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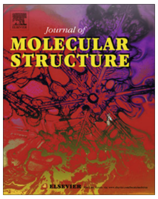

Synthesis, crystal structures, and properties of two novel cadmium(II)–organic frameworks based on asymmetric dicarboxylate and N-donor ligands

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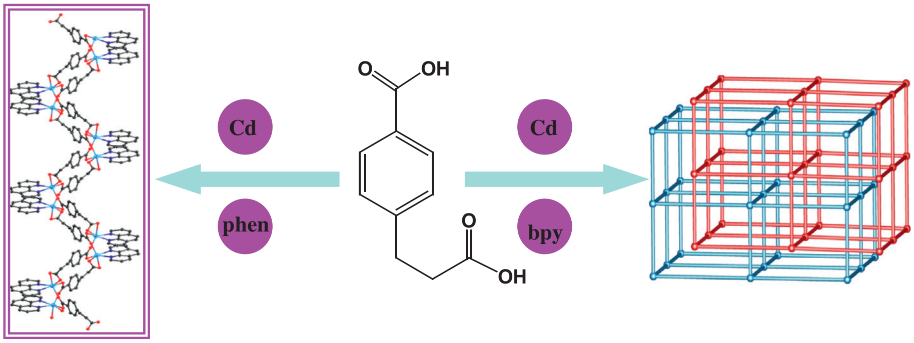
HIGHLIGHTS

- Two cadmium(II) complexes with asymmetric dicarboxylate and N-donor ligands.
- The synthesis shows that N-donor ligand plays the key role.
- **1** Displays a double zigzag chain structure.
- **2** Shows a 2-fold interpenetrated 3D α-Po structural topology.

GRAPHICAL ABSTRACT

Two novel cadmium coordination polymers have been synthesized and characterized. **1** is a double zigzag chain structure. **2** Displays a 2-fold interpenetration 3D α-Po structural topology. The thermal stabilities and luminescence properties were also studied.



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ABSTRACT

Two novel cadmium(II)–organic frameworks with asymmetric dicarboxylate and N-donor ligands, namely $[Cd(cpa)(phen)]_n$ (**1**) and $\{[Cd_2(cpa)_2(bpy)_{1.5}]\cdot 0.5H_2O\}_n$ (**2**) (H_2cpa = 3-(4-benzoic)propionic acid, phen = 1,10-phenanthroline, bpy = 4,4′-bipyridine) have been hydrothermally synthesized and characterized by elemental analyses, FT-IR spectra, single-crystal X-ray diffraction analyses, TGA, powder XRD and fluorescent measurements. **1** displays a double zigzag chain structure containing 8-number and 22-number circles. **2** Shows a 6-connected 3D polymer network based on tetranuclear cadmium cluster units. The most striking feature of **2** is that a pair of identical 3D networks are interlocked with each other to the form a 2-fold interpenetrated 3D α-Po structural topology. The diverse structures of two complexes indicate that the skeleton of N-donor ligands plays a great role in the assembly of such different frameworks. In addition, the thermal stabilities, XRD and photoluminescence properties of **1**–**2** were also studied.

Introduction

The crystal engineering of metal–organic frameworks (MOFs) has grown into a highly active area that attracts significant interest because of their intriguing aesthetic structures and topological features as well as their promising applications as functional materials [1–8]. In order to build these molecular architectures with interesting compositions and topologies, judicious selection of appropriate polydentate organic ligands and metal ions could be the key factors of a successful approach [9–13]. According to

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previous literature, the construction of MOFs mainly depends on the nature of the organic ligands (spacers) and metal ions (nodes) [14–15]. Especially organic ligands play crucial roles for the designed synthesis of some interesting coordination networks, such as the donating type, the flexibility, and the geometry of the organic ligands [16–18]. Among various organic ligands, many polycarboxylate ligands are often employed as bridging ligands to construct metal–organic frameworks, due to their extension ability both in covalent bonding and in supramolecular interactions (H-bonding and aromatic stacking) [19–26]. However, in contrast to symmetrical carboxylates, the use of unsymmetrical carboxylate ligands has been reported infrequently [27–31].

3-(4-Benzoic) propionic acid (H_2cpa) is such an asymmetric dicarboxylate, having two different substituent groups attached to the benzene ring. One is carboxyl, the other is propionyloxy. So H_2cpa ligand has four potential donor atoms, which can induce rich coordination modes and many interesting structure with higher dimensions. Furthermore, they have two carboxyl groups that may be completely or partially deprotonated, and can provide hydrogen bond donors and acceptors. Meanwhile, it possesses both rigidity and flexibility, since propionyloxy can freely rotate around the C–C bonds according to the small change in the coordination environment in order to minimize steric hindrance. However, to the best of our knowledge, compound based on H_2cpa ligand have been documented very little to date [32–35]. In this regard, Wang et al. have reported a serial of divalent metal (cobalt, nickel, znic, cadmium and copper) frameworks with H_2cpa ligand. Due to semirigid carboxyphenylpropionate of this ligand, it enables the formation of microporous and interpenetrating metal–organic frameworks [33–34]. More recently, Ag(I) and Co(II) carboxyphosphonates with a 1D chain structure have also been obtained by our group [35]. On the other hand, d^{10} metal (particularly of Zn^{II} and Cd^{II}) complexes have attracted extensive interest in recent years in that they not only exhibit appealing structures but also possess photoluminescent properties [36]. But there has been only three reported polymers constructed by Cd^{II} metal and cpa ligand hitherto. Furthermore, the combination of N-donor ligands with polycarboxylate is a good choice for the construction of novel topology and networks. The introduction of N-containing auxiliary ligands, such as phen, bpy into the [M-cpa] system may lead to new structural evolution and fine-tuning the structural motif of these metal–organic hybrid compounds. In the meanwhile, the bulky aromatic rings on phen help to direct the spatial arrangement of the bridging groups and form extensive $\pi\cdots\pi$ interactions between coordination layers. However, it is accompanied by even more uncertain factors. Thus, the prediction of mixed-ligand architectures is a challenging scientific endeavor. So we have recently engaged in the research of synthesizing the novel compounds with H_2cpa and N-donor mixed ligands. Fortunately, we have now isolated two novel compounds, $[Cd(cpa)(phen)]_n$ (**1**) and $\{[Cd_2(cpa)_2(bpy)_{1.5}]\cdot 0.5H_2O\}_n$ (**2**). In this paper, we report their synthesis, characterization, crystal structure, and photoluminescence.

Experimental section

Materials and methods

All chemicals and reagents were used as received from commercial sources without further purification. All reactions were carried out under hydrothermal conditions. Elemental analyses (C, H, N) were determined with a Elementar Vario EL III elemental analyzer; IR spectra were recorded as KBr pellets on a Bruker EQUINOX55 spectrophotometer in the 4000–400 cm^{-1} region. Fluorescence spectra were performed on a Hitachi F-4500 fluorescence spectrophotometer at room temperature. Thermogravimetric analyses (TGA) were performed in a nitrogen atmosphere with a heating rate of 10 °C min^{-1} with a NETZSCHSTA 449C thermogravimetric analyzer. The X-ray powder diffraction pattern was recorded with a Rigaku D/Max 3III diffractometer.

Synthesis

*Synthesis of [Cd(cpa)(phen)]n (**1**)*

A mixture of $Cd(NO_3)_2\cdot 4H_2O$ (0.0308 g, 0.1 mmol), H_2cpa (0.0194 g, 0.1 mmol) and phen (0.0198 g, 0.1 mmol) in molar ratio 1:1:1 and water (10 mL) was stirred for 30 min in air, then sealed in a 25 mL Telfon-lined stainless steel container, which was heated to 160 °C for 96 h. After cooling to room temperature at a rate of 5 °C h^{-1}, the colorless block crystals were obtained in ca. 65% yield (based on Cd). Anal. (%) calcd for $C_{22}H_{13}N_2O_4Cd$: C, 54.85; H, 2.72; N, 5.81; O, 13.28%. Found: C, 54.88; H, 2.74; N, 5.77; O, 13.24. IR (KBr cm^{-1}): 3419 m, 3057 w, 2930 w, 1589 s, 1541 s, 1396 s, 1230 w, 1101 w, 848 m, 777 w, 729 m, 638 w.

*Synthesis of $\{[Cd_2(cpa)_2(bpy)_{1.5}]\cdot 0.5H_2O\}_n$ (**2**)*

An identical procedure with **1** was followed to prepare **3** except that phen was changed to bpy (0.0182 g, 0.1 mmol). The colorless block crystals were obtained in ca. 58% yield (based on Cd). Anal. (%) calcd for $C_{35}H_{29}N_3O_{8.5}Cd_2$: C, 49.31; H, 3.43; N, 4.93; O, 15.95. Found: C, 49.34; H, 3.40; N, 4.89; O, 15.94%. IR (KBr cm^{-1}): 3440 m, 3051 w, 1605 s, 1547 s, 1407 s, 1211 m, 1069 m, 1007 w, 874 w, 813 m, 627 m.

Single crystal X-ray crystallography

Intensity data were collected on a Bruker Smart APEX II CCD diffractometer with graphite-monochromated MoKα radiation (λ = 0.71073 Å) at room temperature. Empirical absorption corrections were applied using the SADABS program. The structures were solved by direct methods and refined by the full-matrix least-squares based on F^2 using SHELXTL-97 program [37]. All non-hydrogen atoms were refined anisotropically and the hydrogen atoms of organic ligands were generated geometrically. Crystal data and structural refinement parameters for **1** and **2** are

Table 1
Crystal data and structure refinement for complexes **1** and **2**.

Complex	1	2
Empirical formula	$C_{22}H_{13}CdN_2O_4$	$C_{35}H_{29}Cd_2N_3O_{8.5}$
Formula weight	481.74	852.41
Crystal system	Monoclinic	Monoclinic
Space group	C2/c	P21/c
a (Å)	25.811(2)	11.7616(8)
b (Å)	10.3107(9)	20.4431(14)
c (Å)	18.5708(15)	17.2742(8)
α (°)	90.00	90.00
β (°)	130.6910(10)	127.810(3)
γ (°)	90.00	90.00
V (Å$^{3)}$	3747.4(5)	3281.4(4)
Z	8	4
D calc Mg m^3	1.708	2.076
μ (mm^{-1})	1.198	1.355
F (000)	1912	1696
Crystal size (mm)	0.24 × 0.15 × 0.18	0.21 × 0.13 × 0.15
θ Range (°)	2.08–26.41	2.41–28.40
λ (MoKα) Å	0.71073	0.71073
Reflections collected	3824	7971
Unique reflections	3038	7272
Parameters	245	418
S on F^2	1.047	1.062
R_1, wR_2[a] [$I > 2\sigma(I)$]	0.0393, 0.0973	0.0482, 0.1170
R_1, wR_2[a] (all data)	0.0506, 0.1041	0.0524, 0.1196
$\Delta\rho$ max and min (e $Å^{-3}$)	0.604 and −0.479	1.925 and −1.054

[a] $R_1 = \Sigma||F_o|-|F_c||/\Sigma|F_o|$, $wR_2 = [\Sigma w(F_o^2-F_c^2)^2/\Sigma w(F_o^2)^2]^{1/2}$.

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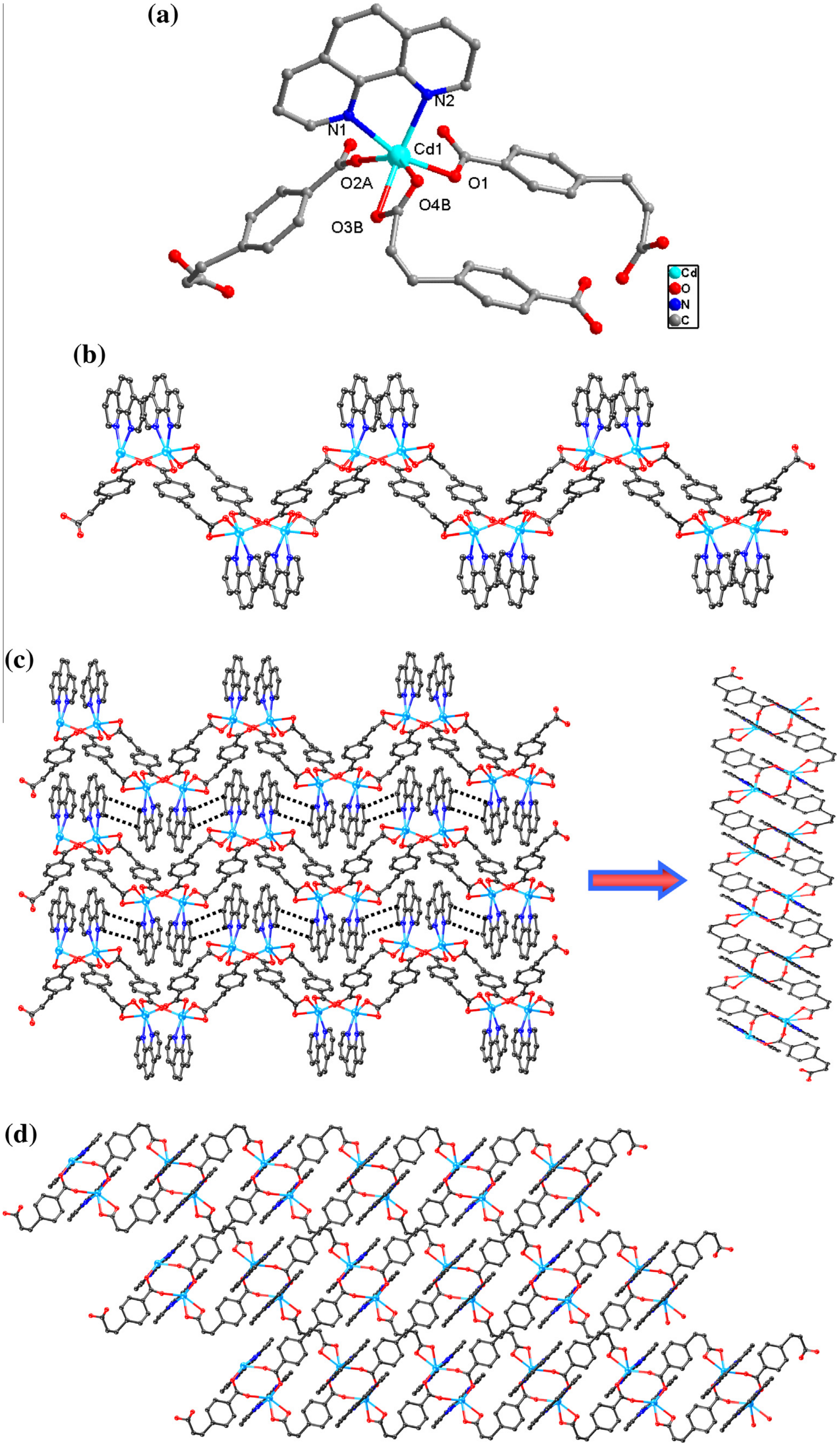

Fig. 1. (a) The coordination environment of the Cd^{II} ion in **1**, all H atoms are omitted for clarity; (b) View of the 1D double zigzag chain. (c) View of the 2D bilayer supramolecular network. (d) View of the 3D supramolecular structure by hydrogen bonds and C–H$\cdots\pi$ interaction.

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summarized in Table 1. Selected bond distances and bond angles are listed in Table S1.

Results and discussion

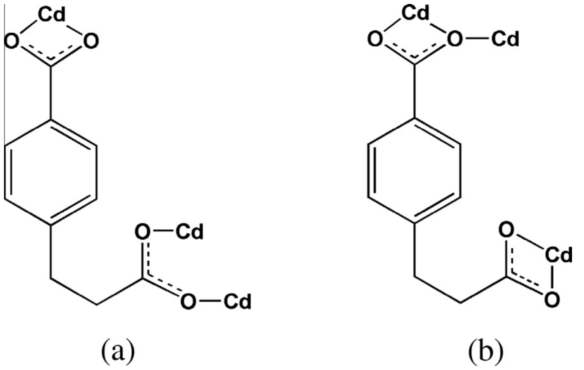
*Crystal structure of [Cd(cpa)(phen)]$_n$ (**1**)*

X-ray single-crystal diffraction analysis reveals that compound **1** crystallizes in the monoclinic, *C*2/*c* space group with an asymmetric unit that contains one crystallogra-phically unique Cd^{II} ion, one (cpa)$^{2-}$ ion and one phen ligand. As shown in Fig. 1a, the environment of the coordination sphere of the Cd^{II} ion features a distorted octahedron geometry consisting of two oxygen atoms of chelating bidentate carboxylic groups, two oxygen atoms of monodentate bridging carboxylic groups, and two nitrogen atoms deriving from one chelating phen ligand. The Cd(II) atom exhibits a distorted octahedron geometry. Four atoms (N2, O2A, O3B and O4B) comprise the equatorial plane, while another two atoms (O1, N1) occupy in the axial position. The bond lengths of Cd–O are comparable to the published ones [38], varying between 2.208(3) and 2.366(4) Å.

Interestingly, the (cpa)$^{2-}$ ligand assume bridging bidentate/chelating bidentate fashion (Scheme 1a). One carboxylate group chelates with one Cd^{II} ion bidentately, while the other adopts a bidentate coordination mode and connect with two Cd^{II} ions. Cd1 and the symmetry formed Cd^{2+} ion are connected by two bridging bidentate carboxylic groups from two independent (cpa)$^{2-}$ ligands to generate a binuclear unit and form a {$Cd_2O_4C_2N_2$} 8-numbered circle. (based on the distances of Cd···Cd 4.008 Å). The adjacent dimeric Cd$_2$ units are linked by four carboxylate groups of four (cpa)$^{2-}$ ligands generating a double zigzag chain (Fig. 1b), thus forming a novel {$Cd_2O_4C_{16}$} 22-membered ring containing a type of pore with size of ca. 9.536 Å × 10.459 Å based on the distances of Cd1···Cd1 and C8···C8. The double zigzag chain is decorated with two phen ligands at the same side. The neighboring double zigzag chains recognize each other to generate a 2D bilayer supramolecular network via the π···π stacking interactions with edge-to-edge distances of 3.307 Å between phenyl rings of adjacent phen ligands, hydrogen bonding interaction [C19–H19···O4] = 2.623 Å] and C–H···π interaction (C15–H15···π = 2.764 Å) (Fig. 1c). Then 2D bilayer supramolecular network are found to pack on top of each other, displaying a slipped geometry stabilized through C–H···O hydrogen bonds interactions and C–H···π interaction [C4–H4···π = 2.832 Å], which is developed into 3D supramolecular structure (Fig. 1d).

*Crystal structure of {[Cd$_2$(cpa)$_2$(bpy)$_{1.5}$]·0.5H$_2$O}$_n$ (**2**)*

As compared to polymer 1, the phen ligand is replaced by bpy, and the resulting structure is a 2-fold interpenetrated 3D α-Po structural topology. The asymmetric unit of **2** has two crystallographically independent Cd^{II} ions, two (cpa)$^{2-}$ anions, one and half bpy ligands and half lattice water molecules (Fig. 2a). Cd1 center is seven-coordinated and exhibits a distorted pentagonal bipyramid environment supplied by two nitrogen atoms [Cd1–N1 = 2.336(4), Cd1–N3 = 2.326(4) Å] from two bridging bpy ligands, five oxygen atoms of three separated (cpa)$^{2-}$ ligands, in which five oxygen atoms O1, O2, O6, O5 and O1A in the equatorial plane and two nitrogen atoms N1 and N3 in the axial positions. The Cd1–O bond lengths are in the range of 2.275(4)–2.456(4) Å, in good agreement with previous studies [39]. Whereas the Cd2 center ligated by two pairs of chelating oxygen atoms from two different (cpa)$^{2-}$ ligands, one bridging oxygen atom from one carboxylate group of one (cpa)$^{2-}$ ligand and one nitrogen atom [Cd2–N2 = 2.322(4) Å] from one bridging bpy ligand to complete a distorted trigonal prism geometry. O3C, O6A and O7 form the top plane of the trigonal prism while the bottom plane is completed by O4C, O8 and N2A. The Cd2–O bond distances range from 2.260(3) to 2.410(4) Å, which are also comparable to those in the literatures [40].



Scheme 1. The coordination modes of cpa^{2-} ligand in **1** and **2**.

It worth to noting that the (cpa)$^{2-}$ ligand adopts a pentadentate chelating and bridging coordination mode; one carboxylate group chelate with one Cd^{II} ion bidentately, whereas the other carboxylate group adopts a chelate/bridge tridentate coordination mode connecting two Cd^{II} ions (Scheme 1b), which is different from those of compound **1**. To our knowledge, up to now this coordination mode has never been observed in other metal–cpa complexes [32–35]. Based on these connection modes, a pair of Cd1 centers and two Cd2 centers are bridged by carboxylate groups of eight (cpa)$^{2-}$ ligands to form a tetranuclear cadmium cluster unit{$Cd_4O_{18}N_4$}. Four cadmium atoms (Cd1A, Cd2A, Cd1 and Cd2) do not locate in a line with the distances of Cd1···Cd1A (3.799 Å) and Cd1···Cd2 (4.346 Å), respectively, in which the polyhedrons of Cd1 and Cd1A centers are in an edge sharing, while the polyhedrons of Cd1 and Cd2 centers are in vertex-sharing mode (Fig. 2b). If the tetranuclear cadmium unit is considered as a secondary building unit (SBU), the adjacent tetranuclear cadmium units are interlinked by eight (cpa)$^{2-}$ ligands into 2D network running along four directions. Interestingly, the adjacent Cd1, Cd2 centers are connected by four carboxylate groups of two (cpa)$^{2-}$ ligands along two different alternating directions to form a novel {$Cd_4O_6C_{16}$} 26-membered ring containing a type of pore (I) with size of ca.10.911 Å × 12.103 Å based on the distances of Cd2···Cd2 and O6···O6. While the adjacent Cd2 centers are connected by four carboxylate groups of two (cpa)$^{2-}$ ligands along two different alternating directions to form a novel {$Cd_2O_4C_{16}$} 22-membered ring. Then the 2D layer is further interconnected by bpy ligands to form a 3D framework (Fig. 2c). Further topological analysis suggests the α-Po framework for it, if we define the tetranuclear Cd$_4$ unit as a node. More remarkably, due to the flexible conformation of cpa^{2-} ligands, the {$Cd_2O_4C_{16}$} 22-membered ring pass through {$Cd_4O_6C_{16}$} 26-membered ring from different networks to interlocked with each other, thus directly leading to the formation of a 2-fold interpenetrated 3D architecture (Fig. 2d).

*Thermal properties and XRPD measurement of complexes **1–2***

To identify the thermal stabilities of these complexes, the TGA measurements were carried out (Fig. S1). The TGA result indicates that **1** does not decompose up to 302 °C. Then followed by a continuously two-step weight loss of 76.35% from 302 °C to 610 °C (calcd. 76.26%), corresponding to the loss of (cpa)$^{2-}$ and phen ligands. The remaining weight of 27.11% is cadmium oxide that is in agreement with the calculated value of 26.65%. Compound **2** first lost its lattice water molecule below 135 °C, the weight loss

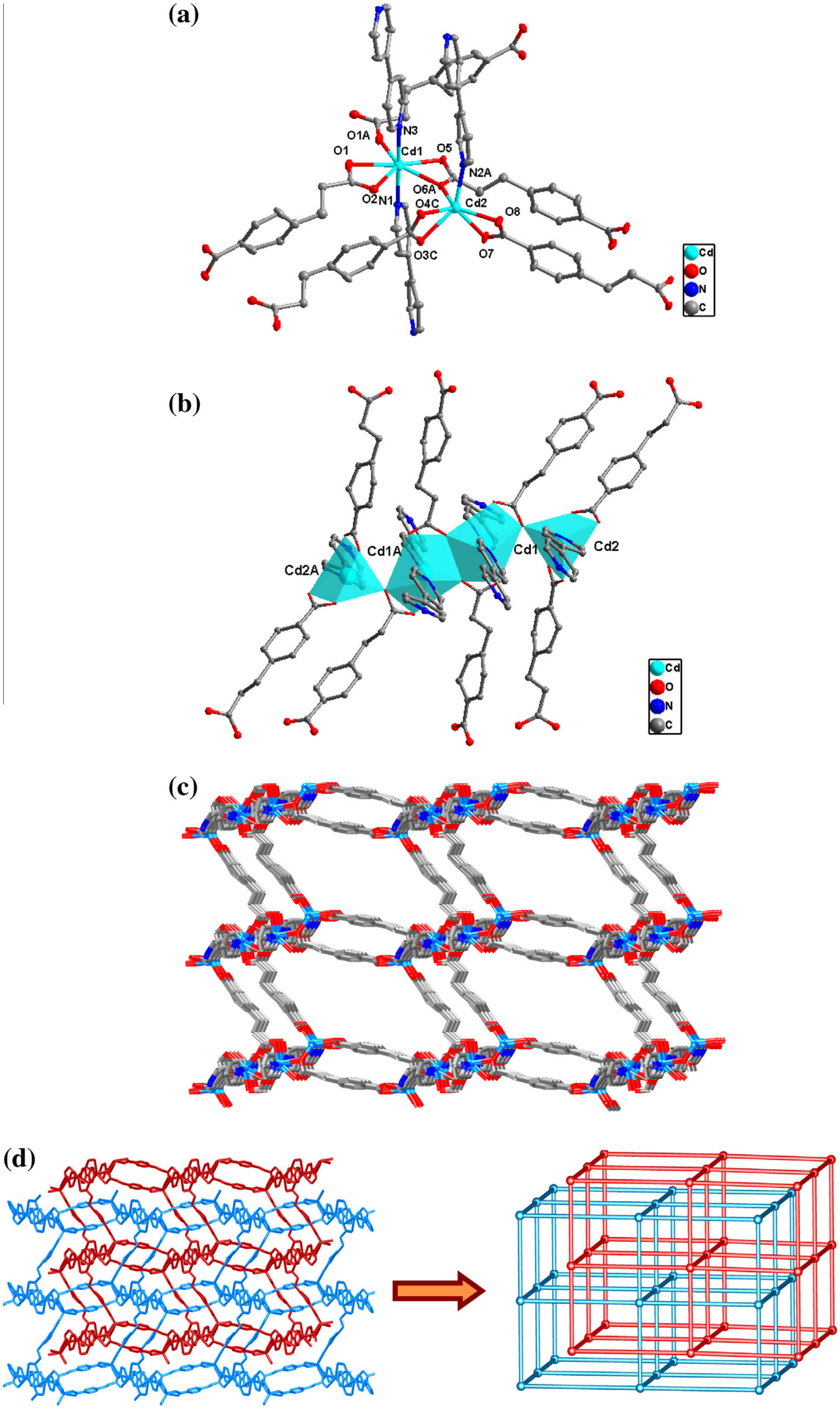


Fig. 2. (a) The coordination environment of the Cd^{II} ion in **2**, all H atoms are omitted for clarity. (b) Perspective view of the tranuclear cadmium unit along *a*-axis. (c) View of the 3D network along *a*-axis. (d) View of the 2-fold interpenetrating 3D α-Po net of 2 along *a*-axis.

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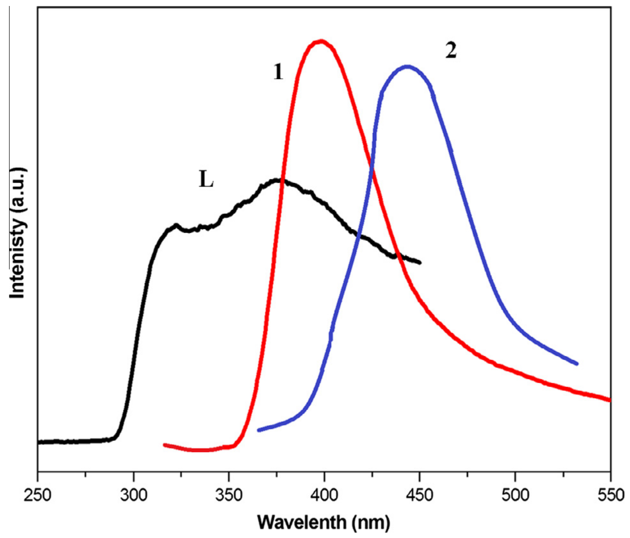

Fig. 3. The emission spectra of H_2cpa ligand, **1** and **2** in the solid state at room temperature.

found of 1.5% was consistent with that calculated (1.1%). Then followed by a continuously weight loss of 72.2% from 306 to 545 °C (calcd. 72.6%), corresponding to the loss of $(cpa)^{2-}$ and bpy ligands. TGA of **2** shows that the 3D structure is not decomposed and is stable below 306 °C. The remaining weight of 29.8% is the final product CdO (calcd. 30.1%).

In order to confirm the phase purity of the bulk materials, X-ray powder diffraction (XRPD) experiments were carried out on complexes **1–2**. The XRPD experimental and computer-simulated patterns of all of them are shown in Fig. S2. Although minor differences can be seen in the positions, intensities, and widths of some peaks, it can also be considered that the as synthesized materials are homogeneous.

*Luminescent properties of complexes **1**–**2***

Taking into account the excellent luminescent properties of d^{10} metal complexes, the luminescence of **1**–**2** and free ligand were investigated (Fig. 3). The ligand exhibits two emission bands at 323 nm and 377 nm upon excitation at 230 nm. It was found that strong emissions occur at around 398 nm and 447 nm upon excitation under λ_{ex} = 310 nm (for **1**) and 398 nm (for **2**). The emission bands for 1–2 may be due to σ-donation from the carboxylate ligands to the Cd(II) center and are tentatively attributed to the ligand-to-metal charge transfer (LMCT) [41–43]. By comparing the emission spectra of **1**–**2** and H_2cpa ligand, we can conclude that the enhancement of luminescence in **1**–**2** may be attributed to the rigidity of **1**–**2**. This rigidity is favor of energy transfer and reduces the loss of energy through a radiationless pathway [44–47].

Conclusion

In summary, we succeeded in access and structurally characterized two novel coordination polymers by appropriately combining different auxiliary ligands with H_2cpa ligand in the presence of $Cd(NO_3)_2 \cdot 4H_2O$ through hydrothermal method. Complex **1** shows a 1D double zigzag chain structure containing 8-number and 22-number circles. **2** displays a 2-fold 3D + 3D parallel interpenetration framework contains tetranuclear cadmium cluster units as secondary building blocks. The successful isolation of the two complexes indicates that it is promising to build up unusual architectures through the control of auxiliary ligands. Complexes **1–2** show photoluminescence at room temperature.

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.molstruc.2014.05.028.

Crystallographic data for the structural analyses have been deposited with the Cambridge Crystallographic Data Centre, CCDC no. 958163 (**1**) and 958164 (**2**). Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: (internat.) +44 1223 336 033; e-mail: deposit@ccdc.cam.ac.uk). Supplementary data associated with this article can be found, in the online version, at the website.

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