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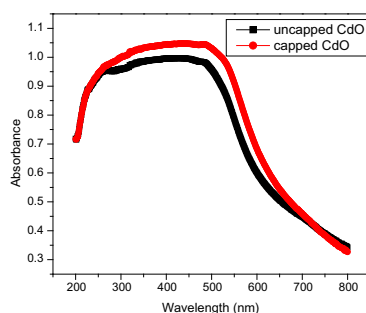
Synthesis, FTIR, UV–Vis and Photoluminescence characterizations of triethanolamine passivated CdO nanostructures

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HIGHLIGHTS

- The present work confirms the optical absorption and band gap of CdO nanostructures.
- Photoluminescence spectra shows different emissions for CdO nanostructures.
- The functional groups and the chemical bonding with triethanolamine have been confirmed by FTIR spectra.
- FESEM and TEM have been proved to be very supportive in the morphological features.

GRAPHICAL ABSTRACT



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ABSTRACT

In this study, triethanolamine (TEOA) capped CdO nanostructures had been synthesized by wet chemical method annealed at 648 K were reported. The structural, morphological and optical properties of the samples were studied by X-ray diffraction (XRD), Field emission scanning electron microscopy (FESEM) with energy dispersive spectra (EDS) analysis, Fourier transform infrared (FTIR) spectroscopy, UV–Vis spectroscopy and Photoluminescence (PL) techniques. The XRD spectrum showed that all the samples were cubic in structure. The presence of functional groups and chemical bonding had been confirmed by FTIR. UV–Vis measurements showed decreased band gap energy for TEOA capped CdO, when compared with uncapped CdO. The PL spectra of the CdO systems showed the red emission.

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Introduction

Low dimensional nanoscale semiconducting metal oxide materials have attracted much attention because they have new functional properties with numerous applications [1–3]. Nanocrystalline CdO is an important n-type semiconductor metal oxide with rock-salt structure (FCC). The direct band gap energy of this semiconductor is 2.2–2.7 eV and indirect band gap energy is 1.36–1.98 eV [4]. Recently different values of band gap energy have been reported

in the literature [5]. In recent years, many researchers have focused on cadmium oxide due to their applications in several areas of research; specifically in optoelectronics and other applications including solar cells [6–7], photo transistors [8], transparent electrode and gas sensors [9]. Reduction in the dimensionality of such materials from the three dimensional bulk phases to the zero dimensional nanoparticles can lead to enhanced linearity determined by the quantum size effects and other mesoscopy effects. Because of these interesting possibilities there has been more effect to prepare nanoparticle of CdO [10]. CdO nanoparticles have been synthesized with different methods such as, solgel [11], solvothermal [12], micro emulsion method [13], precipitation method [14] and sonochemical method [15]. Among these methods wet

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chemical method is the simplest and most cost effective method for synthesizing nanoparticles. The surfactants play an essential role in controlling morphology of nanostructure because of their soft-template effect, their ability to modify the chemical kinetics and simple maneuverability. However the ability to understand and foretell the final structure/morphology is still limited [16]. Recently some nanoparticles have been synthesized with amines as an organic ligand. Archana et al. report the synthesis of triethylamine capped ZnSe nanostructures with bean like morphology by using an inexpensive wet chemical method [17]. Navaneethan et al. [18] synthesized the monodispersed ZnO nanosheets by a facile wet chemical method using hexamethylenetetramine (HMTA) as an organic ligand. Optical and surface morphological properties of triethylamine passivated lead sulfide nanoparticles were synthesized and characterized by various techniques [19]. Navaneethan et al. had report the synthesis of ZnO nanorods by hydrothermal growth [20]. To the best of our knowledge, there is no report of synthesis of CdO nanoparticles with TEOA used as a surfactant. In this study triethanolamine capped CdO nanostructures are synthesized by simple wet chemical route.

Experimental

All chemicals are procured from E-Merck with 99.9% purity and used without further purification. Synthesis of CdO nanostructures are as follows; 0.1 M of cadmium acetate and 0.1 M of sodium hydroxide is dissolved in 100 ml deionised water and 5 ml concentration of TEOA is added in the solution and whilst stirring at 1000 rpm in a beaker. The reaction is continued for 10 h at 303 K. The milky white precipitation is washed with water several times and dried at 343 K for 3 h. The dried samples are annealed at 648 K for 2 h. Finally a brown color TEOA capped CdO sample is obtained. Similar procedure is carried out without TEOA to identify the effect of capping amine molecule and is termed as uncapped CdO.

The synthesized nanoparticles are characterized using an X'Pert PRO (PANalytical) advanced X-ray diffractometer with CuK α radiation (1.5406 Å) with 2 θ° value ranging between 20° and 80°. The morphology and particle size of the creation are observed by HITACHI, Japan model no; SU660 Field Emission Microscope at an accelerating voltage of 15KV. TEM analysis is made with JEOLJEM 2100F and EDS with HORIBA, Japan. UV-Vis spectrum is measured over the region 200–900 nm by UV-2600 Shimadzu spectrophotometer. The functional group of the synthesized material is analyzed by Fourier transform infrared spectroscopic method (FTIR) over the region 4000–400 cm⁻¹ using JASCO-FTIR 6300 Type A, Serial No., A021161024. The Photoluminescence spectrum of the synthesized material is recorded over the region 500–900 (a.u) using Jobin–Yvon Fluorolog-3 spectro fluorometer.

XRD analysis

Fig. 1 represents the XRD of synthesized CdO nanoparticles. All the diffraction peaks are reflections of samples indexed to cubic phase of CdO. It is matched well with the standard data of JCPDS 05-0640. XRD diffraction peaks analogous to TEOA capped CdO are of higher intensity than the diffraction peaks with uncapped CdO nanostructures. This is due to the re-orientation of CdO nanostructures in the presence of TEOA. Thus the XRD results confirmed the formation of pure CdO nanostructures.

FESEM and TEM analysis

Morphology and size of the nanostructures are observed by FESEM and TEM analysis respectively. The FESEM image of the

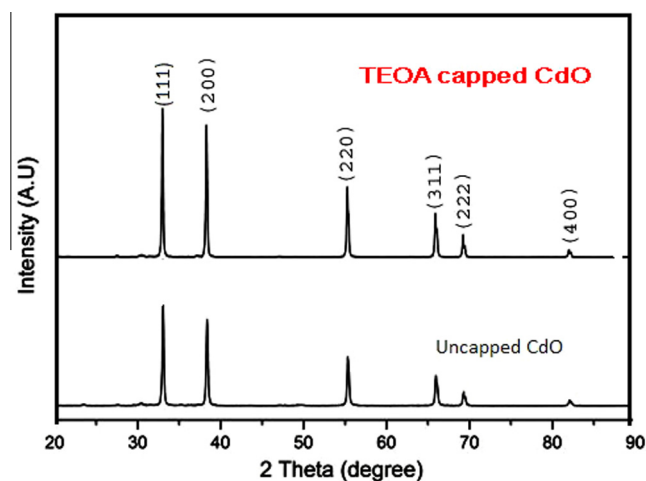
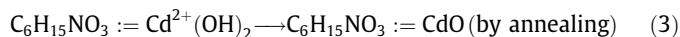
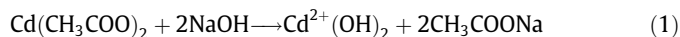


Fig. 1. XRD pattern of CdO nanostructures.

uncapped CdO is presented in Fig. 2(a) and corresponding TEM image is in Fig. 2(b) indicates the agglomerated without any specific morphology. TEOA capped CdO nanoparticles exhibited the distinguished morphology and it is presented in FESEM image in Fig. 2(c) and TEM image in Fig. 2(d). The shape of synthesized TEOA capped nanoparticle is well defined shape of nanospheres. From the TEM images the size of nanospheres are 42.6–71.7 nm. The Agglomeration is restricted by surface passivation of the amine molecule. Based on the morphological studies, the creation mechanism of a nanospheres are explained as follows; Cd²⁺ ions are released from cadmium acetate and OH⁻ ions are released by sodium hydroxide in aqueous solution. This results are in the formation of a cadmium hydroxyl ions. Since the basic structure of this ions consists of agglomerated and irregular nanospheres morphology. Due to the addition of TEOA molecule to the cadmium hydroxyl ions, restricts the growth of agglomeration. Finally annealing at 648 K leads to the creation of monodispersed CdO nanospheres. The formation mechanism to produce CdO nanostructures is obtained from [18] and stated as follows;



EDS Analysis

Fig. 3 shows the EDS pattern with the presence of Cadmium and Oxygen peaks, and it confirmed the absence of other impurities among the prepared CdO sample. The presence of cadmium and oxygen ions present in CdO and atomic percentages are found to be at 37.39% and 50.14% respectively.

FTIR analysis

The existence of strong FTIR interactions between cadmium oxide and triethanolamine molecules are confirmed by the FTIR spectrum in the wavelength region of 4000–400 cm⁻¹ for the synthesized CdO nanoparticle is presented in Fig. 4. The broad FTIR absorption band at 3457 cm⁻¹ is assigned to stretching vibrations of hydroxyl group (O–H) of water molecules. The FTIR bands observed at 3254 and 3248 cm⁻¹ are assigned to N–H asymmetric and symmetric stretching vibrations respectively. The FTIR bands

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