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Two- and three-dimensional photoluminescent Cd(II)-carboxylate coordination frameworks bridged by benzenepentacarboxylate and N-donor ligands

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Reactions of $Cd(OAc)_2 \cdot H_2O$, benzenepentacarboxylic acid (H_5bpc) , 2,2'-bpy/4,4'-bpy, and Et_3N yield two new coordination polymers $[Cd_5(bpc)_2(2,2'-bpy)_4(H_2O)_4]$ (1) and $[Cd_5(bpc)_2(4,4'-bpy)_2(H_2O)_4] \cdot 3H_2O$ (2). Complex 1 is a 2-D structure based on six-connected Cd-carboxylate layers. Adjacent layers are linked by π - π interactions and hydrogen bonds to generate a layered supramolecular network. Complex 2 is a 3-D coordination framework. The bpc ligands adopting μ_7 -bridging mode connect Cd(II) ions to form a 3-D open framework with elliptic channels, in which the coordinated 4,4'-bpy ligands fill to support the whole framework. Complex 2 exhibits strong photoluminescence at room temperature.

Keywords: Cd(II); Coordination polymer; Benzenepentacarboxylate; Hydrothermal synthesis; Photoluminescent property

1. Introduction

Crystal engineering of coordination polymers provides a solid foundation to understand how molecules can be organized and how functions can be achieved [1–3]. Thus, much effort has been directed toward designing structure because their intriguing properties and potential applications are strongly dependent on their structures [4–6]. In rational design of metal-organic coordination polymers, certain factors are always taken into account, coordination nature of the metal ion, the functionality, flexibility, and symmetry of the organic ligands, and the template effect of structure-directing agents [7–10]. Aromatic carboxylates, such as 1,2-benzenedicarboxylate [11, 12], 1,4-benzenedicarboxylate [13, 14], 1,2,3-benzenetricarboxylate [15, 16], 1,3,5-benzenetricarboxylate [17–19], 1,2,4,5-benzenetetracarboxylate [20, 21], 1,2,3,4-benzenetetracarboxylate [22], and 1,2,3,4,5,6-benzene-hexacarboxylate [23–25] have been extensively employed in construction of coordination polymers.

By investigation and comparison of coordination polymers from various carboxylic acid ligands, we hope to delineate the influence of geometry and topology of coordination frameworks on photoluminescence, and prepare and design materials with excellent photoactivity and stability. 1,2,3,4,5-Benzenepentacarboxylic acid (H₅bpc) has been proved

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by us and others to act as excellent building blocks with charge and multiconnecting ability in the construction of functional coordination polymers for photoluminescent or magnetic properties [26, 27]. Learning from our previous study on cyclohexane-polycar-boxylate coordination polymers [28–31] and biphenyl-tetracarboxylate Cd(II) complexes [32–34], the relatively rigid structure of H_5 bpc is more useful to produce coordination polymers with active photoluminescent properties when reacted with d^{10} metals. We report here two new metal-organic coordination polymers, 2-D six-connected layer $[Cd_5(bpc)_2(2,2'-bpy)_4(H_2O)_4]$ (1) and 3-D framework $[Cd_5(bpc)_2(4,4'-bpy)_2(H_2O)_4]$ 3H₂O (2).

2. Experimental

2.1. Materials and physical measurements

Reagents and solvents were commercially available and used as received. C, H, and N microanalyses were carried out with an Elementar Vario-EL CHNS elemental analyzer. FTIR spectra were recorded from KBr pellets from $4000-400\,\mathrm{cm}^{-1}$ on a Bio-Rad FTS-7 spectrometer. X-ray powder diffraction (XRPD) intensities for **2** were measured at 293 K on a Rigaku D/max-IIIA diffratometer (Cu-K α , λ =1.54056 Å). The crushed poly-crystal-line powder sample was prepared by crushing the crystals and scanned from 5–60° with a step of 0.1°/s. Calculated patterns of **2** were generated with PowderCell. The emission/excitation spectra were measured on an Edinburgh FLS-920 spectrophotometer equipped with a continuous Xe-900 Xenon lamp.

2.2. Hydrothermal synthesis

- **2.2.1.** [Cd₅(bpc)₂(2,2'-bpy)₄(H₂O)₄] (1). A solution of Et₃N (0.255 g, 2.50 mM) in H₂O (5.0 mL) and a mixture of 1,2,3,4,5-benzenepentacarboxylic acid (H₅bpc) (0.077 g, 0.25 mM) and 2,2'-dipyridine (2,2'-bpy) (0.156 g, 1.00 mM) in H₂O (5.0 mL) were added to an aqueous solution (5.0 mL) of Cd(OAc)₂· H₂O (0.266 g, 1.00 mM) and stirred. The resultant solution was heated in a stainless steel reactor with a Teflon liner at 180 °C for 130 h. After 14 h cooling to room temperature, some colorless flake-like crystals of 1 (yield *ca*. 6% based on Cd) were obtained, isolated by filtration, and washed with water. IR (KBr, cm⁻¹): 3425s, 1621vs, 1433s, 1358s, 1348s, 1250s, 1153 m, 1121w, 1025w, 920w, 770s, 736 m, 665 m, 615w, 550 m, 494w. Elemental Anal. Calcd for $C_{62}H_{42}N_8O_{24}Cd_5$ (%): C, 40.36; N, 6.07; H, 2.29. Found (%): C, 40.45; N, 5.58; H, 2.39.
- **2.2.2.** $[Cd_5(bpc)_2(4,4'-bpy)_2(H_2O)_4]3H_2O$ (2). A solution of Et₃N (0.255 g, 2.50 mM) in H₂O (5.0 mL) and a mixture of H₅bpc (0.154 g, 0.50 mM) and 4,4'-dipyridine (4,4'-bpy) (0.078 g, 0.50 mM) in H₂O (5.0 mL) were added to an aqueous solution (5.0 mL) of Cd $(OAc)_2$ · H₂O (0.266 g, 1.00 mM) and stirred. The resultant solution was heated in a stainless steel reactor with a Teflon liner at 150 °C for 140 h. After 14 h cooling to room temperature, colorless brick-like crystals of **2** (yield *ca.* 80% based on Cd) were obtained, isolated by filtration, and washed with water. IR (KBr, cm⁻¹): 3418s, 1600vs, 1420s, 1362s, 1323 m, 1221w, 1071 m, 1008w, 948w, 899 m, 811s, 732s, 634s, 605 m, 570 m,

495 m, 448w. Elemental analysis Calcd for $C_{42}H_{32}N_4O_{27}Cd_5$ (%): C, 31.79; N, 3.53; H, 2.03. Found (%): C, 31.62; N, 3.62; H, 2.09.

2.3. X-ray single-crystal structure determination

Crystallographic data for 1 and 2 were collected on a Bruker Smart Apex CCD diffractometer with Mo K α (λ =0.71073 Å) radiation at 293(2) and 150(2) K, respectively. Absorption corrections were applied by using multi-scan program SADABS [35]. The structure was solved with direct methods and refined with full-matrix least-squares with the SHELXTL program package [36]. Anisotropic thermal parameters were applied to all nonhydrogen atoms. Organic hydrogens were generated geometrically (C–H 0.96 Å); water hydrogens were located from difference maps and refined with isotropic temperature factors. Crystal data, as well as details of data collection and refinements are summarized in table 1. Crystallographic data for the structures reported in this article have been deposited in the Cambridge Crystallographic Data Center with CCDC reference number 720489 for 1 and 720490 for 2.

3. Results and discussion

3.1. The crystal structure of 1

X-ray crystallography shows that **1** contains three crystallographically unique Cd(II), one of which lies on a special position, one unique fully deprotonated bpc, two 2,2'-bpy, and two coordinated water molecules (figure 1(a)). Cd(1) lies on a general position and has six-coordinate distorted octahedral geometry, coordinating to four carboxylate oxygens

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Table 1.	Crysian	ogrannic	data and	refinement	parameters	tor I	ana z
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Complex	1	2	
Empirical formula	C ₆₂ H ₄₂ N ₈ O ₂₄ Cd ₅	C ₄₂ H ₃₂ N ₄ O ₂₇ Cd ₅	
Formula weight	1845.04	1586.72	
Crystal system	Monoclinic	Monoclinic	
Space group	$P2_1/n$	$P2_1/n$	
a (Å)	12.9114(14)	13.686(6)	
$b(\mathring{A})$	15.8277(16)	25.852(10)	
c(A)	14.5521(15)	14.458(6)	
β (°)	95.549(2)	109.946(5)	
Volume (Å ³)	2959.9(5)	4808(3)	
Z	2	4	
Calculated density (g m ⁻³)	2.070	2.192	
Absorption coefficient (mm ⁻¹)	1.863	2.276	
F(000)	1804	3072	
Crystal size (mm)	$0.19 \times 0.06 \times 0.04$	$0.12 \times 0.09 \times 0.07$	
Reflections collected	10,185	31,853	
Independent reflections	$5463 (R_{\text{int}} = 0.0384)$	9243 $(R_{\text{int}} = 0.0694)$	
Data/restraints/parameters	5463/6/460	9243/42/703	
Goodness-of-fit on F^2	1.001	1.048	
Final R indices $[I > 2\sigma(I)]$	$R_1^a = 0.0492, wR_2^b = 0.1120$	$R_1^a = 0.0408, wR_2^b = 0.0956$	
R indices (all data)	$R_1^a = 0.0685$, $wR_2^b = 0.1212$	$R_1^a = 0.0548, \ wR_2^b = 0.1029$	

 $^{{}^{}a}R_{1} = \sum ||F_{o}| - |F_{c}||/\sum |F_{o}|; {}^{b}wR_{2} = [\sum w(F_{o}{}^{2} - F_{c}{}^{2})^{2}/\sum w(F_{o}{}^{2})^{2}]^{1/2}.$

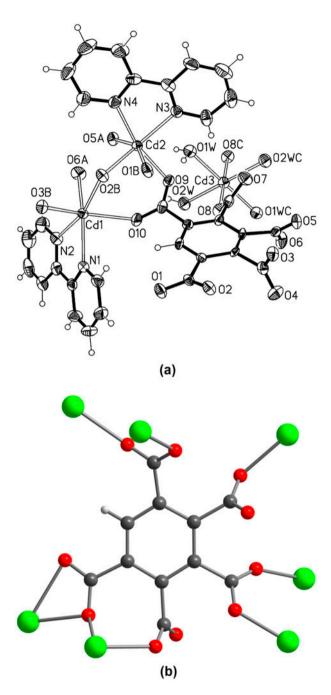


Figure 1. (a) ORTEP drawing of coordination environments of Cd with thermal ellipsoids at 50% probability and (b) the coordination mode of the bpc in 1.

from three different bpc (Cd-O=2.233(5)-2.383(5) Å) and two pyridyl nitrogens (Cd-N=2.331(6) and 2.336(6) Å). Similar to Cd(1), Cd(2) also lies on a general position

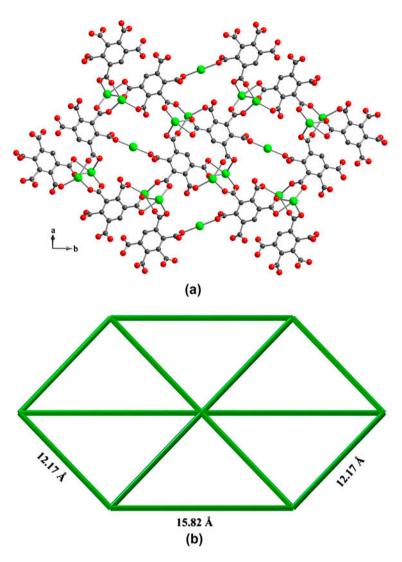


Figure 2. (a) View of 2-D Cd-bpc layer and (b) the six-connected topology in 1.

and has six-coordinate distorted octahedral geometry, coordinating to four carboxylate oxygens from three different bpc (Cd–O=2.186(5)–2.320(6) Å, and a weak coordination interaction Cd–O=2.558(5) Å) and two pyridyl nitrogens (Cd–N=2.294(6) and 2.320(6) Å). Not coordinated to 2,2'-bpy, Cd(3) lies on a center of symmetry and has six-coordinate octahedral geometry, coordinating to two carboxylate oxygens from two different bpc (Cd–O=2.285(5) Å) and four waters (Cd–O=2.323(5) and 2.393(6) Å). The carboxylates of bpc have diverse coordination modes, including monodentate, bridging and μ_3 - η^1 : η^2 , to link seven Cd ions (figure 1(b)). Two adjacent bpc link six other Cd/Cd₂-units to form a Cd₁₀(bpc)₂ second building unit, which is connected to generate a 2-D Cd-bpc layer by coordination interactions (figure 2(a)). The topology of the individual layer in 1 can be simplified by considering just the Cd₁₀(bpc)₂ units as the nodes and Cd–O interactions as

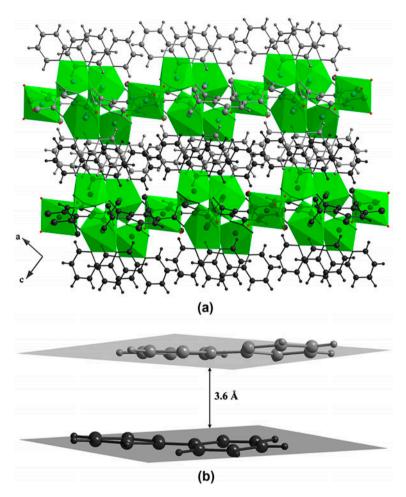


Figure 3. (a) View of stacking layers viewed along the b-axis and (b) the π - π stacking interaction between 2,2'-bpy in 1.

the links, resulting in a six-connected topology, which can be symbolized as $3^6.4^6.5^3$ (figure 2(b)) [37, 38]. The distances between nodes are 12.17 and 15.82 Å, respectively. The chelated 2,2'-bpy ligands occupy coordination sites of Cd to prevent the layers extending to a 3-D framework (figure 3(a)). Adjacent layers are linked by weak π - π interactions (3.6 Å) of 2,2'-bpy ligands (figure 3(b)) and hydrogen bonds between water molcules and carboxylates (O1w···O3ⁱ = 2.820 Å, O1w-H1wa···O3ⁱ = 161.2°; O1w···O9 = 2.847 Å, O1w-H1wb···O9 = 176.8°; O2w···O4ⁱ = 2.856 Å, O2w-H2wb···O4ⁱ = 165.0°; i: x - 1/2, -y - 5/2, z - 1/2) to form a 3-D supramolecular structure.

3.2. The crystal structure of 2

With the bridging 4,4'-bpy, the structure of **2** is a 3-D framework based on bpc ligands. X-ray crystallography shows that there are five crystallographically unique Cd(II) ions, all of which lie on general positions, two fully deprotonated bpc, two 4,4'-bpy, two coordinated,

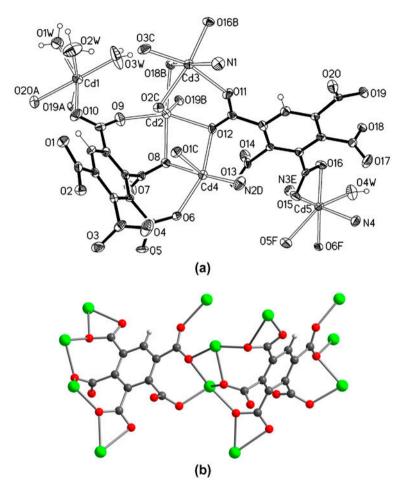


Figure 4. (a) ORTEP drawing of coordination environments of Cd with thermal ellipsoids at 50% probability and (b) the coordination modes of bpc in 2.

and two lattice water molecules (figure 4(a)). Cd(1) has six-coordinate distorted octahedral geometry, coordinating to three carboxylate oxygens from two different bpc ligands (Cd–O=2.261(4)–2.429(3) Å) and three waters (Cd–O=2.231(4)–2.390(4) Å). Cd(2) also has six-coordinate distorted octahedral geometry, coordinating to six carboxylate oxygens from three different bpc (Cd–O=2.234(3)–2.345(3) Å). Cd(3) and Cd(4) have similar six-coordinate distorted octahedral geometry, coordinating to five carboxylate oxygens (Cd–O=2.214(3)–2.438(4) Å) and one pyridyl nitrogen (Cd–N=2.334(5) and 2.339(5) Å), respectively. Cd(5) has seven-coordinate distorted pentagonal bipyramidal geometry, coordinating to four carboxylate oxygens from two different bpc (Cd–O=2.380(4)–2.488(4) Å, and a weak coordination interaction Cd–O=2.512(4) Å), two pyridyl nitrogens (Cd–N=2.256(4) and 2.271(4) Å), and one water molecule (Cd–O=2.425(4) Å). There are two crystallographically distinct bpc ligands in one unit cell, both of which adopt μ_7 -bridging through μ_2 - η^1 : η^1 , μ_2 - η^0 : η^2 and μ_3 - η^1 : η^2 carboxylates (figure 4(b)). With the bridging of μ_2 -O, the five unique Cd ions are linked to form "Y"-shaped Cd₅-units which are further

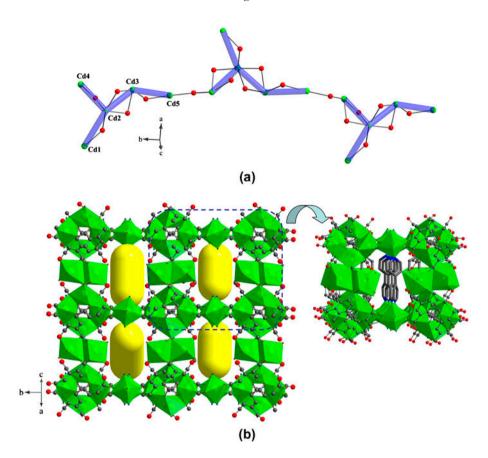


Figure 5. (a) View of 1-D Cd-O chain composed of "Y"-shaped Cd₅-unit and bridged by μ_2 -O of carboxylate and (b) view of the 3-D framework and the elliptic Cd-bpc constructed channels filled with 4,4'-bpy in 2.

connected to a 1-D waved chain along the b-direction (figure 5(a)). The bpc ligands further connect Cd-O chains to form a 3-D framework with elliptic channels (figure 5(b)). The 4,4'bpy ligands fill in the channels through coordination to Cd to support the framework, compared to the network constructed by cyclohexanehexacarboxylate and 4,4'-bpy [30]. Lattice located in the channels through rich hydrogen waters also water and carboxylate $(O1w \cdot \cdot \cdot O8^i = 2.919 \text{ Å}, O1w \cdot H1wa \cdot \cdot \cdot O8^i = 170.25^\circ;$ $O1w \cdot \cdot \cdot O14^{i} = 2.834 \text{ Å}, \quad O1w \cdot H1wb \cdot \cdot \cdot O14^{i} = 169.88^{\circ}; \quad O2w \cdot \cdot \cdot O7w^{ii} = 2.811 \text{ Å}, \quad O2w \cdot \cdot \cdot O7w^{ii} = 2.811 \text{ Å}$ $H2wa \cdots O7w^{ii} = 150.34^{\circ}; O2w \cdots O4^{iii} = 2.819 \text{ Å}, O2w - H2wb \cdots O4^{iii} = 179.45^{\circ}; O3w \cdots$ $O7^{i} = 2.890 \text{ Å}, O3w-H3wa...O7^{i} = 161.61^{\circ}; O3w...O17^{vi} = 2.874 \text{ Å}, O3w-H3wb...$ $O17^{iv} = 161.52^{\circ}; O5w \cdot \cdot \cdot O15 = 2.807 \text{ Å}, O5w \cdot \cdot \cdot O15 = 164.57^{\circ}; O6w \cdot \cdot \cdot O17^{iv} = 164.57^{\circ};$ 2.830 Å, $O6w-H6wb \cdot \cdot \cdot O17^{iv} = 160.48^{\circ}$; $O7w \cdot \cdot \cdot O6w = 2.898$ Å, $O7w-H7wb \cdot \cdot \cdot \cdot O6w = 2.898$ Å, $O7w-H7wb \cdot \cdot \cdot O6w = 2.898$ Å 176.16°; i: -x+1, -y, -z; ii: x-1/2, -y+1/2, z-1/2; iii: -x, -y, -z; iv: -x+1, -y, -z+1).

3.3. Photoluminescent property of 2

Inorganic-organic coordination complexes, especially with d¹⁰ closed shell metal centers, have been investigated for photoluminescent properties with potential applications as

luminescent materials [39, 40]. The photoluminescence of **2** was studied in the solid state at room temperature and measurement of the XRPD patterns confirms the purity of the complex (Supplementary material). The emission of free H₅bpc at 363 nm is attributed to $\pi^* \rightarrow n$ or $\pi^* \rightarrow \pi$ transition, similar to those reported for free benzenepolycarboxylic acids [22, 26]. Compared with emission of the free ligands, the emission of **2** was observed at 446 nm upon excitation at 345 nm with a red shift (Supplementary material). Replacement of the proton by Cd(II) ions may decrease the $\pi^* \rightarrow n$ or $\pi^* \rightarrow \pi$ gap of the carboxylate, resulting in a red shift of the emission peaks [41]. Therefore, the red-shifted emission can be attributed to decrease in HOMO-LUMO gaps of the complex by coordination interactions of bpc \rightarrow Cd(II) and interligand contacts. Properties of the excited state are also related to the local coordination geometry and intermolecular interactions such as hydrogen-bond interactions.

4. Conclusion

Two Cd(II) coordination polymers have been synthesized and characterized using benzene-pentacarboxylic acid (H₅bpc) in the presence of 2,2'-bpy/4,4'-bpy as auxiliary ligands by hydrothermal reactions. The difference of hydrothermal condition and auxiliary ligands results in two distinct structures bridged by bpc ligands. Complex 1 is a supramolecular network based on 2-D Cd-bpc layers with a six-connected topology, while 2 is a 3-D coordination framework with elliptic channels filled with coordinated 4,4'-bpy. Compared with the cyclohexane-polycarboxylate and biphenyl-tetracarboxylate, H₅bpc with relatively rigid geometry can be an excellent candidate for constructing photoluminescent coordination polymers with potential applications as solid materials.

Supplementary material

Simulated & experimental XRPD data, the excitation and emission spectra of **2**. Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC No. 720489 for **1** and 720490 for **2**. Copies of this information can be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44 1223 336 033; E-mail: deposit@ccdc.cam.ac.uk).

Acknowledgments

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