# Zinc(II) and Cadmium(II) Coordination Polymers Based on 5-Hydroxylisophthalate and Semi-rigid Bis(imidazole) Ligands: Syntheses, Structural and Photoluminescent Properties

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**Abstract.** Two new coordination polymers, namely,  $[Zn(bdc)(mbix)]_n$ (1) and  $\{[Cd_2(bdc)_2(mbix)_2]\cdot H_2O\}_n$  (2)  $[H_2bdc = 5$ -hydroxylisophthalic acid, mbix = 1,3-bis(imidazol-1-yl-methyl)benzene], were obtained under hydrothermal conditions and characterized by elemental analysis, IR spectroscopy, as well as powder- and single-crystal X-ray diffraction. Compound 1 exhibits a 2D puckered layer with

# Introduction

The interest for coordination polymers (CPs) or metal-organic frameworks (MOFs) continues to grow and the researches aimed to obtain new stable compounds, potentially useful in numerous different fields as luminescence, adsorption, heterogeneous catalysis, chemical sensing, photochemical areas, and so on.<sup>[1-15]</sup> Metal-organic coordination polymers exhibit diversified structures because of the different selected metal ions, the bridging modes and configurations of organic ligands adopted, and the various reaction conditions, which render it difficult to predict the construction of molecular architectures. Among the numerous strategies for constructing coordination polymers, the employment of the self-assembly of transition metal salts with di- or multi-carboxylic acids and/ or multidentate N-donor ligands under hydro/solvo-thermal conditions has become an effective approach.[16-20] Benzene-1,3-dicarboxylic acid (isophthalic acid, H<sub>2</sub>bdc) and its derivatives with special conformations, such as with a 120° angle between two carboxylic groups, present versatile coordination modes, can yield predetermined networks and have been widely utilized to construct coordination polymers.<sup>[21-23]</sup> Compared to H<sub>2</sub>bdc, coexistent non-coordinated groups as electronwithdrawing or electron-donating groups have a profound impact on the electron density of whole ligands, and thereby different physical phenomena can be produced.<sup>[24-26]</sup>

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[a] College of Mechanical and Material Engineering Research Institute of Materials China Three Gorges University Yichang, P. R. China (4,4) topology, which is further interconnected by hydrogen bonding interactions leading to a 3D supramolecular architecture. Compound **2** features a 5-connected 3D network with **bnn** ( $4^{6}.6^{4}$ ) topology based on the dinuclear Cd<sup>II</sup> subunits. In addition, solid-state properties such as thermal stabilities and photoluminescent properties of compounds **1** and **2** were also investigated.

We were interested in the syntheses, structures, and properties of coordination polymers derived from 5-*R*-1,3-bdc (R = OH, NO<sub>2</sub>), and transition metal salts to construct coordination polymers. As a result, many interesting frameworks with diverse topologies and different physical properties could be obtained.<sup>[27,28]</sup> In continuation of our research, 5-hydroxylisophthalic acid (H<sub>2</sub>bdc) was used to assemble with Zn<sup>II</sup> or Cd<sup>II</sup> salts, incorporating the semi-rigid ligand 1,3-bis(imidazol-1-yl-methyl)benzene (mbix) in this work. Two new coordination polymers with distinct topological motifs, [Zn(bdc)(mbix)]<sub>n</sub> (1) and {[Cd<sub>2</sub>(bdc)<sub>2</sub>(mbix)<sub>2</sub>]·H<sub>2</sub>O}<sub>n</sub> (2), were obtained. Their syntheses, crystal structures, thermal stabilities, and photoluminescent properties were also investigated.

# **Experimental Section**

**General:** All the chemicals were received as reagent grade and used without any further purification. FT-IR spectra were recorded as KBr pellets with a Thermo Electron NEXUS 670 FTIR spectrometer. Elemental analyses were performed with a Perkin-Elmer 2400 Series II analyzer. Thermogravimetric (TG) curves were recorded with a NETZSCH 449C thermal analyzer with a heating rate of 10 K·min<sup>-1</sup> in air atmosphere. X-ray powder diffraction (XRPD) analyses were recorded with a Rigaku Ultima IV diffractometer (Cu- $K_{\alpha}$  radiation,  $\lambda = 1.5406$  Å). Solid-state fluorescence spectra were recorded with a FLS920 fluorescence spectrophotometer at room temperature. The simulated powder patterns were calculated using Mercury 2.0. The purity and homogeneity of the bulk products were determined by comparison of the simulated and experimental X-ray powder diffraction patterns.

Synthesis of  $[Zn(bdc)(mbix)]_n$  (1): A mixture of  $Zn(CH_3COO)_2$ · 2H<sub>2</sub>O (21.9 mg, 0.1 mmol), H<sub>2</sub>bdc (18.2 mg, 0.1 mmol), mbix (23.8 mg, 0.1 mmol), and NaOH (4.0 mg, 0.1 mmol) in H<sub>2</sub>O (8 mL) was stirred for 30 min. The reaction mixture was placed in a 23 mL Teflon-lined stainless steel autoclave and was sealed and heated at

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120 °C for 120 h. The autoclave was allowed to cool to room temperature for 48 h. Pale yellow crystals of compound **1** were obtained in 59.0% yield (based on Zn). C<sub>22</sub>H<sub>18</sub>N<sub>4</sub>O<sub>5</sub>Zn ( $M_r$  = 483.77): calcd. C 54.62; H 3.75; N 11.58%; found C 54.12; H 3.56; N 11.82%. **IR** (KBr):  $\tilde{v}$  = 3086 m, 1625 m, 1578 s, 1338 s, 1313 s, 1248 s, 1110 m, 1054 m, 795 m, 778 s, 753 s, 740 m, 728 m, 658 s cm<sup>-1</sup>.

Synthesis of {[Cd<sub>2</sub>(bdc)<sub>2</sub>(mbix)<sub>2</sub>]·H<sub>2</sub>O}<sub>*n*</sub> (2): The same synthetic method as that of 1 was used except that  $Zn(CH_3COO)_2 \cdot 2H_2O$  (21.9 mg, 0.1 mmol) was replaced by Cd(CH<sub>3</sub>COO)<sub>2</sub>·2H<sub>2</sub>O (26.7 mg, 0.1 mmol). Pale yellow crystals of compound 2 were obtained in 55.7% yield (based on Cd). C<sub>44</sub>H<sub>38</sub>N<sub>8</sub>O<sub>11</sub>Cd<sub>2</sub> ( $M_r = 1079.62$ ): calcd. C 48.95; H 3.55; N 10.38%; found C 48.63; H 3.23; N 10.09%. **IR** (KBr):  $\tilde{v} = 3516$  s, 3116 s, 2900 w, 2342 w, 1571 s, 1542 m, 1424 m, 1367 m, 781 m, 718 m cm<sup>-1</sup>.

**X-ray Crystallographic Studies:** Single crystal X-ray diffraction analyses of complexes **1** and **2** were carried out with a Rigaku XtaLAB mini diffractometer with graphite monochromated Mo- $K_{\alpha}$  radiation ( $\lambda = 0.71073$ ). The collected data were reduced using the program CrystalClear<sup>[29]</sup> and an empirical absorption correction was applied. The structure was solved by direct methods and refined based on  $F^2$  by the full-matrix least-squares methods using SHELXTL.<sup>[30,31]</sup> All nonhydrogen atoms were refined anisotropically. The position of hydrogen atoms attached to water in **2** were located in a difference Fourier map and refined isotropically with a distance restraint of O–H = 0.85(1) Å and with  $U_{iso}$ (H) = 1.5 $U_{eq}$ (O). The crystallographic data for complexes **1** and **2** are given in Table 1. Selected bond lengths and angles for **1** and **2** are listed in Table S1 (Supporting Information).

 Table 1. Crystallographic data for complexes 1 and 2.

	1	2
Formula	C <sub>22</sub> H <sub>18</sub> N <sub>4</sub> O <sub>5</sub> Zn	C44H38N8O11Cd2
Formula weight	483.77	1079.62
Crystal system	monoclinic	monoclinic
Space group	$P2_1/n$	$P2_1/n$
a /Å	10.8861(2)	10.250(4)
b /Å	16.587(2)	34.619(12)
c /Å	12.105(2)	11.942(4)
a /°	90	90
β /°	112.746(7)	100.138(5)
γ /°	90	90
V/Å <sup>3</sup>	2015.8(5)	4172(3)
Ζ	4	4
$D_{\rm c}$ /g·cm <sup>-3</sup>	1.594	1.719
<i>F</i> (000)	992	2168
Data/restraints/parameters	4622/0/290	9596/3/594
R <sub>int</sub>	0.1261	0.1131
GOF	1.004	1.009
$R_1^{\rm a} [I > 2\sigma(I))$	0.0430	0.0478
$wR_2^{a} [I > 2\sigma(I)]$	0.1145	0.1127
$R_1$ (all data)	0.0491	0.0592
$wR_2$ (all data)	0.1207	0.1206
$\frac{\Delta \rho_{\rm max, min}  / e \cdot {\rm \AA}^{-3}}{}$	0.610 / -0.659	0.638 / -1.365

a)  $R_1 = \Sigma ||F_0|| - |F_c|/\Sigma |F_0|$ . b)  $wR_2 = \{\Sigma [w(F_0^2 - F_c^2)^2]/\Sigma [wF_0^2]^2\}^{1/2}$ .

Crystallographic data (excluding structure factors) for the structures in this paper have been deposited with the Cambridge Crystallographic Data Centre, CCDC, 12 Union Road, Cambridge CB21EZ, UK. Copies of the data can be obtained free of charge on quoting the depository numbers CCDC-956283 and CCDC-956284 for 1 and 2 (Fax: +44-1223-336-033; E-Mail: deposit@ccdc.cam.ac.uk, http:// www.ccdc.cam.ac.uk).

**Supporting Information** (see footnote on the first page of this article): Two X-ray crystallographic files (CIF), selected bond lengths and angles, simulated and experimental X-ray powder diffraction (XRPD) patterns, and TGA curves.

## **Results and Discussion**

#### Structural Description of $[Zn(bdc)(mbix)]_n$ (1)

Single crystal structure analysis shows that complex 1 crystallizes in the monoclinic space group  $P2_1/n$ . The asymmetric unit of 1 contains one Zn<sup>II</sup> ion, one bdc<sup>2-</sup> ligand, and one mbix ligand (Figure 1a). The zinc(II) ion is tetracoordinated by two carboxylate oxygen atoms from two  $bdc^{2-}$  ligands [Zn1-O1 = 1.968(2), Zn1–O3A = 1.978(2) (Å), A: x-1/2, -y+1/2, z+1/2] and two nitrogen atoms from two mbix ligands [Zn1-N1 =2.017(7), Zn1-N4B = 2.030(1) Å, B: -x+1/2, y+1/2, -z-1/2] in a distorted tetrahedral coordination arrangement. The two carboxylate groups of  $bdc^{2-}$  ligand all exhibit  $\mu_1$ -n<sup>1</sup>:n<sup>0</sup> monodentate mode. The adjacent Zn<sup>II</sup> ions are bridged by µ<sub>2</sub>-bridging bdc<sup>2-</sup> ligands to form a 1D [Zn(bdc)], chain. Such 1D chains are further interlinked by trans conformational mbix ligands with a dihedral angle between imidazole and phenyl of 75.36(9) and 65.19(6)° to form a 2D puckered (4,4) layer (Figure 1b) with grid dimensions  $9.6 \times 13.9 \text{ Å}^2$  (based on the separation of the metal ions). The 2D layers are further linked by hydrogen bonding between the hydroxyl oxygen atom and the uncoordinated carboxylate oxygen atom (O5-H5···O2<sup>#1</sup> 2.659(3) Å, #1: x+1/2, -y+1/2, z+1/2), resulting in a 3D supramolecular network (Figure 2a, b).



**Figure 1.** (a) Coordination environment of the  $Zn^{II}$  ion in **1** (symmetry codes: A: 1/2-x, 1/2+y, -1/2-z; B: -1/2+x, 1/2-y, 1/2+z). (b) View of the 2D puckered layer with (4,4) topology of **1**.



Figure 2. (a) 3D hydrogen-bonded supramolecular network of 1. (b) The hydrogen bonds between the adjacent layers.

#### Structural Description of $\{[Cd_2(bdc)_2(mbix)_2] \cdot H_2O\}_n$ (2)

Single-crystal X-ray diffraction analysis reveals that 2 also crystallizes in the monoclinic space group of  $P2_1/n$  and the asymmetric unit of 1 contains two Cd<sup>II</sup> ions, two bdc<sup>2-</sup> ligand, two mbix ligands, and one lattice water molecule (Figure 3a). The structure contains a dinuclear Cd<sup>II</sup> unit built through two bridging carboxylates. The Cd1 ion is hexacoordinate and shows a distorted octahedral arrangement formed by four carboxylate oxygen atoms from three bdc<sup>2-</sup> ligands in the equatorial plane and two nitrogen atoms from two mbix ligands in axial positions. The Cd2 ion exhibits a distorted trigonal bipyramidal arrangement with three carboxylate oxygen atoms from three bdc<sup>2-</sup> ligands and two nitrogen atoms from two mbix ligands. The bdc<sup>2-</sup> ligands feature two coordination modes: one acts as a  $\mu_3$ -bridging mode with two carboxylate in  $\mu_1$ - $\eta^1$ : $\eta^1$  and  $\mu_2$ - $\eta^1$ : $\eta^1$  modes; the other acts also as a  $\mu_3$ bridging mode with two carboxylate in  $\mu_1$ - $\eta^1$ : $\eta^0$  and  $\mu_2$ - $\eta^1$ : $\eta^1$ modes. The adjacent dinuclear Cd<sup>II</sup> ions are bridged by bdc<sup>2-</sup> ligands to form a 1D  $[Cd_2(bdc)_2]_n$  ribbon chain (Figure 3b). The infinite chain comprises 8- and 16-membered rings built up by Cd<sup>II</sup> ions and bdc<sup>2-</sup> ligands. The chains are linked by trans-conformational mbix ligands, which adopt in different orientations, thus extending the chains into a 3D framework (Figure 4a). To better understand the structure of 2, a topological analysis is performed using the TOPOS 4.0 program.<sup>[32]</sup> Topologically, we consider the dinuclear Cd<sup>II</sup> unit as a fiveconnected node, while bdc<sup>2-</sup> and mbix ligands serve as linkers. Thus, a 5-connected 3D network with **bnn**  $(4^{6}.6^{4})$  topology is constituted (Figure 4b).



**Figure 3.** (a) Coordination environment of the Cd<sup>II</sup> ion in **2** (symmetry codes: A: -1+x, y, z; B: -1/2+x, 1/2-y, 1/2+z; C: 1-x, -y, -z). (b) View of 1D [Cd<sub>2</sub>(bdc)<sub>2</sub>]<sub>n</sub> ribbon chain.

The structural differences with the same organic ligands may mainly be attributed to different central metal atoms, re-



**Figure 4.** (a) 3D framework of **2**. (b) Schematic representation of the 5-connected  $(4^{6},6^{4})$  network.

sulting in different coordination environments and ligating tendencies. In **1**, the Zn<sup>II</sup> ion displays a distorted tetrahedral coordination arrangement and is joined by organic ligands into a 2D puckered (4,4) layer. Due to the higher coordination number, in **2**, the Cd<sup>II</sup> ions adopt six-coordinate distorted octahedral arrangement and five-coordinate trigonal bipyramidal arrangement, respectively. The organic ligands link the dinuclear Cd<sup>II</sup> subunits into a 5-connected **bnn** (4<sup>6</sup>.6<sup>4</sup>) 3D network.

#### XRPD and TG Analyses

To check the purity and homogeneity of the bulk products of compounds **1** and **2**, the as-synthesized samples were measured by X-ray powder diffraction (XRPD) at room temperature. As shown in Figures S1 and S2 (Supporting Information), the peak positions of the experimental patterns are in good agreement with the simulated patterns generated from singlecrystal diffraction data, which clearly indicate the good purity and homogeneity of the compounds.

To investigate the thermal stability of these compounds, the thermogravimetric analyses were carried out on in an air atmosphere. As shown in Figures S3 and S4 (Supporting Information), for **1**, the framework is stable up to 260 °C, and a sharp weight loss from 260 to 600 °C corresponds to the decomposition of the organic ligands, and the residual weight at ca. 20.58 % (calcd. for ZnO, 19.46%). Compound **2** shows the first mass loss of 1.97 % below 100 °C due to the release of the guest water molecules (calcd. 1.67%). The further weight loss between 170 to 500 °C is attributed to the loss of organic ligands and the residual weight at ca. 23.52 % (calcd. for CdO, 23.79%).

#### **Photoluminescent Properties**

The luminescent properties of coordination polymers containing  $d^{10}$  metal ions have attracted much interest due to their various applications in chemical sensors, photochemistry, and electroluminescent display. Therefore, in this work, solid state luminescent studies of compounds **1** and **2** were investigated in the solid state at room temperature and shown in Figure 5.

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Figure 5. Solid-state emission spectra of 1 and 2 at room temperature.

Compounds 1 and 2 exhibit two intense emission maxima at 343 and 350 nm, respectively, upon excitation at 280 nm. The emissions are neither metal-to-ligand charge transfer (MLCT) nor ligand-to-metal charge transfer (LMCT) in nature since  $Zn^{II}/Cd^{II}$  ion is difficult to oxidize or reduce due to its  $d^{10}$  configuration.<sup>[33–35]</sup> According to the reported literature, the free H<sub>2</sub>bdc and mbix show fluorescent emission bands at 365 nm and 466 nm.<sup>[36,37]</sup> Thus, they may be assigned to a mixture characteristics of intraligand ( $\pi^* \rightarrow$  n or  $\pi^* \rightarrow \pi$  transition) and ligand-to-ligand charge transition (LLCT), as reported for other  $Zn^{II}/Cd^{II}$  CPs constructed from mixed N-donor and O-donor ligands.<sup>[38,39]</sup>

### Conclusions

Two new Zn<sup>II</sup>/Cd<sup>II</sup> coordination polymers with the ligand 5hydroxylisophthalicate (bdc<sup>2–</sup>), and the semi-rigid co-ligand 1,3-bis(imidazol-1-yl-methyl)benzene (mbix) were hydrothermally synthesized under similar conditions. The structural analyses indicate that the metal ions play an important role in the assembly of final structures. Compound **1** exhibits a 2D puckered (4,4) layer, whereas **2** features a 5-connected **bnn** (4<sup>6</sup>.6<sup>4</sup>) 3D network based on the dinuclear Cd<sup>II</sup> subunits. Many more systematic studies for the design and construction of new coordination polymers with different metal ions, 5-hydroxylisophthalic ligands and semi-rigid N-containing co-ligands are underway in our group.

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