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Solid state preparation of CdO nano-particles by thermolysis of a precursor as compacted nanosheet

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ABSTRACT

A novel complex of cadmium (II) $[CdL_{0.5}(NO_3)(H_2O)]$(1), L= *N*,*N*-bis (*O*-hydroxyacetophenone) ethylene diamine, have been synthesized as single crystal and nano size. Compound 1 in nano size was obtained under sonochemical irradiation and has been characterized by IR, ^{1}HNMR spectroscopy, X-ray powder diffraction (XRD) and scanning electron microscopy (SEM). The gray single crystal of compound 1 has been synthesized by slow evaporation and characterized by IR, ^{1}HNMR spectroscopy. Then, Compound 1 in nano size was used as initial reagent to obtain nano-particles of cadmium (II) oxide at 673 K by direct calcination. The obtained CdO nano-particles were characterized by XRD pattern as well as SEM images.

Keywords: *Nanoparticle; CdO; Sonochemistry; Cadmium (II) complex; Thermolysis.*

INTRODUCTION

Complexes have the ability to be highly crystalline materials that are constructed from molecular building blocks (metal ions, bridging ligands, counter anions and guest molecules). [1–3]. The selection or design of suitable ligands containing certain features, such as flexibility, versatile binding modes, and the ability to form hydrogen bonding, is crucial to the construction of metal–organic frameworks. Also, the combination of both π–π interactions and hydrogen bonding has proved to be particularly useful for assembly of complex structure and coordination polymers [4–7]. To date, the synthesis of huge structures in nano size has generated interest and they are synthesized by different methods and conditions, such as microwave, sonochemistry [8–10]. Complexes of Cd (II) with nitrogen donor ligands have also been used successfully in ligand exchange chromatography [11].

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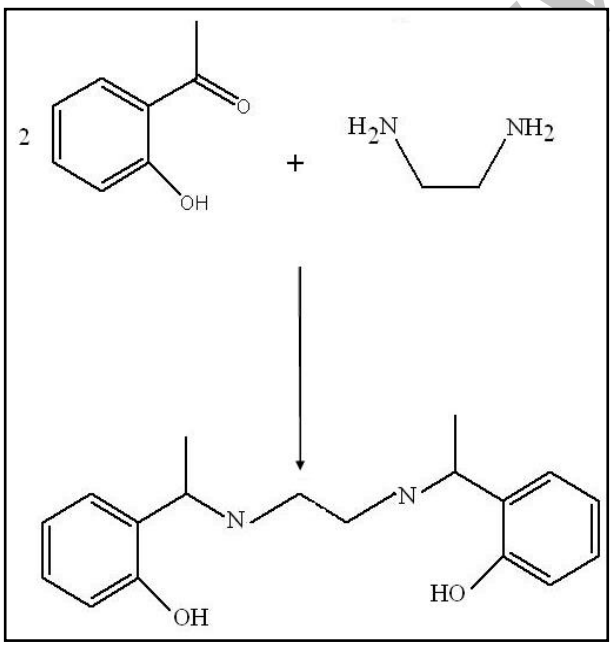
By decreasing the size of the coordination polymer crystallites from the bulk form to nano-size, their properties and applications could be improved. CdO-based transparent conductive oxides are very interesting because of their relatively simple crystal structures, high carrier mobility and sometimes nearly metallic conductivities. In this work, we report the preparation of cadmium (II) oxide nanoparticles from the complex (1) in nanosize as a precursor by electrical calcination in furnace.

EXPERIMENTAL

Synthesis of HPED as ligand (L)

N,N_-bis(*o*-hydroxyacetophenone)ethylene diamine Schiff base (HPED)was prepared by the procedure that reported in the literature [12]. The synthesis method has been shown in Scheme 1.

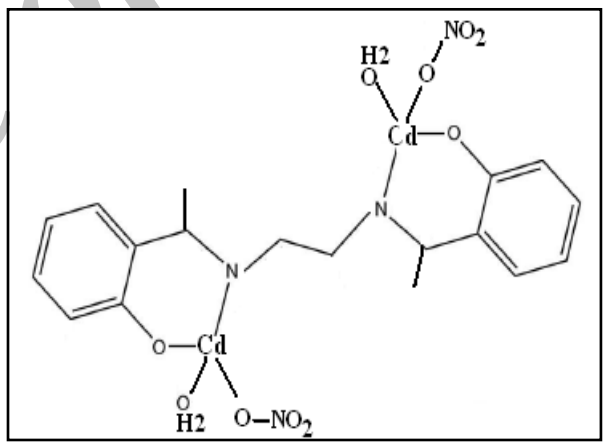
The reaction mixture containing o-hydroxyacetophenone (20 mmol, 2.22 g) and ethylene diamine (10 mmol, 0.6 g) in methanol was refluxed at 60 °C for about 45 min. The reaction mixture after cooling at low temperature produced yellow colored crystals, which were filtered and recrystallized in methanol.



Scheme1. Synthesis diagram of ligand L

Synthesis of compound (1) as single crystal and nano-size

To prepare compound 1, 0.145 g (1 mmol) cadmium (II) nitrate and 0.230 g (1mmol) sodium perchlorate were dissolved in methanol. This mixture was added into methanolic solution of 0.296 g (1 mmol) Schiff base ligand (L) under refluxing at 60ºC. The solution was retained two days at room temperature for slow evaporation and gray single crystals obtained. Scheme 2 shows cadmium (II) connects to nitrate anion, ligand (L) and H_2O molecules. To prepare the nanostructure of compound 1 by sonochemical process; an alcoholic (10 mL methanol) solution of 0.296 g (1 mmol) Schiff base ligand (L) was added under ultrasonic irradiation into a 10 mL methanolic solution of 0.145 g (1 mmol) cadmium(II) nitrate and 0.230 g (1 mmol) sodium perchlorate. m.p. = 250°C. IR (selected bands; in cm^{-1}): 577(w), 754(m), 1025(m), 1273(m),1343(m), 1607(s), 3365(s). ^{1}HNMR (DMSO, δ): 2.4(3H), 3.9(1H), 6.8(2H), 7.2(1H), 7.6(2H).



Scheme 2. The structure of compound 1

Preparation of cadmium (II) oxide nanoparticles by direct calcination

CdO nanoparticles were prepared by heating (0.421 mg, and 0.5 mmol) of nano-sized compound 1 at 673 K in electrical furnace.

RESULTS AND DISCUSSION

Gray shiny single crystals of compound 1, [$CdL_{0.5}(NO_3)$ (H_2O)], were isolated from slow evaporation method. The IR spectra of single crystal and nano size of compound 1 display the same absorption bonds. IR spectrum display some characteristic absorption at 1634 cm^{-1} and 1290 cm^{-1} are assigned to C= N and C–O(phenolic group), respectively. The relatively weak absorption bands at around 1205-1585 cm^{-1} is due to vibrations of the pyridine rings. Also, the characteristic band of the nitrate anion appears at 1343 cm^{-1} and the broad absorption band at 3365 cm^{-1} are assigned to the *v* (H_2O) modes. This broad band is attributable to *v* (O-H···X), indicating the presence of hydrogen bonds. IR spectra of compound 1 in bulk from and nano size are the same.

The morphology, structure and the size of compound 1 as nano-sheet were investigated by Scanning Electron Microscopy (Figure 1a, b).

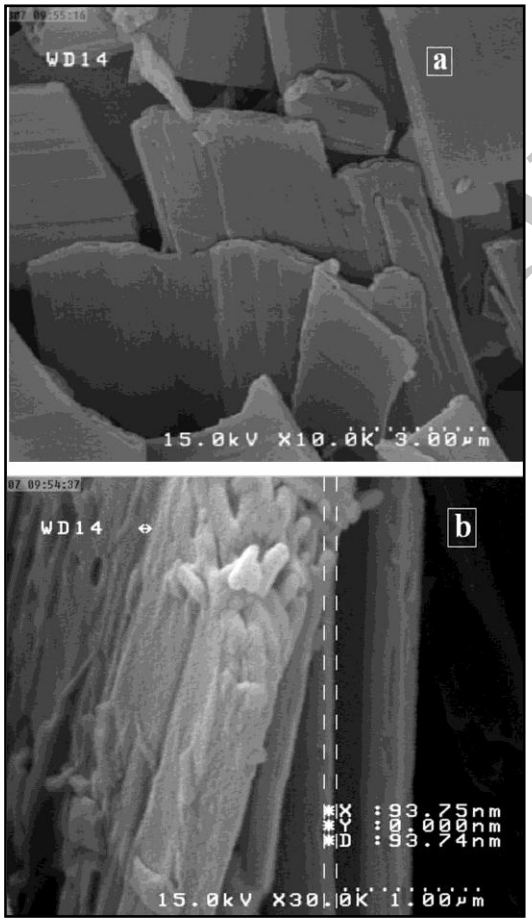


Fig. 1. SEM images of compound **1**nano-structure.

Figure 1a is an image of nano-sheets from the front and illustrates compound 1 as nano-sheets in 3µm scale bar. Figure 1b shows the edge of nano-sheets in 1µm scale bar. As shown in these figures the nano-sheets are stuck together and formed thicker sheets.

Figure 2 shows the SEM image of cadmium (II) oxide nanoparticles from the decomposition of compound 1 at 400°C by direct calcinations. The observed particles have 40–60 nm range sizes as figure shows.

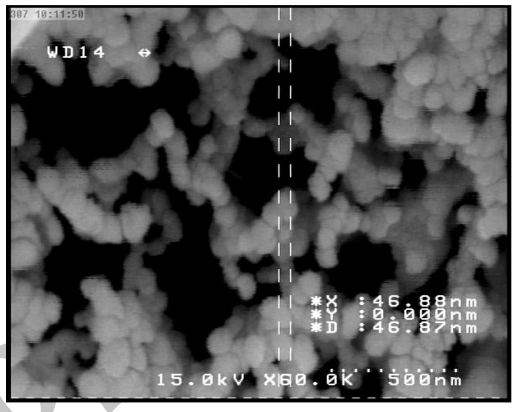


Fig. 2. The SEM image of cadmium(II) oxide nanoparticles prepared by calcination of nano-size compound 1.

Figure 3 shows the XRD pattern of cadmium (II) oxide after calcinations for 2 hours at 400°C. The XRD pattern match the standard pattern of cadmium (II) oxide with these lattice parameters (Sys: Cubic S.G.: Fm3m a= 4.6953Å and z = 4) which are the same as reported values (JCSD: 050640). Sharp diffraction peaks shown in Figure 3 indicate good crystallinity of CdO nanoparticles.

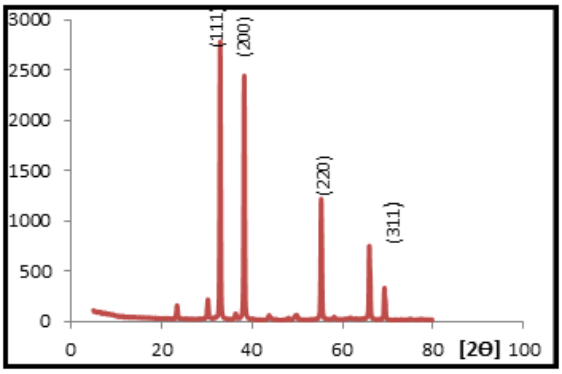


Fig. 3. XRD pattern of the prepared CdO nanoparticles

The EDAX shows the presence of cadmium and oxygen as the elementary components (Figure 4).

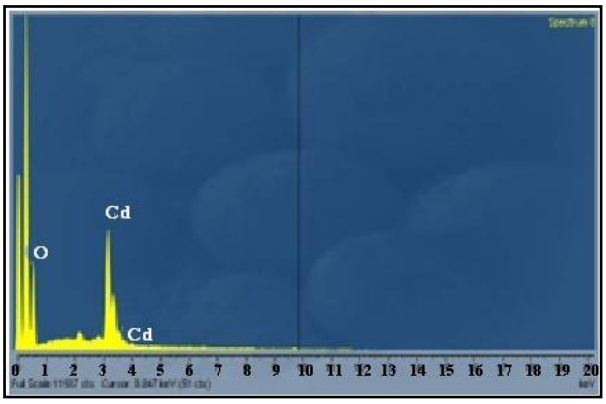


Fig. 4. Energy-dispersive X-ray analysis

The histogram of obtained cadmium (II) oxide nanoparticles from decomposition method has been shown in Figure 5.

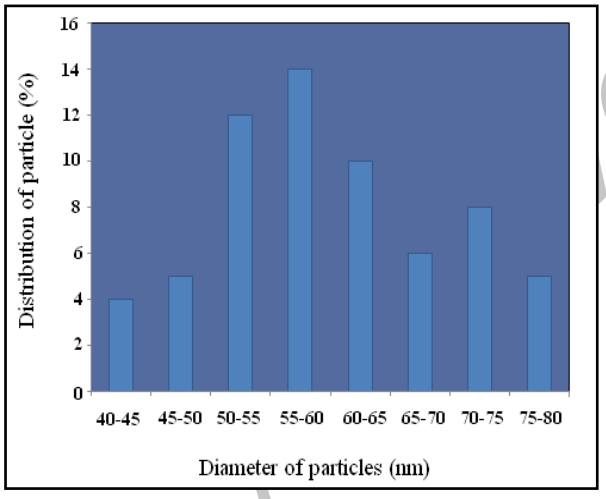


Fig. 5. The histogram of CdO nanoparticles

CONCLUSIONS

Nanosheets of compound 1 have been synthesized by the sonochemical method. Nanoparticles of cadmium (II) oxide were fabricated by direct calcination of nano-sized compound 1 and the SEM image of the cadmium (II) oxide nanoparticles shows perfect morphology. This study demonstrates direct calcination could be a method for metal oxide preparation and some other supramolecules such as complex. So, some supramolecular compounds may be suitable precursors for perpetrating other nanoscale materials such as metal oxides.

ACKNOWLEDGMENTS

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