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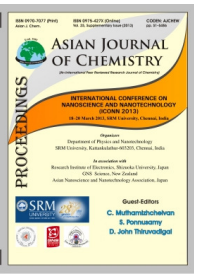

Synthesis and Characterization of CdO Nanoparticles by Novel Wet Chemical Method†

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Cadmium oxide nanoparticles were successfully synthesized by wet chemical method using cadium chloride monohydrate and NaOH in doubly distilled water solution. Triethylamine was used as a capping agent. The synthesized nanoparticles were characterized by field emission scanning electron microscopy was carried out for structural characterization, Energy dispersive X-rays spectroscopy was used for element analysis, X-ray diffraction and UV-visible diffused reflectance spectroscopy. The possible formation mechanism has been discussed with the experimental results.

Key Words: Cadium oxide, Triethylamine, Field emission scanning electron microscopy, X-ray diffraction.

INTRODUCTION

Semiconductor nanomaterials have fascinating physical and chemical properties and useful functionalities when compared with conventional bulk counterparts and molecular materials. Most semiconducting materials, such as the II-VI or III-VI compound semiconductors show the quantum confinement behaviour in the 1-20 nm size range. Among the unique properties of nanomaterials, the movement of electrons and holes in semiconductor nanomaterials is mainly governed by the well-known quantum confinement and the transport properties related to phonons and photons are largely affected by the size and geometry of the materials. The specific surface area and surface-to-volume ratio increase dramatically as the size of material decreases. Parameters such as size, shape and surface characteristics can be varied to control their properties for different applications of interest. These novel properties of semiconductor nanomaterials have attracted significant attention in research and applications in emerging technologies such as nanoelectronics, nanophotonics, energy conversion, non-linear optics, miniaturized sensors and imaging devices, solar cells, catalysis, detectors, photography and biomedicine, *etc.*[1]. The synthesis of group II-IV binary semiconductor in a nanopowder form has been emerging area of research due to their significant optical, physical and chemical properties[2,3]. Cadmium oxide is one of the main precursors to other cadmium compounds. Its crystal structure is cubic. Cadmium oxide is a semiconducting nanomaterial with a band gap of ≈ 2.16 eV at room temperature[4]. It has excellent industrial applications in the area of catalysis, photo catalysis and electrodes for storage batteries. Recently many researchers have been synthesized cadium oxide nanoparticles by different methods[5-7]. In the present investigation our attention has been turned to synthesize and characterizes the triethylamine capped CdO nanoparticles by wet chemical method.

EXPERIMENTAL

All the chemicals procured in the present work were guaranteed reagent (GR-99.9 %) from MERCK, Mumbai, India and used without further purification. Doubly distilled deionized water was used throughout the experiment.

General procedure: CdO nanoparticles with triethylamine (TEA) as capping agent synthesized as follows. The solution was prepared by 0.2 M of cadmium chloride monohydrate was dissolved in 2 mL of doubly distilled water (solution A). 0.2 M of NaOH and 2 mL of triethylamine were dissolved in another 20 mL of doubly distilled water (solution B). The solution B was continuously stirred for three different temperatures. The resulting solution was stirred before 1 h

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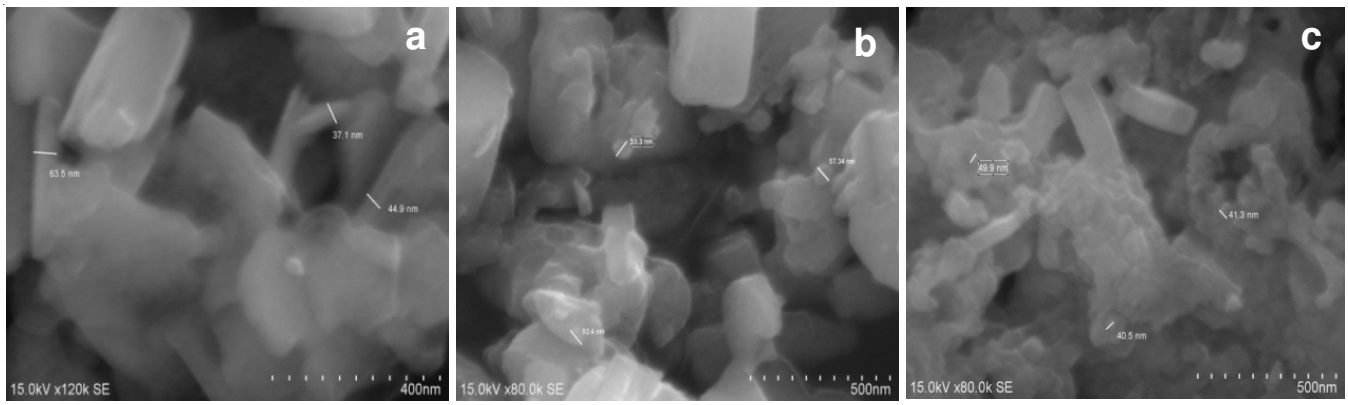

Fig. 1. FESEM image of CdO nanoparticles at different stirring temperature of the solution at (a) 45 ºC (b) 65 ºC and (c) 85 ºC

adding solution A. After 3 h of constant stirring a milky white solution was obtained. The product was rinsed with distilled water many times. The residue was washed in ethanol and allowed it to evaporate at room temperature. Then the $Cd(OH)_2$ nanoparticles were kept at muffle furnace for 3 h. The obtained CdO nanopowder was brown in colour[4].

Detection method: The synthesized nanoparticles were characterized by FESEM with HITACHI, Japan, Model No. SU660, EDX with HORIBA, Japan, Model: EMAX, XRD with 3003 TT and UV-visible DRS with Shimadzu spectrophotometer.

RESULTS AND DISCUSSION

FESEM/EDX analysis: The FESEM images of CdO nanoparticles at three different stirring temperatures (45, 65 and 85 ºC) are presented in Fig. 1. From FESEM image, the size of the particles was found 63.5, 52.4 and 49.9 nm, respectively. Fig. 2 shows the EDX spectrum of the triethylamine capped CdO nanoparticles which confirm the presence of cadmium and oxygen.

XRD analysis: The XRD pattern of CdO nanoparticles are shown in Fig. 3. The particle size was determined using Debye-Scherrer's formula, $d = (0.94\lambda)/(\beta \cos \theta)$, where β is the full width half maximum (FWHM), θ is the diffraction angle, d is the average crystallite grain size and λ is the wavelength of X-rays. All the diffraction peaks were indexed to cubic CdO (JCPDS card No. 652908) nanoparticles. The prominent peak positions corresponding to (111), (220), (400) and (331) planes at angles $2\theta = 29.5$, 16.3, 11.5 and 10.5. The size of particles was 8, 7 and 5 nm at 45, 65 and 85 ºC, respectively.

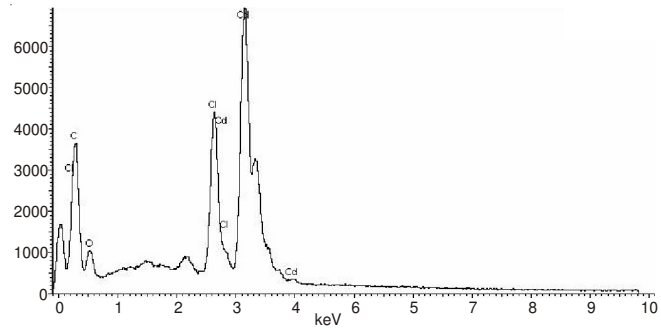

Fig. 2. EDX spectrum of CdO nanoparticles

UV-visible analysis: The UV-visible absorption spectra of CdO nanoparticles capped with triethylamine stirred at three different temperatures are presented in Fig. 4. The band gap of the nanoparticles were determined by Tauc relation[8], $\alpha h\nu = A (h\nu - E_g)^n$. Where α absorption coefficient, $h\nu$ the photo energy, E_g the band gap n = ½ for the direct transitions and n = 2 for the indirect transitions. A graph of $(\alpha h\nu)^{1/2}$ *versus* $h\nu$ is plotted and the extrapolation of the straight line gives the value of energy band gap. Manickathai *et al.*[2] has reported that the band gap energy of CdO nanoparticles capped with ethylene glycol was 3.4 eV. In the present work, the band gap energy were 4.1, 3.05 and 2.7 eV for CdO nanoparticles capped with triethylamine at 45, 65 and 85 ºC, respectively. Table-1 shows the comparison of particle size, band gap energy of the CdO nanoparticles.

Conclusion

The CdO nanoparticles capped with triethylamine have been synthesized by wet chemical method. The prepared

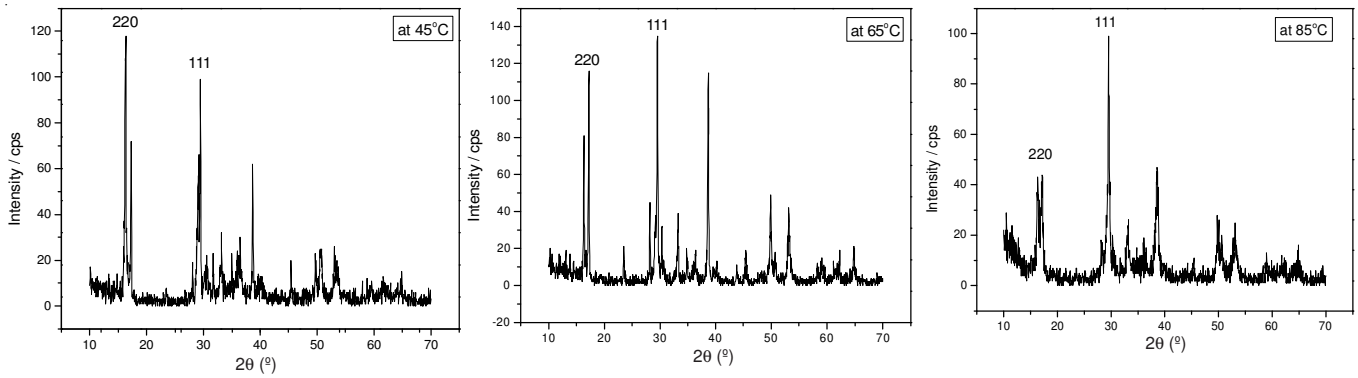

Fig. 3. XRD Pattern of CdO nanoparticles

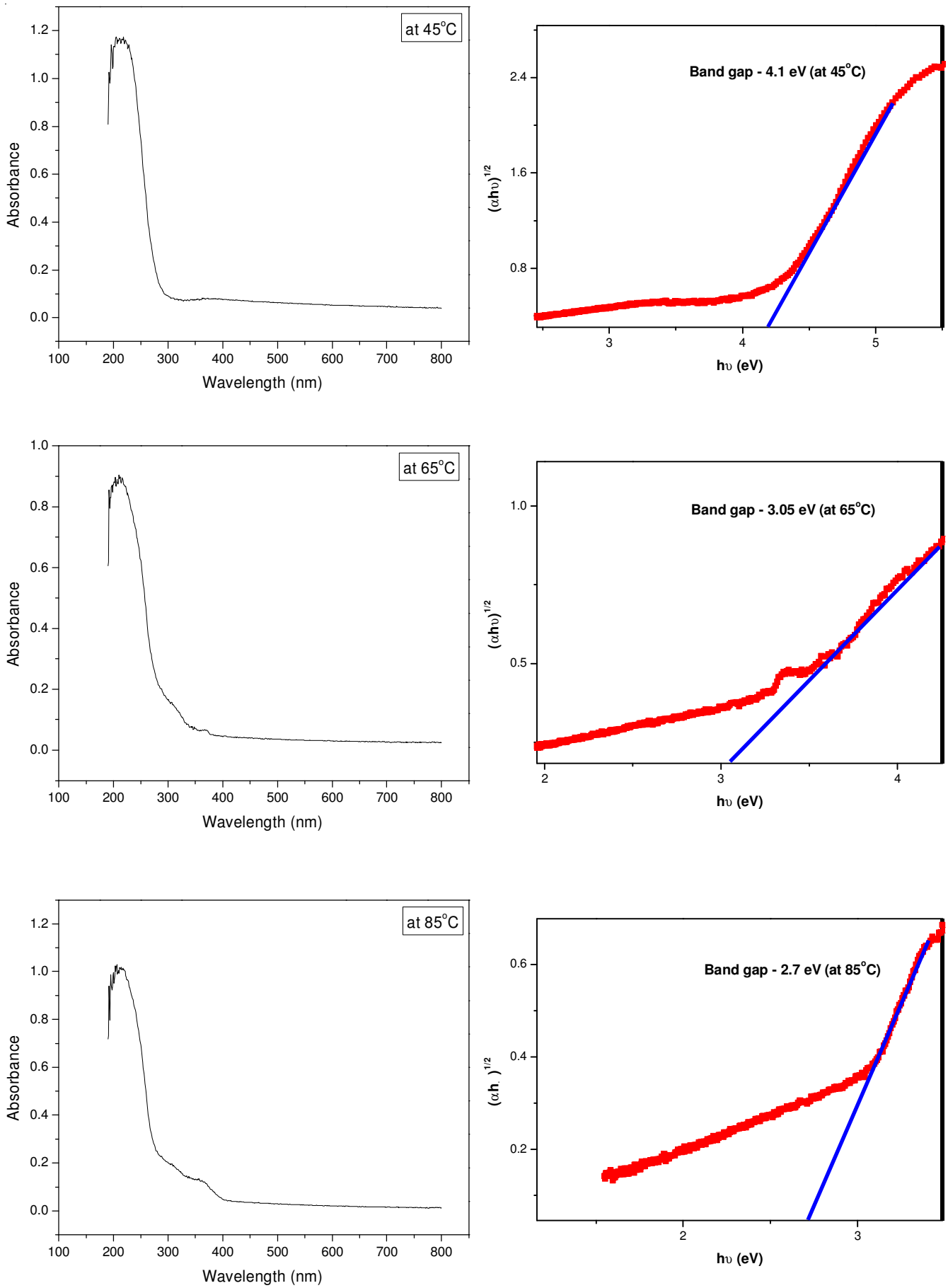

Fig. 4. UV-visible spectra of CdO nanoparticles with Tauc plot

TABLE-1
PARTICLE SIZE AND BAND GAP ENERGY OF THE CdO NANOPARTICLES AT DIFFERENT STIRRING TEMPERATURES

Stirring temperature of the solution (ºC)	Particle size (nm)		Band gap energy (eV)
	From XRD	From FESEM	
45	8	63.5	4.10
65	7	52.4	3.05
85	5	49.9	2.70

samples were stirred at three diffrent temperatures. From the FESEM and XRD analysis it has been found that the size of the nanoparticles were reduced with the increase of stirring temperatures. Also the intensity of the peak observed between the diffraction angle 10-20º was decreases with the increase of temperature. The energy band gap of semiconductors tends to decrease as the temperature is increased[9]. This behaviour can be better understood if one considers that the interatomic spacing increases when the amplitude of the atomic vibrations increases due to the increased thermal energy. An increased interatomic spacing decreases the potential seen by the electrons in the material, which in turn reduces the size of the energy band gap. Based on this fact, from the UV-visible analysis, the energy band gap of the nanoparticles has been decreases with the increase of stirring temperature of the prepared solution and which confirms quantum confinement effect of the semiconducting CdO nanoparticles.

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