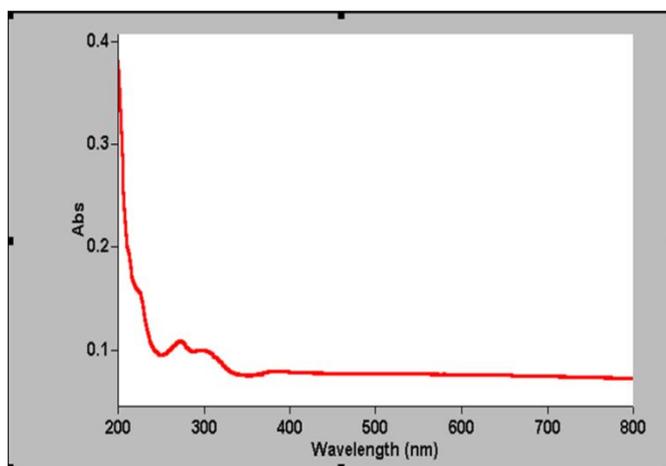


Fig.5 UV-absorption spectrum of ZnO/CdO nanocomposites

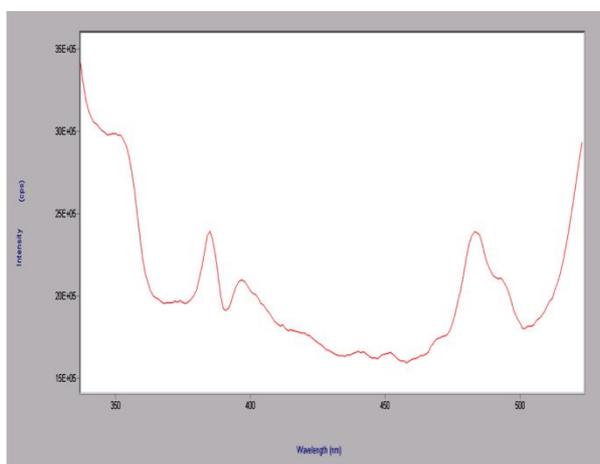


Fig.6 PL emission spectrum of ZnO/CdO nanocomposite

4. Conclusion

In the present work, ZnO/CdO nanocomposites are prepared by facile co-precipitation method. The structural, morphological and optical properties of ZnO/CdO nanocomposites were investigated. ZnO/CdO phase was identified by XRD and FTIR studies. The XRD and TEM analysis confirms the formation of spherical nanocrystals with an average size of 25nm. The PL measurement revealed that the ZnO/CdO nanocomposite contains oxygen defects. Further it should be applicable for the photocatalytic activity.

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