Hindawi
Journal of Nanotechnology
Volume 2017, Article ID 8317109, 11 pages
https://doi.org/10.1155/2017/8317109

Research Article

The Influence of Temperature on the Formation of Cubic Structured CdO Nanoparticles and Their Thin Films from *Bis*(2-hydroxy-1-naphthaldehydato)cadmium(II) Complex via Thermal Decomposition Technique

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Received 26 February 2017; Accepted 26 March 2017; Published 9 May 2017

Academic Editor: Andrey E. Miroshnichenko

Recently, researchers have developed a great interest in the synthesis of metal oxide nanoparticles due to their potential applications in various fields of science and industry, especially in catalysis, due to their high activity. *Bis*(2-hydroxy-1-naphthaldehydato)cadmium(II) complexes were prepared and used as precursors for the synthesis of cadmium oxide nanoparticles via thermal decomposition method using HDA as a stabilizing agent. The prepared complexes were also used as single source precursors to prepare CdO thin films onto the glass substrates by spin coating and were annealed at 250, 300, and 350°C, respectively. The precursors were characterized by Fourier transform infrared (FTIR) spectroscopy, elemental analysis, nuclear magnetic resonance (NMR), and thermogravimetric analysis (TGA). The synthesized CdO nanoparticles and CdO thin films were characterized by ultraviolet-visible (UV-vis) spectroscopy, photoluminescence (PL), X-ray diffraction (XRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM), and atomic force microscopy (AFM).

1. Introduction

Researchers have shown much interest in synthesizing nanometer-sized semiconductor particles recently as such particles demonstrate size-dependent optical and electrical properties. The development of these nanoparticles has been intensively shadowed not only for their fundamental scientific attentiveness but also for numerous technological applications. Semiconductor nanomaterials also exhibit distinctive and significantly modified physical and chemical properties, compared to their bulk materials, which make them of particular interest [1, 2]. There has been great research significance for the synthesis of metal oxide since such materials have high specific surface area and a high fraction of surface atoms. Recently, metal oxide nanomaterials have been extensively studied due to their unique physicochemical characteristics such as their potential applications in optical detection, emission, and electronic and mechanical devices grounded on variable oxidation states.

Among the semiconducting metal oxide nanomaterials, cadmium oxide has attracted prominent attention for several applications such as photodiodes and solar cells due to its low electrical resistivity and high optical transmittance in the visible region of the solar spectrum. CdO is a II–VI binary oxide and is classified as an n-type semiconducting material having the direct band gap of 2.2 to 2.5 eV [3–6]. Cadmium oxide nanostructured materials are also used as gas sensors, transparent electrodes, optoelectronic devices, windows, flat panel display, photo transistors, and catalysts [7, 8].

It has been reported that the physical and chemical properties of CdO materials are relative to CdO stoichiometry as well as particle size and shape, which, in turn, depend

on its preparation methods and synthetic conditions [3, 9]. Different synthetic routes for the synthesis of ultrafine and nanosized cadmium oxides particles to obtain defined properties have been used and some of them include chemical precipitation [10], hydrothermal technique [11], microemulsion [4], solvothermal process [3], microwave-assisted method [12], and mechanochemical process [13, 14]. Some researchers have tried to modify several techniques for the preparation of CdO thin films with the aim to improve chemical and physical properties of this material which include thermal decomposition [15, 16], MOCVD (metal-organic chemical vapour deposition) [17], r.f. sputtering [18], and Langmuir-Blodgett deposition technique [19].

Ramazani and Morsali [20] reported the synthesis of cadmium oxide nanoparticles through thermal decomposition of organocadmium complexes. We have recently reported the synthesis of ZnO and CdO nanoparticles using *bis*(2-hydroxy-1-naphthaldehydato)zinc(II) and cadmium(II) complexes via thermal decomposition technique using trioctylphosphine oxide as a capping agent. The spherical nanoparticles of ZnO and CdO revealed the face-centred cubic phase on both materials [21]. Azizar Rahman and Khan reported the synthesis of CdO thin films by a spray pyrolysis technique onto a glass substrate at different temperatures. It was discovered that the grain size increases, while the dislocation density decreases as the annealing temperature was increasing [22].

Thermal decomposition process can be regarded as an alternative technique for the synthesis of cadmium oxide nanomaterials since it is fast, free of toxic solvents, of low cost, and effective compared to the other synthetic routes. Nanomaterials with controlled morphologies can be synthesized through the good choice of the precursor and by choosing the proper calcination conditions [23]. 2-Hydroxy-1-naphthaldehyde is a less expensive ligand that has been extensively used in biological activities to determine free amino acid groups in polymer substances. The metal complexes based on this ligand can be used to synthesize nanomaterials that can be applied in biological activities [24]. Current investigations have verified that formulated nanomaterials exhibit good antibacterial activities and establish the antimicrobial formulations [25–27]. The influence of nanomaterials on the bacteria is very substantial since these materials signify the lowest level and hereafter enter the food chain in the environmental systems [28, 29]. Cadmium oxide nanomaterials have been proven for their antibacterial activities. This material is nontoxic and chemically stable which is competent in photocatalytic oxidation [30, 31]. Therefore, CdO nanomaterials synthesized from *bis*(2-hydroxy-1-naphthaldehydato)cadmium(II) complex entertain potentially promising application in the struggle against antibiotic resistant pathogenic bacteria which pose an unceasing threat to human and animal life [32, 33].

In the present work, the preparation of cadmium(II) complexes and the synthesis of cadmium oxide nanoparticles through thermal decomposition of *bis*(2-hydroxy-1-naphthaldehydato)cadmium(II) complex into hexadecylamine (HDA) at different decomposition temperatures is reported. This complex was also used as a single source precursor to prepare CdO thin films at different annealing temperatures on the glass substrate by spin coating method. The synthesized CdO materials were characterized by ultraviolet-visible (UV-vis) spectroscopy, photoluminescence (PL), X-ray diffraction (XRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM), and atomic force microscopy (AFM).

2. Experimental

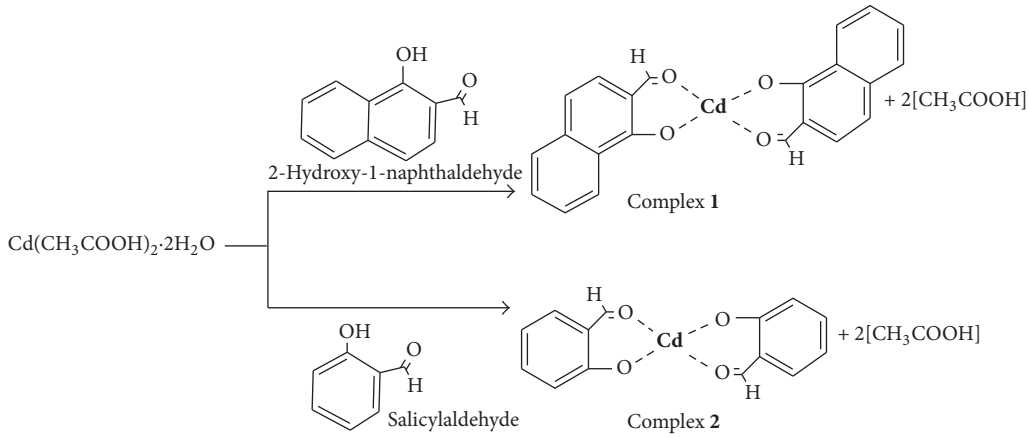
2.1. Materials and Reagents. Cadmium acetate dihydrate, 2-hydroxy-1-naphthaldehyde, salicylaldehyde, hexadecylamine (HDA), ethanol, acetone, and toluene were reagents purchased from Sigma-Aldrich and were all used without further purification.

2.2. Preparation of the Precursors. *Bis*(2-hydroxy-1-naphthaldehydato)cadmium(II) and *bis*(salicylidene)cadmium(II) complexes were prepared as described previously [21, 34]. A brief description of each synthesis is given below.

*2.2.1. Preparation of Bis(2-hydroxy-1-naphthaldehydato)cadmium(II) Complex $[Cd(C_{11}H_6O_2)_2]$ **(1)**.* In a typical experiment, a 20 mL ethanolic solution of Cd $(CH_3CO_2)_2{\cdot}2H_2O$ (5 mmol) was added to a 20 mL ethanolic solution of 2-hydroxy-1-naphthaldehyde (HNA) (10 mmol) to form a homogeneous solution. The solution was stirred and refluxed at 60°C for about 1 hr. The coloured precipitate that settled from the solution was centrifuged and washed with ethanol three times. The reddish/green product was dried in a vacuum oven at room temperature, weighed, and characterized. Percentage yield: 69%. m.pt. 180–186°C. CHN analysis: Calc.: C, 58.10; H, 2.66; O, 14.14. Found: C, 57.77; H, 2.70; O, 15.22%. Significant IR bands: ν(C=O): 1647 cm^{-1}, ν(C-O): 540 cm^{-1}. *^{1}H NMR δ (ppm): (400 MHz, DMSO-d_6, 295 K): δ = 10.89 (s, 1H, CH-1), 8.99 (m, 1H, CH-2), 8.21 (m, 1H, CH-3), 7.92 (m, 1H, CH-4), 7.61 (m, 1H, CH-5), 7.42 (m, 1H, CH-6), 7.21 (m, 1H, CH-7). ^{13}C NMR (400 MHz, DMSO-d_6, 296 K) δ (ppm): 193 (C=O), 164 (C-O), 138 (C-H), 132 (C-H), 130 (C-H), 129 (C-H), 128 (C-H), 127 (C-H), 124 (C-H), 122 (C-H), 111 (C-H).*

*2.2.2. Preparation of Bis(salicylidene)cadmium (II) Complex $[Cd(C_7H_5O_2)_2]$ **(2)**.* *Bis*(salicylidene)cadmium(II) complex was prepared by the same method as described in Section 2.2.1. by using salicylaldehyde instead of 2-hydroxy-1-naphthaldehyde. The complex was obtained as a cream white solid. Percentage yield: 47%. m.pt. 201–207°C. CHN analysis: Calc.: C, 54.66; H, 3.28; O, 20.80. Found: C, 53.99; H, 3.42; O, 20.57%. Significant IR bands: ν(C=O): 1647 cm^{-1}, ν(M-O): 540 cm^{-1}. *^{1}H NMR δ (ppm): (400 MHz, DMSO-d_6, 295 K): δ = 10.81 (s, 1H, CH), 8.98 (m, 1H, CH), 8.18 (m, 1H, CH), 7.81 (m, 1H, CH), 7.64 (m, 1H, CH), 7.47 (m, 1H, CH), 7.35 (m, 1H, CH). ^{13}C NMR (400 MHz, DMSO-d_6, 296 K) δ (ppm): 194 (C=O), 163 (C-O), 139 (C-H), 132 (C-H), 130 (C-H), 129 (C-H), 128 (C-H), 127 (C-H), 124 (C-H), 120 (C-H), 112 (C-H).*

2.3. Synthesis of Cadmium Oxide (CdO) Nanoparticles. Cadmium oxide nanoparticles were synthesized by thermal



Scheme 1: The preparation of *bis*(2-hydroxy-1-naphthaldehydato)cadmium(II) complex 1 and *bis*(salicylidene)cadmium(II) complex 2.

decomposition of *bis*(2-hydroxy-1-naphthaldehydato)cadmium(II) $[Cd(C_{11}H_6O_2)_2]$ complex in hexadecylamine (HDA). In a typical experiment, 5.0 g of HDA was transferred into a two-necked flask that contained 0.5 g of the complex. The mixture was refluxed and heated to 120°C under nitrogen environment. The temperature was maintained for one hour and the mixture was allowed to cool to a temperature of about 70°C. Addition of methanol (30 mL) produced a brown precipitate which was separated by centrifugation. The brown residue was washed three times with methanol and redispersed in toluene for characterization. The same procedure was repeated at 160 and 200°C.

2.4. Deposition of Cadmium Oxide Thin Films by Spin Coating. Prior to the experiment, the glass substrate (often used as a slide in microscope) was first cut into equal pieces of 2 × 1.5 cm using a diamond glass cutter and it was ultrasonically cleaned with acetone for 1 hour and then dried in open air. Exactly, 0.15 g (0.33 mmol) of the cadmium oxide precursor was dissolved in 20 mL of acetone in a small beaker. The precursor solution was then deposited on the glass substrate using the spin coater which was rotated at 3000 rpm for 30 sec. After the deposition, the film was dried at a very low temperature for about 5 mins on the hotplate in order to evaporate the solvent and remove organic residuals. Finally, the substrate with thin film was then annealed at various temperatures of 250, 300, and 350°C for an hour inside the tube furnace under nitrogen gas environment.

2.5. Physical Measurements. Elemental analysis was carried out on a PerkinElmer model 2400 series II CHNS/O analyzer. The FTIR spectra were recorded in the range of 400–4000 cm^{-1} by a Bruker FTIR TENSOR 27 spectrophotometer. The thermal analysis was performed at a 10°C min^{-1} heating rate by a PerkinElmer Pyris 6 TGA up to 600°C in a sealed perforated aluminium pan under nitrogen gas. Optical absorption analysis of the CdO nanoparticles and thin films was conducted with a PerkinElmer Lambda 1050 UV/vis/NIR spectrophotometer. The quartz cuvettes (1 cm path length) were utilized using toluene as a solvent for the analysis of nanoparticles. A PerkinElmer LS 55 spectrofluorimeter was exploited to analyze the photoluminescence of the nanoparticles and their thin films. The transmission electron microscopy (TEM) was carried out on a JEOL 1010 TEM, with an increasing speed voltage of 100 kV, Mega view III camera, and Soft Imaging System TEM software. The powder diffraction patterns were carried out in the high angle $2y$ range (20–90°) using a Bruker AXS D8 diffractometer armed with nickel filtered Cu-Ka radiation (l = 1.5418 Å) at 40 kV and 40 mA at room temperature. The scan speed and step sizes were arranged as 0.05 degrees per minute and 0.00657 degrees as reported by Mlowe and coworkers [35]. The Edwards E306A was used as a coating system to carbon coat the films prior to scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDXS) analysis. SEM analysis was achieved using a Philips XL-30 FEG scanning electron microscope, and EDXS was done using a DX4 detector. The atomic force microscopy (AFM) of the CdO thin films was performed using a Bruker Multimode 8 instrument. The X-ray diffraction studies of the films were carried out on a Bruker AXS D8 diffractometer by using Cu-Ka radiation.

3. Results and Discussion

The effective approaches to the synthesis of cadmium oxide nanoparticles by thermal decomposition and their thin films using annealing technique are reported. Cadmium complexes were obtained by the reaction of the cadmium acetate with a hydroxyl naphthaldehyde or salicylaldehyde ligand. Scheme 1 shows the overall synthetic procedure for the preparation of cadmium(II) complexes.

3.1. Thermogravimetric Analysis (TGA). The thermal properties of both complexes were studied by thermogravimetric analysis (TGA) at the temperature ranging from 20 to 600°C under nitrogen atmosphere. TGA graphs in Figure 1 show single step decompositions of the complexes with rapid

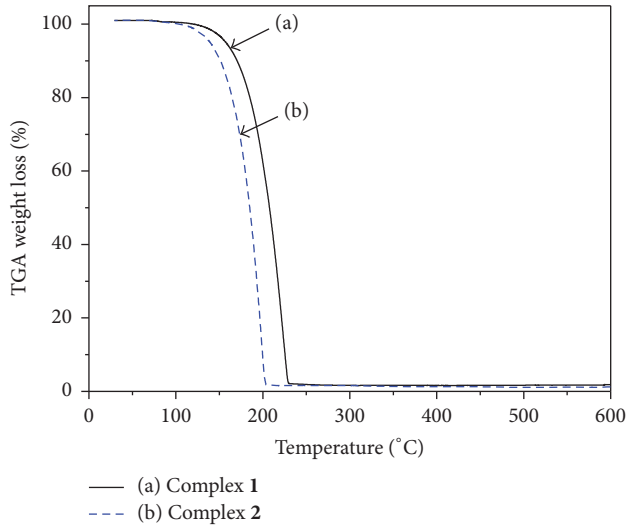


Figure 1: TGA curves of *bis*(2-hydroxy-1-naphthaldehydato)cadmium(II) (complex **1**) (a) and *bis*(salicylidene)cadmium(II) (complex **2**) (b).

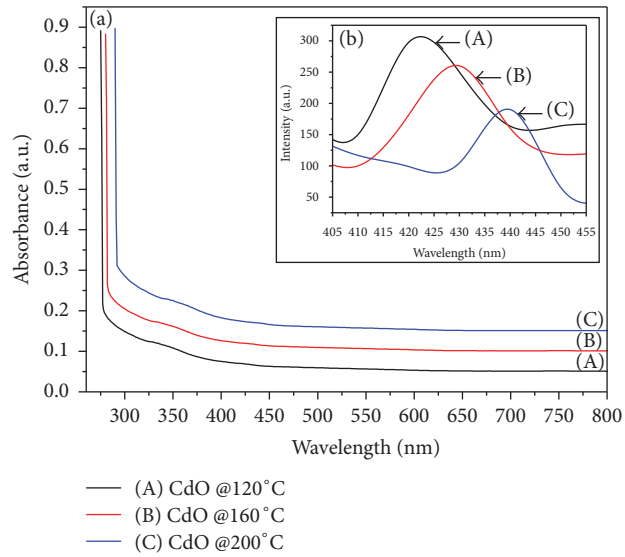


Figure 2: Absorption (a) and emission (b) spectra of HDA-capped CdO nanoparticles synthesized at 120 (A), 160 (B), and 200°C (C).

weight loss between 117 and 235°C for *bis*(2-hydroxy-1-naphthaldehydato)cadmium (II) and between 110 and 205°C for *bis*(salicylidene)cadmium (II) complexes, respectively, which correspond to the weight loss of about 97.1 and 98.3%. Thus, these results confirm that the free CdO thin film without other constituencies can be prepared at annealing temperatures higher than 235 and 205°C for complexes 1 and 2.

3.2. CdO Nanocrystals. The nanoparticles of CdO obtained by the thermal decomposition of *bis*(2-hydroxy-1-naphthaldehydato)cadmium(II) complex in HDA at various temperatures (120, 160, and 200°C) were analyzed by UV-vis, PL, p-XRD, and TEM. The study of optical absorption is essential to apprehend the behaviour of semiconductor nanoparticles. The fundamental property of semiconductors is the band gap, which is the energy separation between the filled valence band and the empty conduction band. Optical excitation of electrons across the band gap is strongly permitted, producing a sudden increase in absorption at the wavelength corresponding to the band gap energy (E_g) [36].

UV-vis Spectroscopy. The influence of reaction temperature on the optical absorption of CdO nanoparticles has been explored. The absorption spectra for CdO nanoparticles synthesized at 120, 160, and 200°C are shown in Figure 2(a). It is evident that all the samples exhibit the well-defined absorption band edges at 342, 353, and 360 nm. The band gap energy

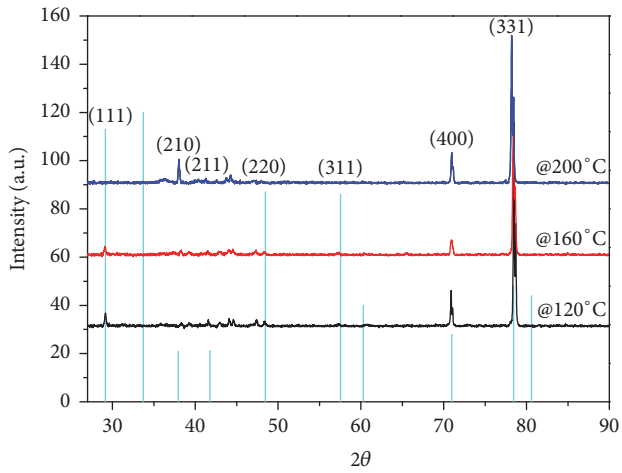


Figure 3: X-ray diffraction pattern of HDA-capped CdO nanoparticles synthesized at 120, 160, and 200°C.

of the CdO particles corresponding to the absorption edges is discovered to be 3.63, 3.51, and 3.44 eV, respectively. The band gap of bulk CdO is 2.30 eV at 300 K [37]. Thus, the CdO nanomaterials for all the samples are suggesting blue shift with respect to the bulk arising from quantum confinement effect of the nanoparticles.

Photoluminescence. The corresponding PL spectra with the excitation wavelength of 280 nm display the narrow band edge emissions, with the maximum at 422, 429, and 439 nm (Figure 2(b)). The bathochromic shift observed is typical for nanostructured semiconductors. The results signify that, upon increase of the reaction temperature, the growth rate of the CdO nanoparticles is increased. The possible reason for this phenomenon could be the fact that higher reaction temperature results in the formation of larger and irregular nanoparticles with reduced crystallinity due to random, faster nucleation and the rapid growth affecting the ligand configuration at the nanocrystal surface [38].

XRD Analysis. The powder X-ray diffraction (p-XRD) patterns of the CdO nanoparticles grown at various temperatures are shown in Figure 3 and could all be indexed to the cubic phase of CdO (JCPDS card number: 00-039-1221). Diffraction peaks at $2y$ = 29.15, 37.95, 41.36, 48.20, 57.36, 70.83, and 78.38° correspond to (111), (210), (211), (220), (311), (400), and (331) planes

TEM Analysis. The TEM images of the CdO nanoparticles synthesized from $[Cd(C_{11}H_6O_2)_2]$ at 120, 160, and 200°C are shown in Figure 4. All the nanoparticles obtained at different temperatures show spherical shaped particles with the average diameters of 17.99, 25.59, and 29.24 nm. The CdO nanoparticles obtained at low temperature (120°C) are aggregated due to the presence of organic materials and the high surface energy of the growth particles in nanoscale. CdO nanoparticles synthesized at higher temperature favour the thermodynamic growth system resulting in the formation of spherical particles with uniform shape (i.e., isotropic particles).

3.3. Deposition of CdO Thin Films by Spin Coating. The deposition of CdO thin films was carried out on the glass substrate at the temperature of 250, 300, and 350°C in a combustion tube which was inserted into the tube furnace under nitrogen gas environment. The prepared films were characterized by UV-vis, PL, XRD, and SEM.

CdO thin films were prepared by spin coating the precursor solution. Figure 5(a) shows UV-vis absorption spectra of the three-layer CdO thin films at 250, 300, and 350°C on the glass substrates. The optical band gap of the thin films deposited at 250°C was 3.65 eV. As the growth deposition temperature of the films increased from 300 to 350°C, the optical band gap was slightly red-shifted from 3.64 to 3.63 eV. The emission spectra of the CdO thin films prepared at different temperatures with the excitation wavelength of 300 nm are shown in Figure 5(b). The spectra are shifted towards the higher wavelength when the annealing temperature is increased with the main emission maxima at 480, 500, and 510 nm.

As CdO materials exhibit direct transition, $(\alpha h\nu)^2$ of the photon energy $h\nu$ can be plotted following Tauc relation [39] as shown in Figures 6(a)–6(c). The energy gap value of the CdO thin films can be found by extrapolating the linear part of the plot to $(\alpha h\nu)^2 = 0$. Figure 6(d) shows the optical band gap energies versus the temperature of the CdO bulk materials in the black solid line with a coloured sphere and the CdO nanostructured materials in the black solid line with a coloured box from the obtained optical spectra; it has been noted that as the annealing temperature is increased as represented in Figures 6(a)–6(c) in the black solid line with a coloured sphere, each plot reveals two optical band gaps at 2.51 and 3.87, at 2.42 and 3.75, and at 2.32 and 3.67 eV, respectively, for CdO thin films prepared at 250, 300, and 350°C. These observations were similar to the previously

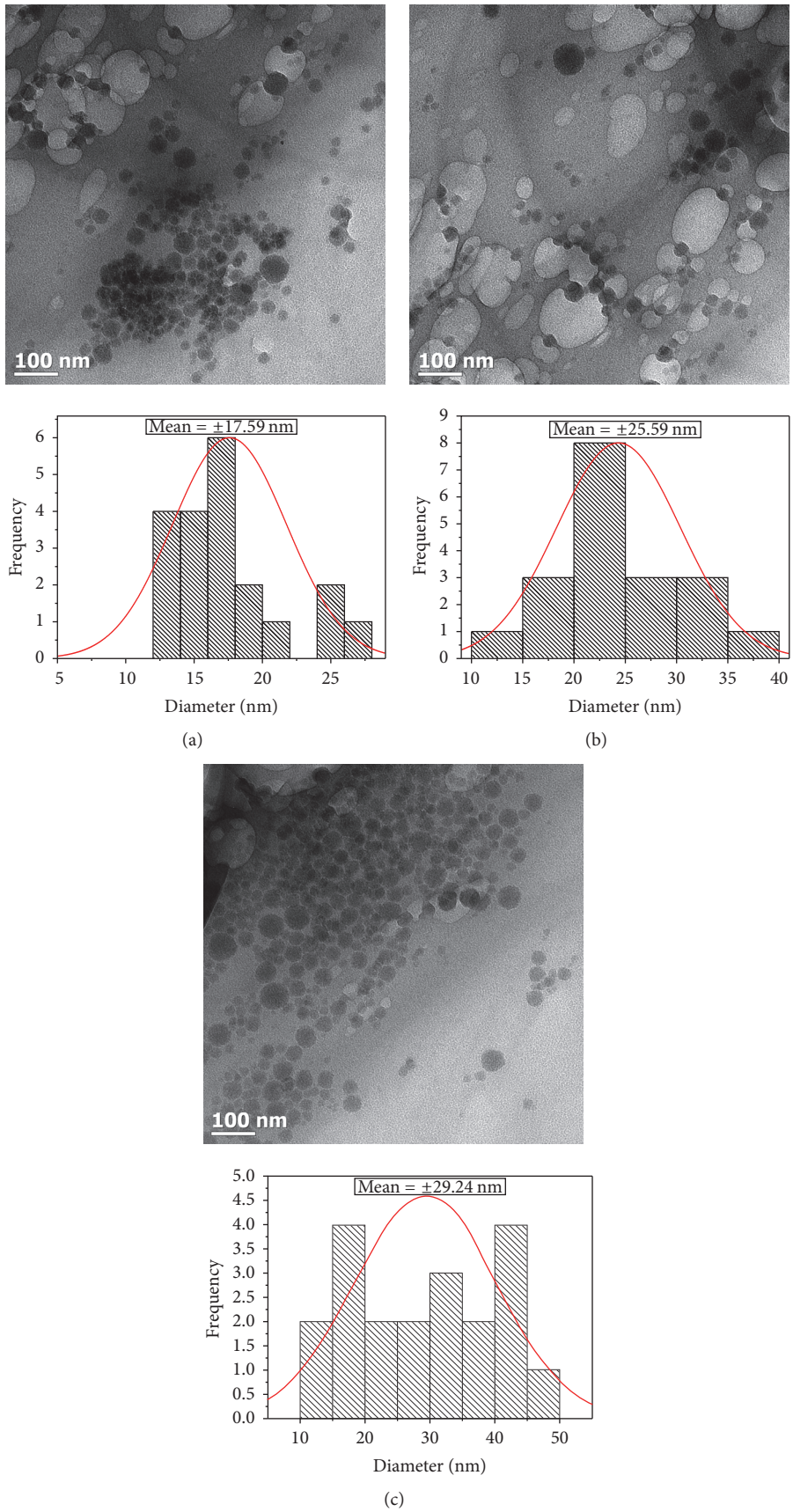


Figure 4: TEM images and frequency distribution of HDA-capped CdO nanoparticles synthesized at 120 (a), 160 (b), and 200°C (c).

Table 1: Elemental analysis by EDX (atomic % of CdO thin films at three different decomposition temperatures).

Temperature (°C)	Cd (%)	O (%)	Cd/O ratio
250	37.83	62.17	0.608
300	39.62	60.38	0.656
350	40.59	59.41	0.683

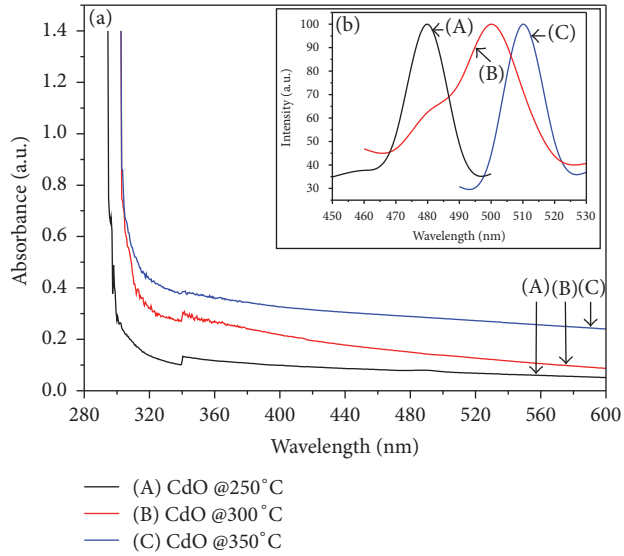


Figure 5: Absorption (a) and emission (b) spectra of CdO thin films prepared at 250 (A), 300 (B), and 350°C (C).

reported results by Al-Shakban and coworkers [40]. The highest energy band gap values in the black dotted lines can be attributed to nanostructured CdO thin films, whereas the lowest values in the black solid lines represent bulk CdO materials. These observations are in good agreement with the reported results by Naje et al. [41].

XRD Analysis. The crystalline structures of the thin films deposited at 250, 300, and 350°C were confirmed by XRD using Cu-Ka (l = 1.5406 Å) radiation. The XRD patterns of the annealed films are illustrated in Figure 7, respectively. The well-structured peaks at 2θ = 37.94, 41.82, 60.26, 78.53, and 80.64° correspond to (211), (222), (400), (331), and (420) planes that correspond to cubic structured nanomaterial which is in good agreement with the literature report (JCPDS card number: 00-039-1221).

SEM Analysis. SEM micrograms and their corresponding paint stroke images (i.e., attachments) of CdO thin films annealed at different temperatures are shown in Figure 8. SEM studies reveal that the morphology of the CdO films change as the deposition temperature is increased. CdO films prepared at 250°C in Figure 8(a) show the rough surface with nonuniform clustered growth predominant on the glass substrate as compared to Figures 8(b) and 8(c). The SEM images also show that slide surface becomes smooth when the annealing temperature becomes high. The corresponding SEM images in the form of paint strokes as attachments in Figure 8 verify SEM images. Energy dispersive X-ray spectroscopy (EDXS) at 20 kV confirmed the stoichiometry of CdO thin films deposited at 250, 300, and 350°C (Table 1). The EDX results prove that the percentage of cadmium increases when the annealing temperature is increased.

The surface morphologies of CdO thin films deposited at 250, 300, and 350°C were measured by AFM in contact mode at 5 × 5 μm area and are represented in Figures 9(a)–9(c). All the AFM images were analyzed in terms of average surface roughness (Ra). The images and data show that the average surface roughness (Ra) of the films decreases with the increase in deposition temperature. The average surface roughness (Ra) of the prepared CdO thin films at 250, 300, and 350°C was found to be 0.943, 0.827, and 0.644 nm, respectively. These results are corroborated with the SEM analysis reported in Figure 8.

4. Conclusions

Schiff base ligands have been utilized successfully for the preparation of cadmium(II) complexes. *Bis*(2-hydroxy-1-naphthaldehydato)cadmium(II) complex was used as a precursor for synthesizing CdO nanoparticles and their thin

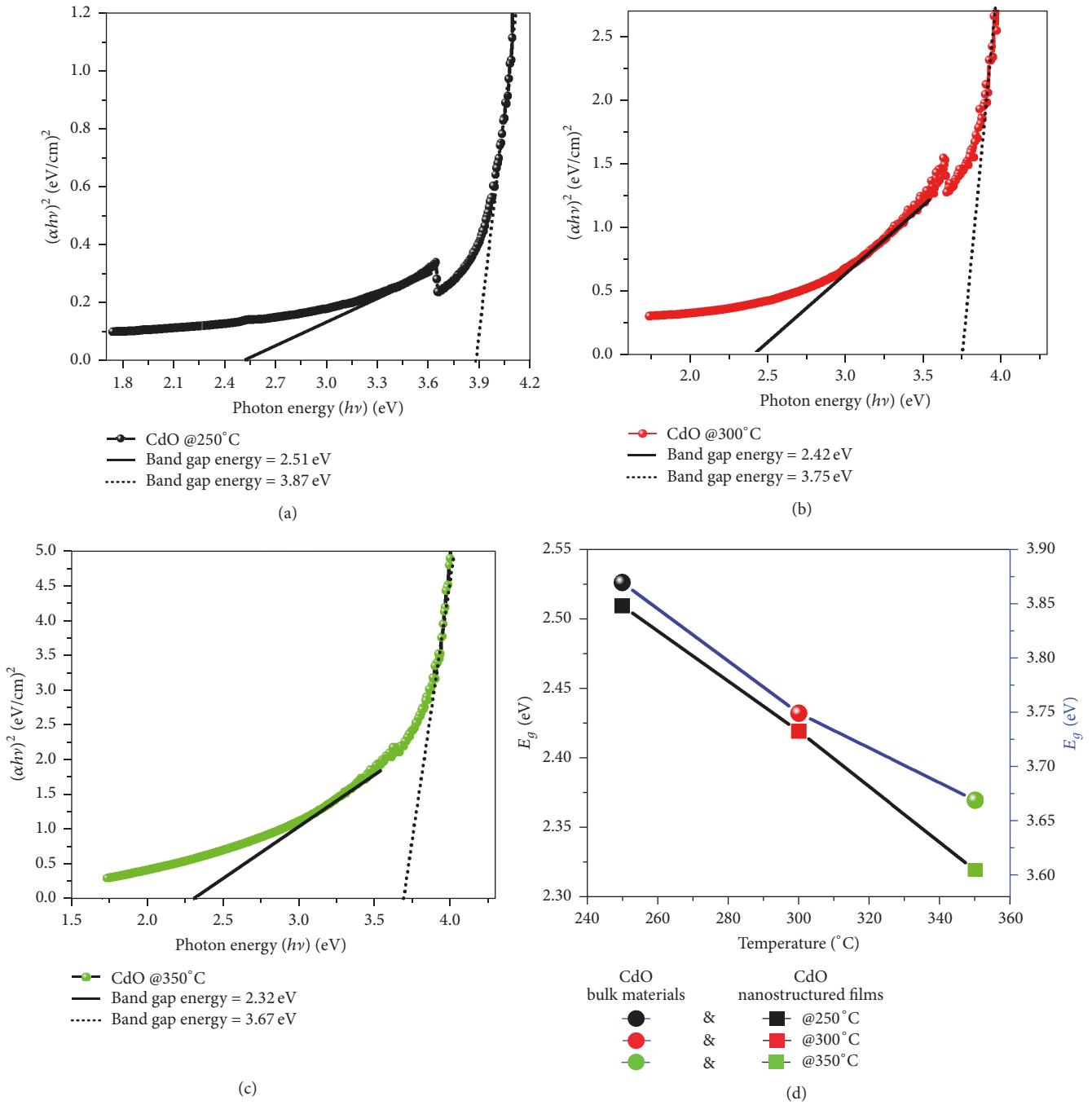


FIGURE 6: Variation of $(\alpha h\nu)^2$ versus $h\nu$ of the CdO thin films annealed at 250 (a), 300 (b), and 350°C (c) and the band gap energy (d).

films were also established. The HDA-capped CdO nanoparticles synthesized at different temperatures show the well-defined absorption band edges, with corresponding narrow band edge photoluminescence emissions. TEM images show spherical shaped particles for all the synthesized particles. CdO nanoparticles and their thin films which were deposited by a spin coater on glass substrates and annealed at different temperatures revealed face-centred cubic phase for all temperatures. The SEM images also show that as the annealing temperature is increased, the slide surface becomes smooth. A significant surface roughness reduction on CdO thin films has been observed by AFM topography, which is in agreement with SEM results.

Conflicts of Interest

The authors of this paper declare that there are no conflicts of interest regarding the publication of this article.

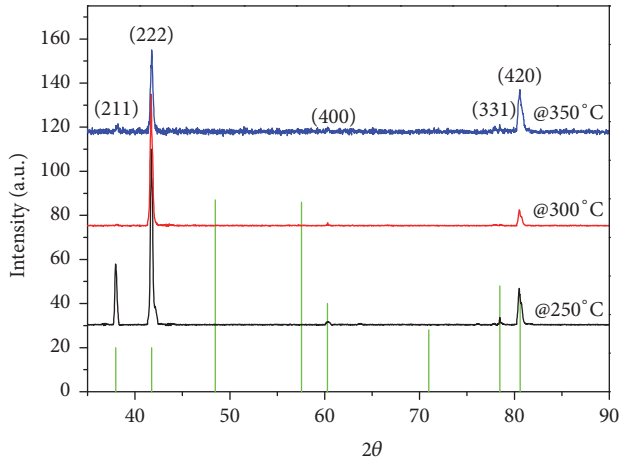
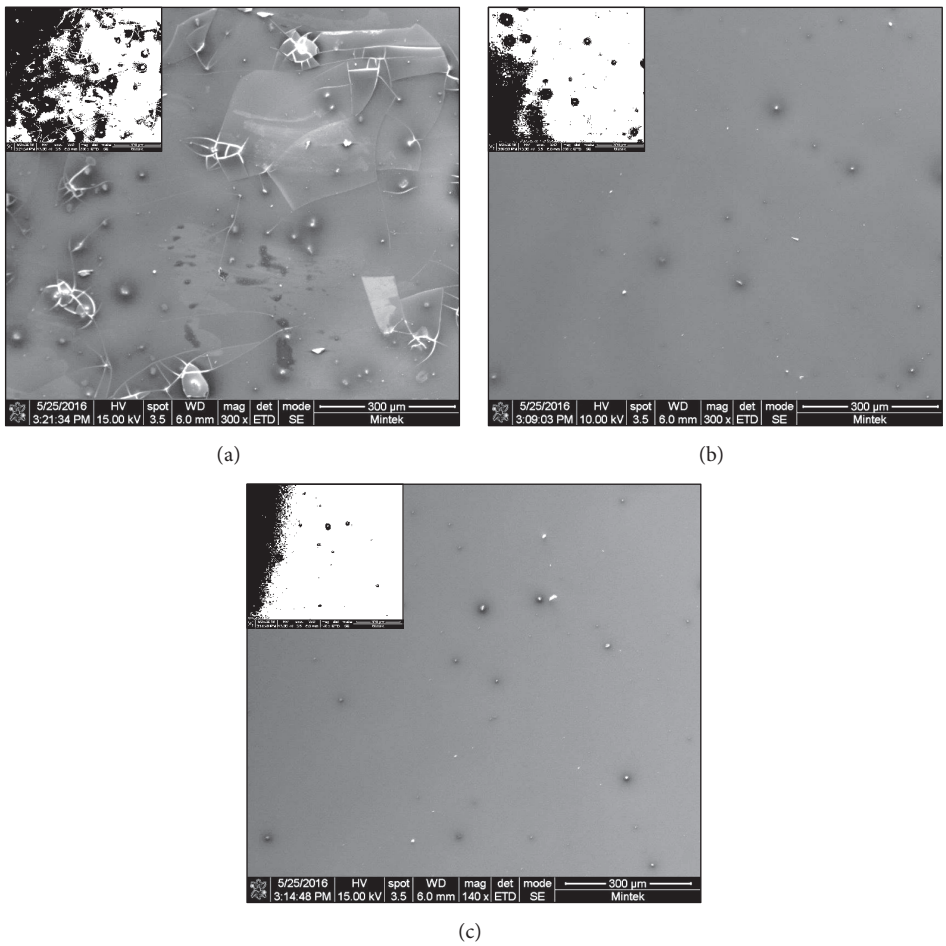


Figure 7: X-ray diffraction patterns of CdO thin films prepared at 250, 300, and 350°C.



(a) (b)

(c)

Figure 8: SEM micrographs and their corresponding paint stroke images (attachments) of CdO thin films prepared at 250 (a), 300 (b), and 350°C (c).

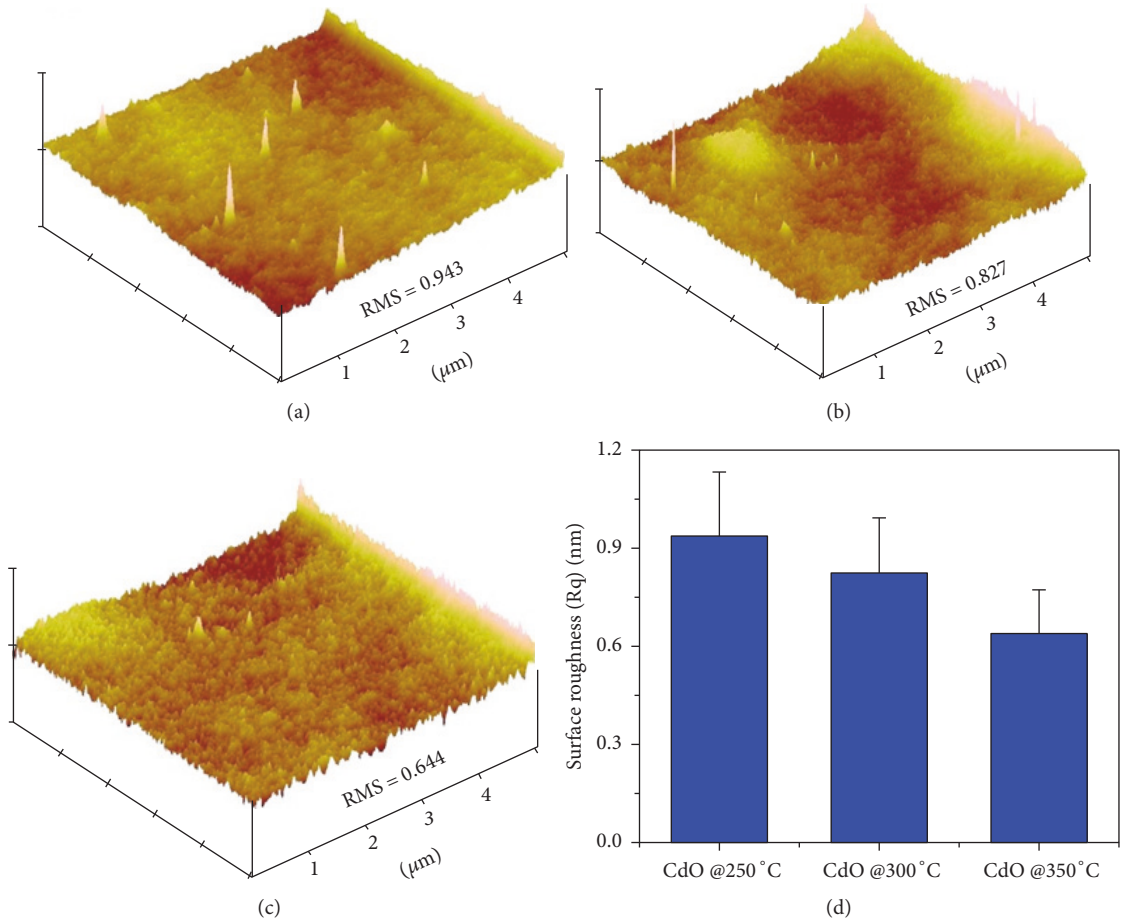


FIGURE 9: 3D AFM height profiles of CdO thin films deposited at 250°C (a), 300°C (b), and 350°C (c) and the surface roughness in terms of root mean square (RMS) roughness (Rq) (d) for scan size of 5.0 μm × 5.0 μm.

Acknowledgments

The authors would like to thank the University of Manchester for allowing them to use their facilities, Vaal University of Technology, and NRF (TTK13071722088: "Thuthuka Grant Holder") for funding this project.

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