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Syntheses, structures, and properties of two novel cadmium coordination polymers with 1D and 2D structures

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ABSTRACT

Two novel complexes $[Cd_2(MIP)_2(BDC)_2]n (1) [MIP = 2-(3-methoxyphenyl)-1H-imidazo[4,5-f][1,10]phe$ nanthroline, BDC = terephthalic acid] and <math>[Cd(IPM)(NDC)]n (2) [IPM = 4-(1H-imidazo[4,5-f][1,10]phenanthrolin-2-yl)-2-methoxyphenol, NDC = naphthalene-1,4-dicarboxylic acid] have been synthesized byhydrothermal reaction and characterized by elemental analysis, IR, single-crystal X-ray diffraction andthermogravimetric analysis (TGA). Complex 1 exhibits 1D zigzag chain structure and complex 2 shows2D layer topology. The intermolecular C—H···O interactions extend the complex 1 into 2D networks,and the existing H-bonds further stabilized the complexes 1–2, which can be proved by TGA experiment.Furthermore, the solid-state fluorescence spectrum of the complex 2 was studied, as well as the ligandIPM. The complex 2 exhibits intense broad emission at 540 nm at room temperature, which is red-shiftedby 45 nm relative to that of free ligand IPM.

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1. Introduction

Recently, rational design and synthesis of novel metal-organic coordination polymers have received more and more intense interest and attentions, because of not only their intriguing structural motifs, but also their potential for use as catalysis, porosity, sensors, magnetism, luminescence, nonlinear optics, chirality, etc. [1–6]. Selection of appropriate multidentate ligands to coordinate metal ions is a key strategy for building metal-organic frameworks (MOFs) [7–9]. Recently, multidentate ligands such as poly-carboxylate and N-heterocyclic ligands are widely used in the rational design to obtain anticipant structures and desired properties [10-12]. Among poly-carboxylate ligands, the best studied are dicarboxylates, tricarboxylates, biphenyldicarboxylate, and oxalate (OX^{2-}) [13-15]. As for the N-heterocyclic ligands, the chelate ligands 2,2'-bipyridine, 4,4'-bipyridine, 1,10-phenanthroline, and their substituted derivatives have played an important role in the construction of coordination polymers [16,17]. The metal ion Cd(II) has d¹⁰ configuration and often adopts similar structure with zinc(II) complexes. Although Cd(II) ion has been shown to enhance the activity in several metalloenzymes, the biological relevance of Cd(II) is relatively unexplored [18-20]. 1D, 2D, or 3D Cd(II) coordination complexes attracted intense attention for their potential applications in catalysis, luminescent materials, NLO materials, and others [21]. In order to obtain novel cadmium(II) functional

complexes and investigate their structure-function relationships, we synthesized two novel N-heterocyclic ligands: 2-(3-methoxyphenyl)-1H-imidazo[4,5-f][1,10]phenanthroline (MIP) and 4-(1Himidazo[4,5-f][1,10]phenanthrolin-2-yl)-2-methoxyphenol (IPM) in view of their following characteristics: (a) they are excellent ligands with two nitrogen atoms, which are similar with 2,2'-bipyridvl-like bidentate chelating molecules: (b) they possess rigidity of coordination to metal atoms. The structures of the ligands MIP and IPM are shown in Scheme 1. Following such a mixed ligand strategy, in this study, we use BDC and NDC (BDC = terephthalic acid, NDC = naphthalene-1,4-dicarboxylic acid) for the second ligands to synthesis two new coordination polymers: [Cd₂(MIP)₂(BDC)₂]n (1) and [Cd(IPM)(NDC)]n (2). Compared with the similar cadmium(II) functional complexes, our complexes 1 and 2 display more stable properties [22,23]. Herein, we report the syntheses, crystal structures, thermogravimetric analysis (TGA), as well as photoluminescence properties of coordination polymer 2 and the ligand IPM.

2. Experimental section

2.1. Materials and physical measurements

The ligands MIP and IPM were prepared according to the description in the literature procedures [24]. All the other chemicals from commercial sources were commercially available, and used without further purification. The FT-IR spectrum was measured with KBr pellets in the range of 4000–400 cm⁻¹ on a Perkin–Elmer 240C





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Scheme 1. The structures of ligands MIP and IPM.

Table 1

Crystal data and details of structure refinement parameters for 1 and 2.

Complex	1	2
Empirical formula	$C_{56}H_{37}Cd_2N_8O_{10}$	C ₃₂ H ₂₀ CdN ₄ O ₆
Formula weight	1206.74	668.92
Crystal system	Triclinic	Monoclinic
Space group	P-1	P21/n
<i>a</i> (nm)	1.1453(3)	1.2555(1)
<i>b</i> (nm)	1.3717(4)	1.4915(1)
<i>c</i> (nm)	1.5911(5)	1.3596(1)
β (°)	94.572(6)	96.231(2)
Volume (nm ³)	2.3892(1)	2.5313(4)
Ζ	2	4
Density (Mg/m ³) (calculated)	1.677	1.755
Absorption coefficient (mm ⁻¹)	0.963	0.922
F(0 0 0)	1210	1344
Crystal size (mm ³)	$0.430 \times 0.370 \times 0.300$	$0.250\times0.189\times0.183$
Theta range (°)	1.55-25.08	2.03-26.01
Reflections collected	22,702	13,505
Unique reflections [R _{int}]	8223 [0.0257]	4959 [0.0296]
Goodness-of-fit on F ²	1.139	0.844
Final R indices $[I > 2\sigma(I)]$	R1 = 0.0976	R1 = 0.0328,
	wR2 = 0.1991	wR2 = 0.1024
R indices (all data)	R1 = 0.1360	R1 = 0.0452
	wR2 = 0.2264	wR2 = 0.1156
Largest difference peak and	2087, -754	1160, -1123
hole (e Å ⁻³)		



Fig. 1. The molecular structure of complex 1 (hydrogen atoms were omitted).

spectrometer. TGA was performed using a Perkin–Elmer TG-7 analyzer at the rate of 10 °C/min rise of temperature in nitrogen atmosphere. Crystal structures were determined on a Bruker SMART APEX II CCD X-ray diffractometer. Carbon, hydrogen and nitrogen elemental analyses were performed with a PE-2400 elemental analyzer. Fluorescence spectra were recorded on a Perkin–Elmer LS 55 luminescence spectrometer.

2.2. Syntheses

[Cd₂(MIP)₂(BDC)₂]n (1): A mixture of MIP (0.099 g, 0.3 mmol), Cd(NO₃)₂·4H₂O (0.098 g, 0.3 mmol), BDC (0.100 g, 0.6 mmol) in distilled H₂O (18 mL) was heated to 170 °C for 72 h in a 30-mL Teflonlined stainless vessel, and afterwards cooled to room temperature at a rate of 5 °C/h. The yellow crystals of complex **1** were obtained in 60% yield based on Cd. $C_{56}H_{37}Cd_2N_8O_{10}$: calcd. C 55.74, H 3.09, N 9.29%; found: C 55.69, H 3.11, N 9.35%. IR (KBr, cm⁻¹): 3139(m), 1559(vs), 1384(vs), 1226(s), 1050(s), 843(s), 520(m), 419(m).

[Cd(IPM)(NDC)]n (**2**): A mixture of IPM (0.100 g, 0.3 mmol), $Cd(NO_3)_2$ ·4H₂O (0.098 g, 0.3 mmol), NDC (0.130 g, 0.6 mmol) in distilled H₂O (18 mL) was heated to 170 °C for 3 days in a 30-mL Teflon-lined stainless vessel. The reaction was cooled to room temperature at a rate of 5 °C/h, and then small yellow crystals of complex **2** were collected in 80% yield based on Cd. $C_{32}H_{20}CdN_4O_6$: calcd. C 57.46, H 3.01, N 8.38%; found: C 57.38, H 2.95, N 8.42%. IR (KBr, cm⁻¹): 3193(s), 1563(vs), 1524(vs), 1484(s), 1403(s), 1352(vs), 1191(s), 855(s), 833(s), 784(m), 638(m), 561(m).

2.3. X-ray crystallography

Single-crystal X-ray diffraction data for complexes **1** and **2** were collected at 293(2) K with a Bruker SMART APEX II CCD diffractometer equipped with a graphite-monochromatized Mo K α radiation ($\lambda = 0.71073$ Å) at 293(2) K in the range of $1.55 \le \theta \le 25.08^{\circ}$ for **1** and $2.03 \le \theta \le 26.01^{\circ}$ for **2**. Absorption corrections were applied using multi-scan technique and all the structures were solved by direct methods and refined by full-matrix least-squares based on F^2 using the programs SHELXS-97 [25] and SHELXTL-97 [26]. Non-hydrogen atoms were refined with anisotropic temperature parameters and all hydrogen atoms were refined isotropically. Experimental details for crystallographic data and structure refinement parameters for complexes **1** and **2** are listed in Table 1.

3. Results and discussion

3.1. Structural analysis of complex 1

The molecular structure is shown in Fig. 1. The 1D zigzag chain structure is shown in Fig. 2. Double-chain structure linked by N–H \cdots O hydrogen bonds is suggested in Fig. 3, and two types of rings in the double-chain structure is shown in Fig. 4. The 2D layer structure is suggested in Fig. 5. Selected bond lengths and bond angles are given in Table 2.

Single-crystal X-ray structural analysis reveals that the asymmetric unit of complex 1 contains two Cd(II) atoms, two MIP ligands and two BDC ligands (Fig. 1). The Cd(II) atom shows a hexa-coordinated arrangement, which arises from four carboxylic oxygen atoms [Cd–O = 2.246(8)–2.560(8) Å] from two distinct chelating BDC ligands, and two nitrogen atoms donors [Cd-N = 2.274(1)-2.297(1) Å] from one chelating MIP ligand, forming a distorted octahedral geometry. The distances of Cd–O/N bond lengths are all consistent with corresponding bond lengths found in the literature [27,21,28,29]. One BDC ligand coordinate to two cadmium ions through four carboxylic oxygen atoms in a bis-chelating fashion, which give rise to a 1D zigzag chain structure. The N-heterocyclic ligands MIP are attached to both sides of this zigzag chain regularly, and the ligands MIP on the same sides are parallel nearly with the angular separation of the zigzag chain are 109.048° and 109.122°. The distances of Cd...Cd are 10.9936 Å and 11.2225 Å (Fig. 2). The asymmetric unit of complex **1** has two BDC ligands.



Fig. 2. 1D zigzag chain structure of complex 1 (hydrogen atoms were omitted).



Fig. 3. Double-chain structure of complex 1 linked by N-H···O hydrogen bonds (dotted lines represent hydrogen bonds).



Fig. 4. Two types of rings in the double-chain of complex 1 linked by N-H...O hydrogen bonds (dotted lines represent hydrogen bonds).

The BDC(1) contains O3, O4, O5, O6 with the Cd—O bond lengths are from 2.249 Å to 2.334 Å, suggests that the coordination abilities of coordinated oxygen atoms are nearly parallel. The BDC(2) contains O1, O2, O7, O8 with the Cd—O bond lengths are from 2.246 Å to 2.560 Å, reveals that the coordination abilities of coordinated oxygen atoms are different to some extent. The differences of coordinated ability for the oxygen atoms result in the distortion of

BDC ligands, which may be the result for the difference of the $Cd\cdots Cd$ distances.

There are two kinds of H-bonds interactions in complex 1: $N-H\cdots O$ interactions and $C-H\cdots O$ interactions. The most interesting aspect of the structure in **1** concerns the intermolecular $N-H\cdots O$ interactions, which help in the construction of the double chains (Fig. 3). Every pair of molecules linked by intermolecular



Fig. 5. 2D layer structure of complex 1 linked by C-H···O hydrogen bonds (dotted lines represent hydrogen bonds).

N—H···O hydrogen bonding [30] [H(4A)···O(8) = 1.986 Å, N(4)···O(8) = 2.844 Å and N(4)—H(4A)···O(8) = 175.29°] with the N atom from ligand IPM and O atom from the dicarboxylate BDC ligand construct two types of rings: 34-membered ring and 30membered ring (Fig. 4). The 34-membered ring contains four Cd atoms with the diagonal distances are 6.873 Å and 19.031 Å, and the 30-membered ring contains two Cd atoms with the diagonal distance is 12.432 Å. These two types of rings arrange alternately in the double-chain structure. Moreover, as suggested in Fig. 5, the existence of C—H···O hydrogen bonds interactions [H(9A)···O(5) = 2.58 Å, C(9)···O(5) = 3.372 Å and C(9)—H(9A) ···O(5) = 143°; H(47A)···O(1) = 2.48 Å, C(47)···O(1) = 3.390 Å and

 Table 2

 Selected bond lengths (Å) and bond angles (°) for complex 1

Bond	Dist.	Bond	Dist.					
Cd(1)-O(1)	2.560(8)	Cd(1)-O(3)	2.334(9)					
Cd(1)-O(2)	2.246(8)	Cd(1)-O(4)	2.249(8)					
Cd(2)-O(5)	2.318(8)	Cd(2)-O(7)	2.281(9)					
Cd(2)-O(6)	2.259(8)	Cd(2)-O(8)	2.520(8)					
Cd(1) - N(1)	2.282(10)	Cd(2)-N(5)	2.297(10)					
Cd(1)-N(2)	2.281(10)	Cd(2)—N(6)	2.274(10)					
Angle	(°)	Angle	(°)					
O(3)-Cd(1)-O(1)	106.2(3)	O(6)-Cd(2)-O(7)	95.7(4)					
O(2)-Cd(1)-O(4)	131.6(4)	O(7)-Cd(2)-O(5)	115.3(3)					
O(2)-Cd(1)-O(1)	53.2(3)	O(5)-Cd(2)-O(8)	89.9(3)					
O(4) - Cd(1) - O(1)	90.5(3)	O(6)-Cd(2)-O(8)	121.4(3)					
O(2)-Cd(1)-O(3)	99.5(4)	O(6)-Cd(2)-O(5)	56.8(3)					
O(4)-Cd(1)-O(3)	56.9(3)	O(7)-Cd(2)-O(8)	53.5(3)					
O(2)-Cd(1)-N(2)	97.2(4)	O(6)-Cd(2)-N(6)	148.5(4)					
O(4)-Cd(1)-N(2)	118.6(3)	N(6)-Cd(2)-O(7)	114.0(3)					
O(2)-Cd(1)-N(1)	88.3(3)	N(5)-Cd(2)-O(5)	141.3(3)					
O(4) - Cd(1) - N(1)	130.9(4)	O(6)-Cd(2)-N(5)	96.5(3)					
N(2)-Cd(1)-O(3)	158.9(4)	N(5)-Cd(2)-O(8)	128.8(3)					
N(1)-Cd(1)-O(3)	94.7(3)	N(6)-Cd(2)-O(8)	86.3(3)					
N(2)-Cd(1)-O(1)	94.2(3)	N(6)-Cd(2)-O(5)	113.9(3)					
N(1)-Cd(1)-O(1)	138.2(3)	O(7)-Cd(2)-N(5)	93.0(3)					
N(2)-Cd(1)-N(1)	73.0(3)	N(6)—Cd(2)—N(5)	72.9(3)					

C(47)—H(47A)···O(1) = 165°] [31] lead the 1D zigzag chain to 2D layer structure. It is noteworthy that the existence of hydrogen bonds reinforces the structural stability of compound **1**, which has been proved by TGA.

3.2. Structural analysis of complex 2

The molecular structure is shown in Fig. 6. The 2D layer structure is suggested in Fig. 7, and the simplified 2D layer structure is shown in Fig. 8. Selected bond lengths and bond angles are listed in Table 3.

As shown in Fig. 6, the asymmetric unit of complex **2** consists of one Cd(II) atom, one IPM ligand, and one NDC ligand. The Cd(II) atom is hexa-coordinated with two nitrogen atoms (N(1), N(2)) from one chelating IPM ligand and four oxygen atoms (O(1), O(2) from one chelating bidentate NDC, O(3A), O(4A) from two distinct bridging monodentate NDC ligand), forming a distorted octahedral geometry. The bond distances of Cd—O in compound **2** are from 2.254(2) Å to 2.376(2) Å, and those of Cd—N bond distances fall in the 2.281(3)–2.365(3) Å range, which are similar with the values reported [27,21,28,29].

The NDC ligands take bis-chelating and monodentate bridging coordination modes [32] to link three metal Cd(II) atoms, and this lead to the formation of 2D layer network (Fig. 7). There are two types of rings in the 2D network: (1) 8-membered ring which contains two Cd atoms with the diagonal distance is 5.0859 Å; (2) 36-membered ring which contains four Cd atoms with the diagonal distances are 10.3066 Å and 17.6226 Å.

In order to get better insight into the framework, we omit the IPM ligands to obtain the simplified 2D (4, 4) network if considering these dimeric motifs as single nodes (Fig. 8). Moreover, there are π - π interactions between the aryl ring of IPM ligand and the aryl ring of the NDC ligand in coordination polymer **2** with distance between cg(1) \rightarrow cg(2) ring centroid is 3.488 Å [33]. Cg(1): C8 \rightarrow C9 \rightarrow C10 \rightarrow N1 \rightarrow C7 \rightarrow C12, Cg(2): C28 \rightarrow C29 \rightarrow C30 \rightarrow



Fig. 6. The molecular structure of complex 2 (hydrogen atoms were omitted).



Fig. 7. The 2D layer structure of complex 2 (hydrogen atoms were omitted).

C31 \rightarrow C22 \rightarrow C23. The existence of hydrogen bond (N3–H(3A) \cdots O4, O5–H(5A) \cdots O2) and π – π interactions reinforce the structural stability of compound **2**, which can be proved by TGA experiment.

tion modes [35]. The characteristic absorption of IPM ligand appear at 1484 cm⁻¹, 855 cm⁻¹ and 784 cm⁻¹, and the absorption peaks at 638 cm⁻¹ and 561 cm⁻¹ are ascribed to v (Cd–N). The IR results are good agreement with their solid structural features from the results of their crystal structures.

3.3. IR spectra

The FT-IR spectra absorption peaks at 3139 cm^{-1} for **1**, and 3193 cm^{-1} for **2** correspond to the C–H stretching mode for the aromatic rings. In complex **1**, there are two peaks at 1559 cm^{-1} and 1384 cm^{-1} which correspond to the antisymmetric and symmetric stretching of carboxyl. The $\Delta v (v_{as}(COO^-) - v_s(COO^-))$ is 175 cm^{-1} (smaller than 200 cm⁻¹), indicates that the carboxyls are bidentately coordinated with Cd(II) atoms [34]. The absorption peaks at 520 cm⁻¹ and 419 cm⁻¹ are ascribed to v (Cd-N). In compound **2**, the strong characteristic absorption peaks at 1563 cm⁻¹ and 1524 cm^{-1} are attributed to the antisymmetric stretching vibration of the coordinated carboxyl groups, and the peak at 1352 cm^{-1} corresponds to the symmetric stretching of carboxyl. The separations are 211 cm⁻¹ and 172 cm⁻¹, which indicates that the carboxyls groups adopt monodentate and bidentate coordinated

Table 3	
Selected bond lengths (Å) and bond angles (°) for complex 2 .	

Bond	Dist.	Bond	Dist.
Cd(1)—O(1) Cd(1)—O(3) Cd(1)—N(1)	2.295(2) 2.254(2) 2.365(3)	Cd(1)-O(2) Cd(1)-O(4) Cd(1)-N(2)	2.376(2) 2.361(2) 2.281(3)
Angle O(1)-Cd(1)-O(2) $O(1)-Cd(1)-O(4)^{#2}$ $O(4)^{#2}-Cd(1)-O(2)$ N(2)-Cd(1)-O(2) $N(2)-Cd(1)-O(4)^{#2}$ $O(3)^{#1}-Cd(1)-N(2)$ O(1)-Cd(1)-N(1) N(2)-Cd(1)-N(1)	(°) 55.34(9) 94.21(10) 87.10(9) 92.88(9) 92.40(9) 121.14(9) 100.89(10) 71.70(9)	Angle $O(3)^{\#1}$ —Cd(1)—O(1) $O(3)^{\#1}$ —Cd(1)—O(4) ^{#2} $O(3)^{\#1}$ —Cd(1)—O(2) N(2)—Cd(1)—O(1) N(1)—Cd(1)—O(2) $O(3)^{\#1}$ —Cd(1)—N(1) $O(4)^{\#2}$ —Cd(1)—N(1)	(°) 91.22(8) 89.29(8) 145.91(8) 147.04(10) 96.63(10) 96.17(9) 163.79(9)



Fig. 8. The simplified 2D layer structure of complex 2 (hydrogen atoms and IPM ligands were omitted).

3.4. Thermal properties

To examine the stability of complexes **1** and **2**, TGA curves have been obtained from crystalline samples in the flowing nitrogen atmosphere at heating rate of 10 °C/min (Fig. 9). In compound **1**, the first weight loss of 28.00% (calcd. 27.61%) between 420 °C and 460 °C is attributable to the loss of BDC ligands. The second weight loss of 54.10% (calcd. 54.08%) in the temperature range of 460–620 °C was ascribed to the release of MIP ligands. In compound **2**, the first weight loss of 31.80% from 440 to 495 °C reveals the loss of the NDC ligands (calcd. 32.32%), and the second weight loss of 47.20% from 495 to 670 °C corresponds to the loss of IPM ligands (calcd. 51.17%). The final products of compounds **1–2** maybe metal oxide CdO [21,23]. It is concluded that the frameworks of complexes **1–2** are very stable.

3.5. Photoluminescent properties

Luminescence property is very important in photochemistry and photophysics [36,37]. So in this study, we research the luminescence of complex **2**, as well as the free ligands NDC and IPM (Fig. 10). The free ligands exhibit emissions at 490 nm for NDC (excitation at 380 nm) and 495 nm for IPM (excitation at 249 nm). Complex **2** shows one broad emission band with the maximum intensity at 540 nm (green region) upon excitation at 325 nm, which is red-shifted by 45 nm relative to the emission wavelength of free ligand IPM. The red-shift suggests that the Cd(II) ions coordinate to ligand IPM, which evokes ligand-to-metal charge transfer (LMCT) [38]. Complex **2** shows more strong emission compared to the photoluminescent emission of similar Cd(II) complexes [39,40], which may be attributed to the rigidity and



Fig. 9. TGA curves of complexes 1 and 2.



Fig. 10. Luminescent spectrum of complex 2 and ligand IPM in solid state at room temperature.

higher dimension of complex **2**. The rigidity is favor of energy transfer and reduces the loss of energy through a radiationless pathway [41]. The coordination polymer **2** may be good candidate for potential photoluminescence material because it is highly thermally stable and insoluble in water and common organic solvents.

4. Conclusions

In summary, we have reported two new cadmium complexes formed by poly-carboxylate and N-heterocyclic ligands. In compound **1**, the BDC ligands function in chelating bis-bidentate coordination mode to form 1D zigzag chain structure; The NDC ligands function in bridging bidentate-chelating bidentate coordination modes, and this lead to the formation of a 2D layer network in compound 2. The differences in the structures of these two Cd(II) complexes were caused by the variations of the coordination modes in poly-carboxylates. It is worthy to note that the intermolecular hydrogen bonds of C–H···O play an important role in the supramolecular structure. Here we studied the syntheses, structures and TG analysis of compounds 1-2, and we also investigate the fluorescence properties of the coordination polymer **2**, as well as the ligand IPM and NDC. This material will give new impetus to the construction of novel functional material with potentially useful physical properties.

Supplementary material

CCDC 814202 and 815977 contain the supplementary crystallographic data for **1** and **2**, respectively. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via http://www.ccdc.cam.ac.uk/datarequest.cif.

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