

Article

Ultrasound-Assisted Synthesis and Crystal Structure of Novel 2D Cd (II) Metal–Organic Coordination Polymer with Nitrite End Stop Ligand as a Precursor for Preparation of CdO Nanoparticles

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Abstract: In the present research, a sonochemical approach was applied to prepare new cadmium(II) coordination 2D polymer, $[Cd(L)(NO_2)_2]_n$ ($L = 1,2$ -bis(1-(pyridin-3-yl)ethylidene)hydrazine) and structurally characterized with various spectroscopic techniques including XRD, elemental analysis, SEM, and IR spectroscopy. The coordination number of cadmium (II) ions is seven (CdN_2O_5) by two nitrogen atoms from two organic Schiff base ligand and five oxygen of nitrite anions. The 2D sheet structures ended by nitrite anions and the nitrite anion displayed the end-stop role. The comprehensive system showed a three-dimensional structure with several weak interactions. The high-intensity ultrasound is regarded as an easy, environmentally-friendly, and flexible synthetic instrument for the compounds of coordination. CdO NPs was obtained by thermolysing 1 at 180 °C with oleic acid (as a surfactant). Further, the size and morphology of the produced CdO nanoparticles were investigated through SEM.

Keywords: crystal structure; coordination polymer; sonochemical route; X-ray crystallography



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1. Introduction

Recently, the development of metal–organic coordination systems has become increasingly attractive for researchers [1–3]. Ascetic architecture, luminescence, topologic diversity, magnetic, storage molecular, adsorption, covering, various preparation manners, and other interesting possible usage are among the factors attracting attention and results in various examinations leading to many research works in the area of coordination polymers and novel group of material chemistry [4–6].

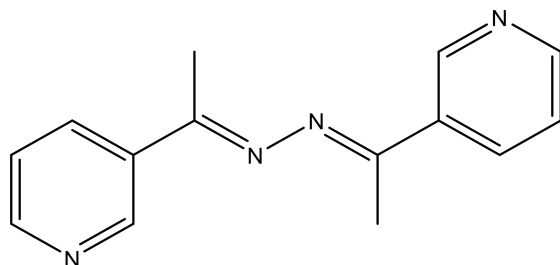
Schiff bases that are a significant series of organic ligands have the capability of coordination by metal ions for forming stable chelates through the N atom of the azo-group as well as other donors, like $-NH_2$ and $-OH$. Schiff bases have advantages of satisfactory biological activity, simple synthetic pathways, high coordination potency, and photochemical features, and they include pharmaceuticals (e.g., antibiotics, anti-inflammatory drugs, and antivirals) and also photoelectric and dye materials [7–12]. Moreover, because of the variety of structure, potential features, and preparative accessibility, metal complexes with Schiff base ligands are considered a significant stereochemical model in metal coordination chemistry. The common coordination mode of these ligands is different and very interesting [13]. Because of the relatively simple yet robust synthetic procedure, Schiff base formation and complexation have formed the basis of a number of elegant reported undergraduate experiments that explore various aspects of Schiff base chemistry, from the formation of organometallic complexes to combinatorial synthesis to spectral analysis. In this experiment, Schiff base ligands are used to demonstrate the effect of metal ions on metal–ligand stoichiometry [14].

Metal–organic coordination systems can be used as templates in order to obtain the desired nano-materials. Via selecting proper metal–organic compounds precursors with special morphologies and under suitable experimental conditions, acquiring the desired morphologies will become possible. Nano metal–organic coordination compounds precursors may also be used to obtain nano-materials with better and more useful properties [15–18]. Depending on the conditions, metal–organic coordination compounds precursor may lead to the preparation of metals, metal oxides, metal sulfides, or other nano-sized materials. With this method, nano-materials can be obtained in different morphologies from different metal–organic coordination systems precursors.

A small number of the transition metals' characteristic attributes are demonstrated by Cadmium and group 12 elements because they do not partially fill d or f electron shells. Cadmium, like zinc, has a preference for a + 2 oxidation state in the majority of its complexes. However, the more important point is that these elements with spherical d^{10} configuration are the facilitators of different coordination geometries, allowing forming of 1D, 2D, and 3D coordination compounds [19–21].

The focus of the present study is the preparation and design of metal–organic coordination systems, and the synthesis of some nano metal–organic coordination polymers have been recently reported by our research team [22–26]. The next phase of our team in this regard is an investigation of the behaviors of Cadmium (II) in terms of coordination by 2-bis(1-(pyridin-3-yl)ethylidene)hydrazine Schiff-base ligand in the present work.

The near similar works on these compounds were reported in previous literature with various composite and applicant (see [27]). Especially, the ligand's structural chemistry is attractive due to its multifunctional coordination state which four aromatic and aliphatic nitrogen can be coordinated to metals (Scheme 1). A simple sonochemical preparation process is described in this work for a nanostructure for this coordination system and its usage in the construction of Cadmium oxide (CdO) NPs.



Scheme 1. Schematic of 2-bis(1-(pyridin-3-yl)ethylidene)hydrazine Schiff-base ligand.

2. Experimental

2.1. Instruments and Materials

All chemical compounds were bought from the Aldrich and Merck (Germany and Merck Company, Darmstadt, Germany) chemical companies. A Bruker Vector 22 FT-IR (Richmond Scientific, Chorley, UK) spectrometer with KBr disks in the area of $400\text{--}4000\text{ cm}^{-1}$ was used for acquiring IR spectroscopy. An Electrothermal 9000 (Keison Products, Chelmsford, UK) apparatus determine the melting points. A multiwave ultrasonic generator (Sonicator_3000; Misonix, Inc., Farmingdale, NY, USA) was applied. X'pert diffractometer (produced by Panalytical, Panalytical, Malvern, UK) monochromatized $\text{Cu K}\alpha$ radiation was employed for performing measurements of PXRD. Mercury 2.4 was used for preparing simulated PXRD powder patterns on the basis of single-crystal data [28]. Elemental analyses were carried out using a linked ISIS-300, Oxford EDS (Oxford Instruments, Abingdon, UK) (energy dispersion spectroscopy) detector. Chemical states and chemical compositions of the CdO nanoparticles were carried out through X-ray photoelectron spectroscopy (XPS) (Model: K-ALPHA, Thermo Fisher, Waltham, MA, USA).

The Scherrer formula was employed for estimating the crystallite size of chosen samples. Followed by gold coating Au, the samples' morphology was assessed using an

SEM (Hitachi, Japan). Using an Xcalibur™ diffractometer with a Sapphire2 CCD detector and Mo K α radiation (monochromator Enhance, Oxford Diffraction Ltd., Abingdon, UK) and ω -scan rotation methods at 150 K, diffraction data were collected for the single crystal. The CrysAlis program package (Oxford Diffraction Ltd., UK) was utilized for data reduction and gathering and refinement of cell parameters [29].

Multi-scan absorption modification with the CrysAlis (Rigaku, Tokyo, Japan) was used for the [Cd(L)(NO₂)₂]_n data. With a full matrix weighted least-squares method (SHELX-2014) (Free Software Foundation, Boston, MA, US) with the $w = 1/[\sigma^2(F_o)^2 + (0.035P)^2]$ weight, where $P = (F_o^2 + 2F_c^2)/3$, we solved the structure with direct processes by SHELXS and adjusted on all F² data anisotropically. Mercury 2.4 was applied for creating molecular exhibits. Crystal data and arrangement modification for 1 are provided in Table 1, and the designated bond lengths and angles can be observed in Table 2.

Table 1. Crystal data and structure refinements of complex.

Chemical Formula	C ₁₄ H ₁₄ CdN ₆ O ₄
M _r	442.7
Crystal system, space group	Orthorhombic, P2 ₁ 2 ₁ 2 ₁
Temperature (K)	120
a, b, c (Å)	4.5208 (1), 16.2510 (2), 22.0539 (4)
V (Å ³)	1620.25 (5)
Z	4
Radiation type	Cu K α
μ (mm ⁻¹)	11.12
Crystal size (mm)	0.38 × 0.10 × 0.03
Diffractometer	Goniometer Xcalibur, detector: Atlas (Gemini ultra Cu)
Absorption correction	Analytical CrysAlis PRO, Agilent Technologies (2010), Analytical numeric absorption correction using a multifaceted crystal model
T _{min} , T _{max}	0.181, 0.723
No. of measured, independent and observes [1 > 2 σ (1) reflections]	20706, 2895, 2742
R _{int}	0.039
(sin θ / λ) _{max} (Å ⁻¹)	0.598
Refinement R[F ² > 2 σ (F ²)], wR(F ²), S	0.020, 0.057, 1.13
No. of reflections	2895
No. of parameters	227
H-atom treatment	H-atom parameters constrained
Δ _{max} , Δ _{min} (e Å ⁻³)	0.30, -0.37

Table 2. Chosen bond lengths [Å] & angles [°] for 1.

1.234 (4)	O2—N5	2.390 (2)	Cd1—O1
1.252 (5)	O3—N6	2.368(2)	Cd1—O3
1.255 (5)	O4—N6	2.470(2)	Cd1—O2
1.335 (5)	N1—C1	2.389(2)	Cd1—O4
1.340 (5)	N1—C5	2.328 (2)	Cd1—O1 ⁱ
1.397 (5)	N2—N3	2.358 (2)	Cd1—N1 ⁱ
1.291 (5)	N2—C6	2.356 (2)	Cd1—N4
1.346 (5)	N4—C12	1.288 (5)	N3—C8
120.2 (2)	Cd1 ⁱⁱ —N1—C5	1.339 (5)	N4—C11
154.44 (9)	N1 ⁱ —Cd1—N4	87.27 (11)	O1—Cd1—N1 ⁱ
121.9 (2)	Cd1 ⁱⁱ —N1—C1	89.09 (10)	O1—Cd1—N4

Symmetry code(s): (i) $-x, y + 1/2, -z + 1/2$; (ii) $-x, y - 1/2, -z + 1/2$.

2.2. Preparation of 1,2-Bis(1-(pyridin-3-yl)ethylidene)hydrazine Ligand

20 mmol (1.9 mL, 2.42 g) of 1-(pyridin-3-yl)ethan-1-one was dissolved in 50 mL of ethanol, then 10 mmol (0.9 mL, 0.91 g) of hydrazine hydrate (35 wt % solution in water), softened in 20 mL of ethanol, was mixed in a drop-wise manner; subsequently, two acetic

acid drops were mixed with it too. The stirring of the reaction mix was done for 24 h at ambient temperature. Finally, the yellow sediments were filtered and air-dried (yield: 77%, 2.56 g product) (Comparable with previous synthesized [30,31]).

FT-IR (KBr, cm^{-1}): 758(m), 823(w), 563(m), 1290(s), 1365(s), 1415(s), 1485(s), 1558(s), 1600(s).

Elemental analysis calculated for $\text{C}_{14}\text{H}_{14}\text{N}_4$: C: 70.57, H: 5.92, N: 23.51%.

Experimental: C: 70.10, H: 6.00, N: 24.00%.

2.3. Synthesis of Nano-Structure of $[\text{Cd}(\text{L})(\text{NO}_2)_2]_n$

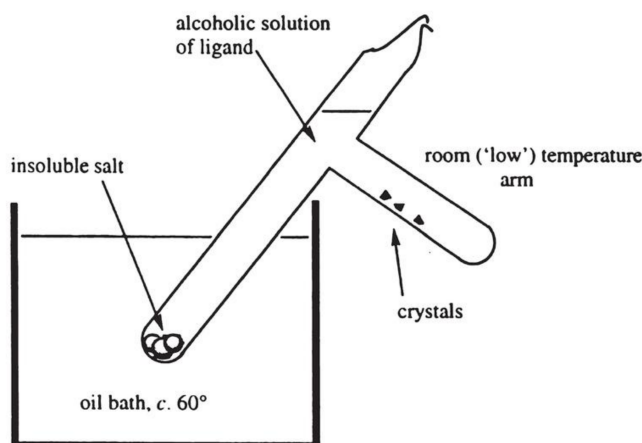
For synthesizing, the nanostructure of $[\text{Cd}(\text{L})(\text{NO}_2)_2]_n$ a solution of cadmium(II) nitrate tetrahydrate (15 mL of a 0.1 M) in water was put in a high-density ultrasonic probe operating at 20 kHz by the highest power output of 60 W. 15 mL of a 0.1 M solution of the Schiff base ligand and 30 mL of a 0.1 M solution of sodium nitrite were added into this solution in a drop-wise manner. The resulting yellow precipitates were filtered, rinsed with H_2O and MeOH, and dried at 50 °C (0.69 g product/yield: 65% decompose point: 210 °C)

Elemental analysis; calculated for $\text{C}_{14}\text{H}_{14}\text{CdN}_6\text{O}_4$: C: 37.98, H: 3.19, N: 18.98%.

Experimental: C: 38.00, H: 4.10, N: 20.00.

2.4. Synthesis of Isolate Single Crystal of $[\text{Cd}(\text{L})(\text{NO}_2)_2]_n$

For isolating $[\text{Cd}(\text{L})(\text{NO}_2)_2]_n$ single crystals, Schiff base ligand (1 mmol/0.24 g), sodium nitrite (2 mmol/0.14 g) and cadmium(II) nitrate tetrahydrate (1 mmol/0.31 g) were put in the primary arm of a branched tube for heating (Scheme 2) [32].



Scheme 2. Brached tube method [32].

Both arms received methanol carefully. The tube was sealed, and the arm containing ligand was submerged in an oil bath at 60 °C; however, the other arm was stored at ambient temperature. Following two weeks, yellow crystals were precipitated in the colder arm for X-ray structure determination that was filtered, rinsed by acetone and ether, and being dried with exposure to air. (0.59 g product/Yield 89%—decompose point: 210 °C) Elemental analysis; calculated for $\text{C}_{14}\text{H}_{14}\text{CdN}_6\text{O}_4$: C: 37.98, H: 3.19, N: 18.98%. Experimental: C: 38.00, H: 4.00, N: 20.00

3. Synthesis of CdO Nanoparticles

The precursor of $[\text{Cd}(\text{L})(\text{NO}_2)_2]_n$ (**1**) (0.02 mmol) was dissolved instantly in 1.0 mL of oleic acid, which formed a light yellow solution. This solution was degassed for 20 min and then heated to 180 °C for 2 h. At the end of the reaction, an orange precipitate was formed. A small amount of toluene and a large excess of EtOH were added to the reaction solution, and cadmium (II) oxide nanoparticles were separated by centrifugation. The light brown solids were washed with EtOH and dried under an air atmosphere (0.01 g product/yield: 58%).

4. Results and Discussion

The reaction of 1,2-bis(1-(pyridin-3-yl)ethylidene)hydrazine Schiff base ligand with a mixture of sodium nitrite and cadmium nitrate, the novel Cd (II) metal–organic coordination polymer $[\text{Cd}(\text{L})(\text{NO}_2)_2]_n$ was obtained. Using ultrasonic irradiation in a methanolic solution, the compound's nanostructure was achieved; then, the single crystalline substance was attained through application of the heat gradients to the reagents solution or branched tube route. Scheme 3 presents the two various routes utilized for preparing $[\text{Cd}(\text{L})(\text{NO}_2)_2]_n$ materials.

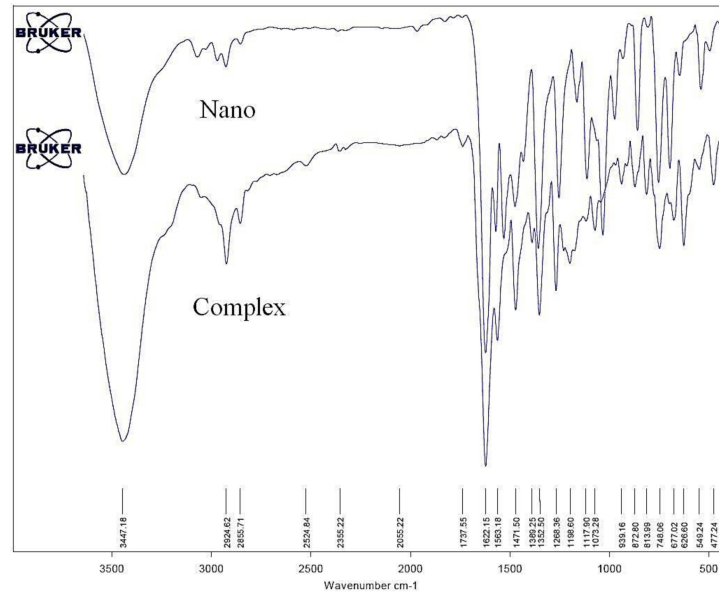
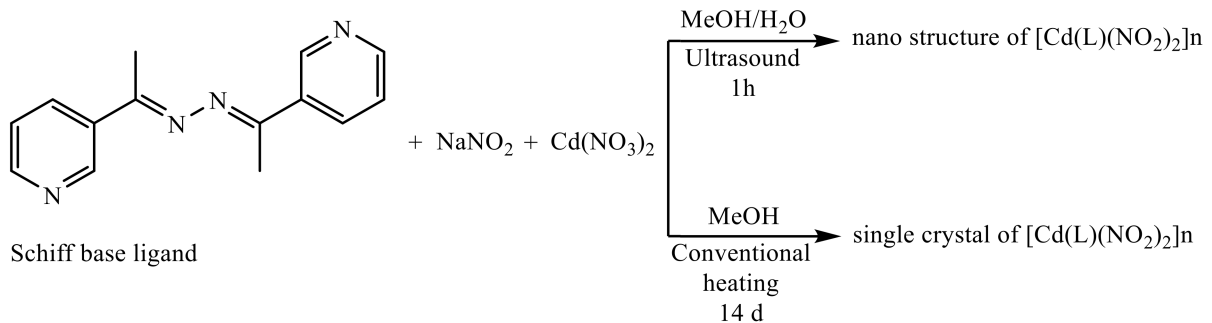


Figure 1. FT-IR spectra of the complex and nano-structures of coordination polymer.



Scheme 3. The synthetic techniques and the produced materials. The FT-IR spectra of synthesized crystal and nano-structure of coordination polymer ($[\text{Cd}(\text{L})(\text{NO}_2)_2]_n$) indicate the characteristic absorption bands for organic Schiff base ligand. The weak band near $3000\text{--}3100\text{ cm}^{-1}$ is assigned to aromatic C–H groups' stretching vibrations. The C=N tensile band is a sharp and strong peak in the 1622 area. The absorption band in the $1400\text{--}1600\text{ cm}^{-1}$ range is attributed to the Schiff base ligand's aromatic ring vibrations. There is a good match between FT-IR spectra of the complex and nano-structure in all areas (Figure 1).

The surface morphology of the synthesized nano coordination compound was investigated through FE-SEM (field emission scanning microscopy) analysis. The morphology of the compound prepared by the sonochemical method is block-like aggregates (Figure 2). SEM assessments illustrate that achieved nanoparticles are of various sizes and have different shapes. Figure 3 showed the Simulated XPRD pattern of $[\text{Cd}(\text{L})(\text{NO}_2)_2]_n$ in comparison with nanostructure XRD pattern, Acceptable matches with slight differences in 2θ were observed between the simulated and experimental XPRD patterns (Figure 3). This indicates that the synthesis obtained by the sonochemical method as nanoparticles is identical to

that obtained by crystallography. The significant broadening of the peaks indicates that the particles are of nanometer dimensions. As it has been estimated by the Sherrer formula ($D = 0.891\lambda/\beta\cos\theta$, where D is the average grain size, λ the X-ray wavelength (0.15405 nm), and θ and β the diffraction angle and full-width at half maximum of an observed peak, respectively) [33], the obtained value is $D = 83$ nm.

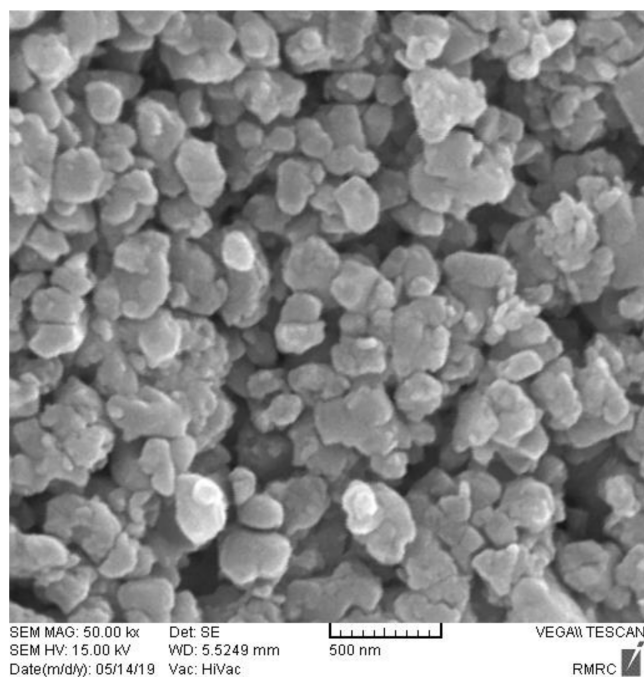


Figure 2. SEM images of the $[\text{Cd}(\text{L})(\text{NO}_2)_2]_n$.

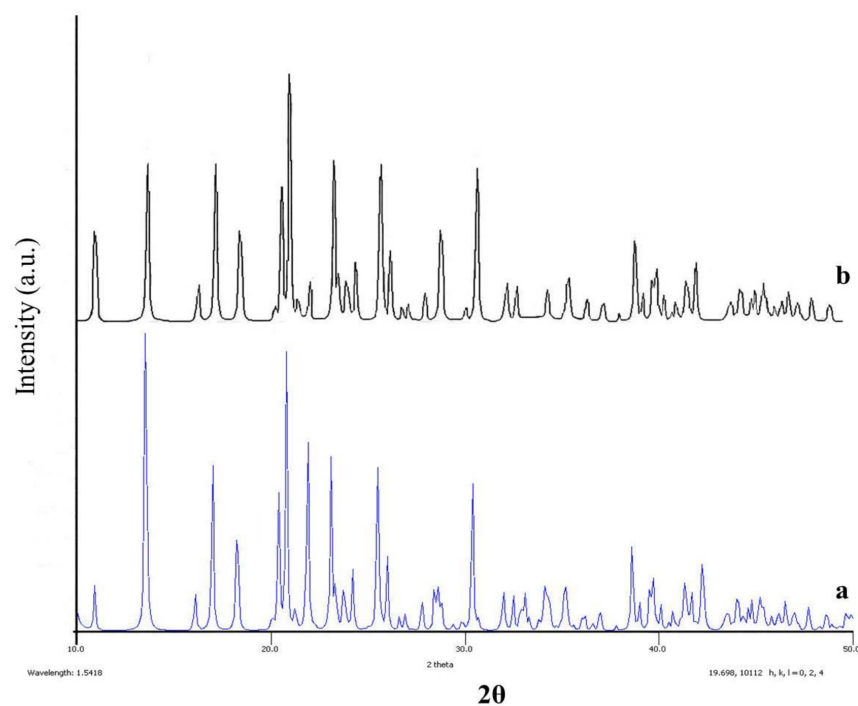


Figure 3. The PXRD patterns of compound $[\text{Cd}(\text{L})(\text{NO}_2)_2]_n$ (**1**): (a) Simulated from single-crystal X-ray data and (b) nanostructure of (**1**) prepared by sonochemical method. Based on the crystal structure of **1**, the composition and stereochemistry of a building block $[\text{Cd}(\text{L})(\text{NO}_2)_2]_n$ were disclosed. A view of the molecular structure is shown in Figure 4.

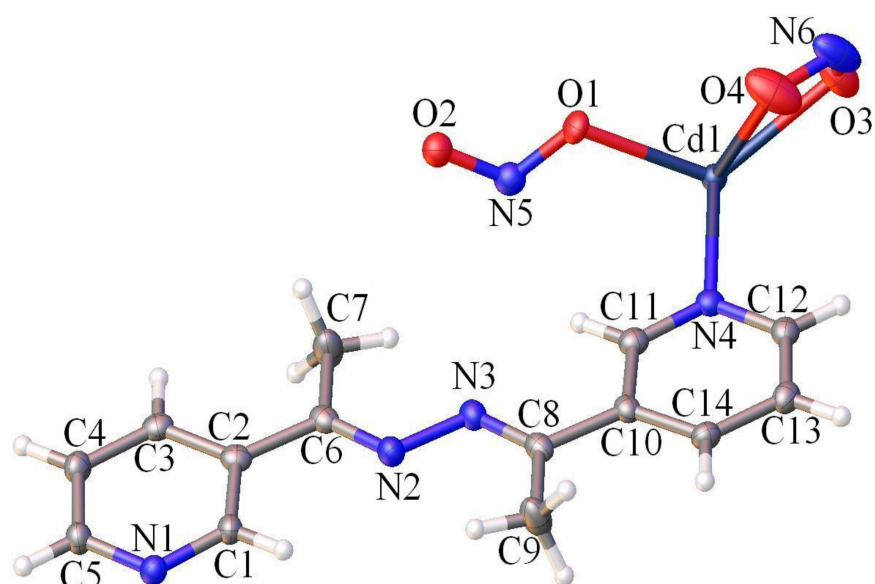


Figure 4. Asymmetric unit of the $[Cd(L)(NO_2)_2]_n$.

The determination of the structure of coordination polymer $[Cd(L)(NO_2)_2]_n$ displayed that the complex crystallizes in the Orthorhombic system with space group $P2_12_12_1$, taking the form of a two-dimensional polymer in the solid-state (Figure 5). The Cd (II) atom is coordinated with two nitrogen atoms of two Schiff base ligands with the Cd–N distances of 2.356 (2), 2.358 (2) Å, five oxygen atoms of three nitrite anions. In addition distance of Cd–O bonds in this complex are around 2.368–2.247 Å. Therefore, the coordination number of the Cd (II) atom is seven, with an orthorhombic system with space group $P2_12_12_1$ (Figures 4 and 5) (See Appendix A for further details). The 2D sheet structures ended with nitrite anions. In this structure, the nitrite anion showed the end-stop role and by connecting directly to the metal, does not allow the system to expand to the third dimension.

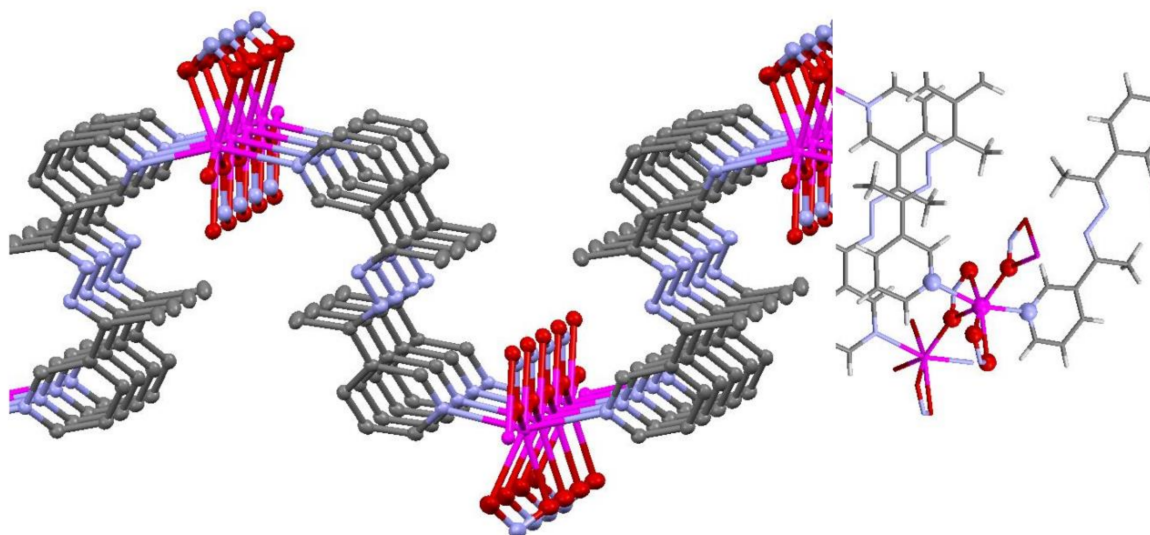


Figure 5. The coordination polymer fragment showing the 2D arrangement of complex (Left), The coordination sphere of Cd(II) (Right).

The structure of other compounds reported from this ligand to Mn, Ag, Co, Zn, Bi, and Hg metals with varying coordination numbers for metals and the same coordination method for ligands is comparable [34–38].

There are many weak interactions exist in the structure; (like short contact involving C-H groups and aromatic interactions) (See Table 3. For example, there are pi–pi stacking interactions between the N1C5C4C3C3C1 plans about 3.770 Å and between the N4C12C13C14C10C11 plans about 3.474 Å. With several weak interactions, the 2D chains interact with neighbors, and the system extended to 3-dimensional structures (Figure 6 and Table 3).

Table 3. Selected non-covalent contacts in the crystal structure of **1** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(13)-H(13)...O(4)	0.96	2.525	3.237(4)	130.9
C(13)-H(7)...C(12)	0.96	2.708	2.789(4)	155.5
C(4)-H(4)...O(3)	0.95	2.572	3.442(5)	151.2
C(1)-H(1)...N(5)	0.96	2.514	3.328(4)	142.6
C(5)-H(5)...O(3)	0.95	2.641	3.674(5)	145.7

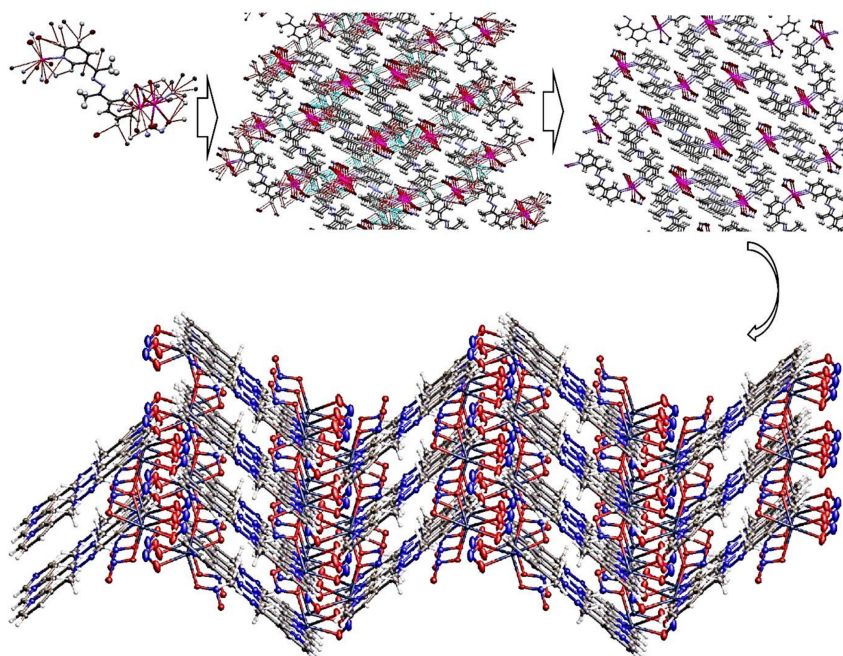


Figure 6. From di nuclear complexes to 3D networks.

The decomposition of precursor $[\text{Cd}(\text{L})(\text{NO}_2)_2]_n$ in oleic acid functioning as a surfactant under an air atmosphere at 180 °C yields nano-powder of cadmium (II) oxide. The PXRD pattern of as-synthesized CdO nanopowder is seen in Figure 7. All the diffraction peaks are matched well with the cubic crystal structure of pure CdO with respect to their positions as found in JCPDS card No.05-0640.

Figure 8 displays the energy-dispersive X-ray spectroscopy (EDS) of the CdO nanoparticles and confirms the existence of the elements Cd/O and the formation of CdO as expected. The atomic percentage composition of the elements Cd and O were observed to be equal to 50.79 and 49.21, respectively.

Figure 9a displays the SEM image of as-prepared CdO nanoparticles. The obtained CdO has the regular nanoparticle shape with diameters of around 30–80 nm according to the size distribution graph of the product which was determined using Nahamin microstructure distance measurement software (Iran). (Figure 9b).

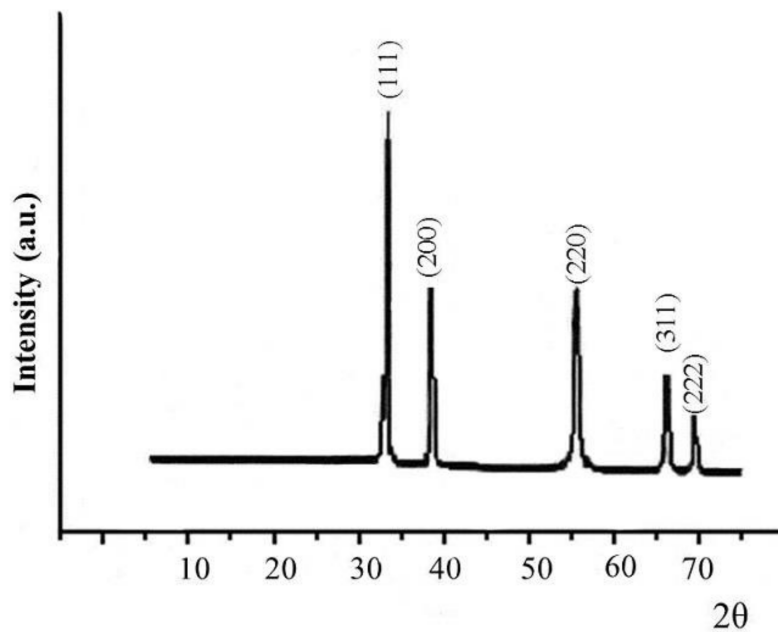


Figure 7. XRD pattern of cadmium oxide obtained by decomposition of (1).

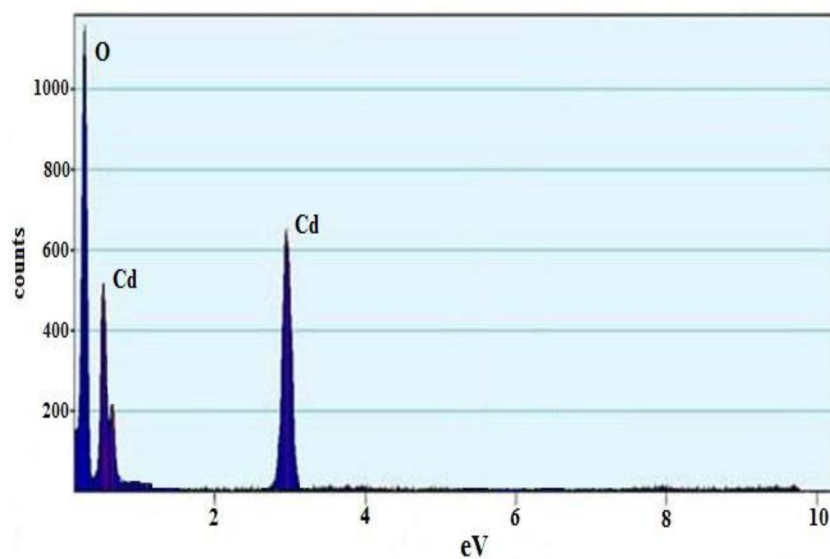


Figure 8. EDX of the as-prepared CdO nanoparticles.

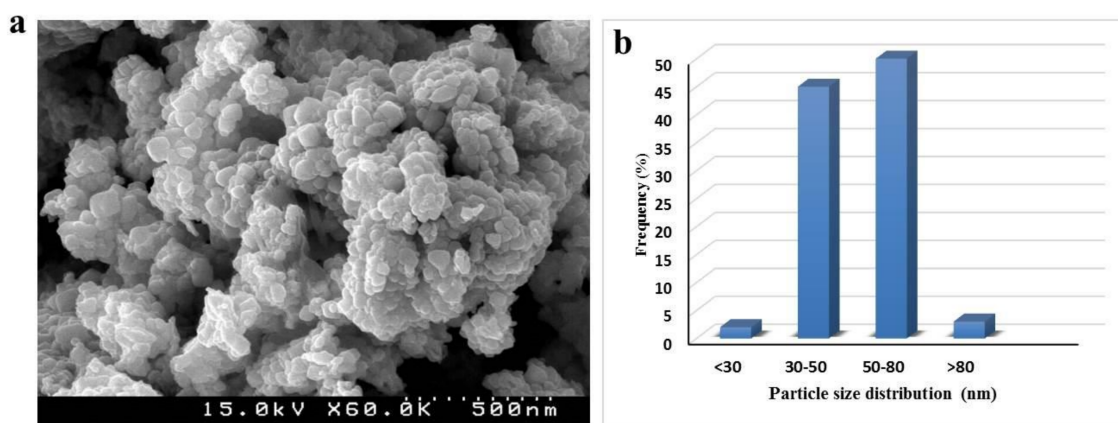


Figure 9. (a) SEM photograph and (b) size distribution graph of nano cadmium oxide (obtained by thermolysis of precursor 1).

The XPS spectrum (Figure 10) confirms the presence of O and Cd in the sample. The binding energy corresponding to the peaks O1s, Cd 3d5/2, and 3d3/2 obtained by XPS analysis is 530.03, 405.6, and 412.9 eV, respectively [39].

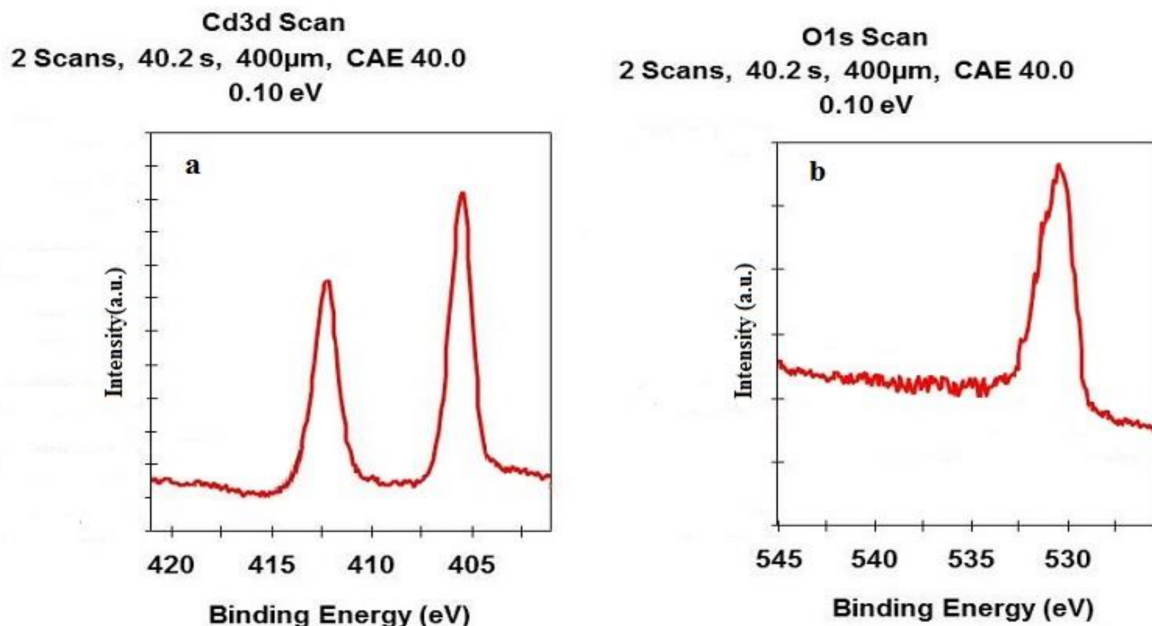


Figure 10. CdO XPS spectra (a) Cd scan and (b) O scan obtained by thermolysis of 1.

The thermal gravimetric (TG) analysis was applied to evaluate the thermal stability of the $[\text{Cd}(\text{L})(\text{NO}_2)_2]_n$ 1. TG was recorded in the temperature range of 20 and 800 °C. The diagram of 1 depicts that the compound remains stable up to 125 °C, and then decomposes up to 210 °C. The first weight loss is related to the removal of the NO_2 unit. The step 2 in the range 300 °C to 380 °C with sharp weight loss is attributed to an organic moiety of 1 with a mass loss of about 45.3%. The remained solid around 800 °C is probably CdO (Figure 11).

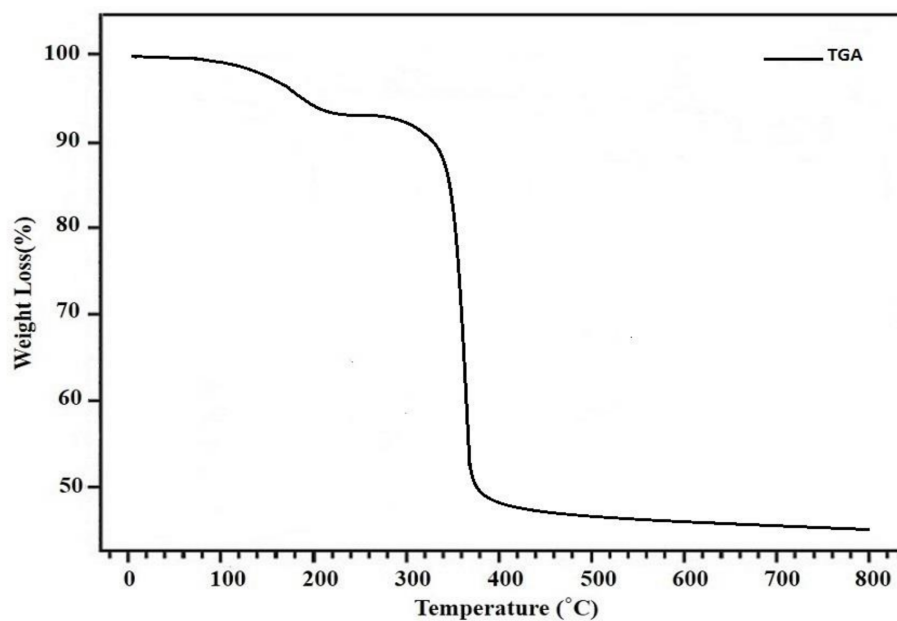


Figure 11. Thermal gravimetric (TG) thermogram of $[\text{Cd}(\text{L})(\text{NO}_2)_2]_n$.

5. Conclusions

In the present study, a novel cadmium (II) coordination polymer $[\text{Cd}(\text{L})(\text{NO}_2)_2]_n$ nanostructure was prepared in an ultrasound-assisted manner and also compared by its crystal structure. The structural characterization of new coordination compounds was carried out by elemental analysis, IR spectroscopy, SEM, and single-crystal crystallography. The complex's structure creates a 2D coordination polymer in solid-state. It was determined that the coordination number of Cd (II) ions is 7 with five oxygen of nitrite anions and two nitrogen atoms from two Schiff base ligand. With several weak interactions, the comprehensive system displayed a three-dimensional structure. Thermolysis of coordination polymer **1** results in CdO nanoparticles.

Author Contributions: Y.H.: supervision, writing—original draft; B.M.: investigation, writing—review and editing; J.D.: writing—review and editing. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: Not applicable.

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Conflicts of Interest: The authors declare no conflict of interest.

Appendix A

Crystallographic data for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication CCDC-2045446 for $[\text{Cd}(\text{L})(\text{NO}_2)_2]_n$ (**1**). Copies of the data can be obtained by request from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +441223336033, eMail: deposit@ccdc.cam.ac.uk).

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