One- and two-dimensional cobalt(II) coordination polymers derived from flexible bis(benzimidazole) and aromatic carboxylate co-ligands

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Abstract Two new coordination polymers, formulated as $[Co(L1)(btec)_{0.5}]_n$ (1) and $\{[Co(L2)(bdc)] \cdot H_2O\}_n$ (2) (L1 = 1,3-bis(2-methylbenzimidazol-1-ylmethyl)benzene, H₂bdc = 1,3-benzenedicarboxylic acid, H_4 btec = 1,2,4,5-benzenetetracarboxylic acid, L2 = 1,3-bis(benzimidazol-1-ylmethyl) benzene), have been hydrothermally synthesized and characterized by physicochemical and spectroscopic methods as well as single-crystal X-ray diffraction. The cobalt atoms present different environments, with a trigonal pyramidal geometry in 1 and a distorted octahedral configuration in 2. Complex 1 shows a 2D (4,4) network linked by L1 and btec⁴⁻ anions, giving an uninodal 4-connected sql topology with a point symbol of $\{4^2 \cdot 6^2\}$, while complex 2 displays a 1D ladder-like chain structure, which is further assembled into a 3D supramolecular architecture via C–H··· π hydrogen bonding interactions. The fluorescence properties of both complexes have been investigated in the solid state.

Introduction

In recent years, the design and synthesis of metal–organic coordination polymers (MOCPs) have been the subject of extensive research, not only due to their variety of structures and interesting properties, but also their potential applications in catalysis, luminescence, gas adsorption, molecular sieves, ion exchange, corrosion inhibition, and antitumor agents [1–4]. The self-assembly of such polymers by hydrothermal reactions is heavily influenced by factors such as the selection of metal ions with different coordination geometries, the nature of the

ligands used, the metal-ligand ratio, solvent, and so on [5-7]. Moreover, the architectures of the resulting coordination networks and their supramolecular organization can also be changed by noncovalent interactions, including hydrogen bonds, $\pi \cdots \pi$, C–H $\cdots \pi$ and/or van der Waals interactions [8– 11]. Hence, systematic research on this topic is crucial in understanding how these factors affect the metal coordination framework for the rational design of crystalline materials. In recent decades, there has been increasing interest in coordination architectures constructed from flexible ligands. In particular, flexible bis(benzimidazole) ligands have been widely used as classic N-containing ligands, which can satisfy the coordination needs of the metal centers and consequently create robust and intricate networks [12-16]. Moreover, this type of ligand contains both the imidazole ring and a larger conjugated π -system, capable of participating in hydrogen bonding and $\pi - \pi$ stacking interactions. Using the mixed-ligand synthetic strategy [17-19], we selected two related flexible bis(benzimidazole) ligands (L1 and L2 only differ by a methyl group) and rigid aromatic polycarboxylate (see Scheme 1) coligands to construct two new MOCPs, namely [Co(L1) $(btec)_{0.5}]_n$ (1) and $\{[Co(L2)(bdc)] \cdot H_2O\}_n$ (2), where L1 = 1,3-bis(2-methylbenzimidazol-1-ylmethyl)benzene, H₂bdc = 1,3-benzenedicarboxylic acid, H_4 btec = 1,2,4,5-benzenetetracarboxylic acid, L2 = 1,3-bis(benzimidazol-1-ylmethyl) benzene. The thermogravimetric analyses and solid-state luminescence properties of the complexes are also reported.

Experimental

Materials and measurements

Reagents and solvents were commercially available and used without further purification. The ligands L1 and L2 were prepared by the literature method [20]. Elemental

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analyses were obtained on a Perkin-Elmer 240C automatic analyzer. IR spectra within the range of 4,000–400 cm⁻¹ were obtained on a Nicolet FTIR Avatar 360 spectrophotometer with KBr pellets. Thermogravimetric analysis (TGA) was obtained on a NETZSCH TG 209 thermal analyzer from room temperature to 800 °C under N₂ atmosphere with a heating rate of 10 °C/min. The luminescence spectra for the powdered solid samples were measured at room temperature on a Hitachi F-4500 fluorescence spectrophotometer. X-ray powder diffraction (XRPD) investigations were carried out with a Rigaku D/Max-2500 diffractometer at 40 kV, 100 mA with Cu Kα ($\lambda = 1.5406$ Å) radiation.

Synthesis of complex 1

A mixture of Co(OAc)₂·4H₂O (0.1 mmol, 25 mg), L1 (0.1 mmol, 36.7 mg), H₄btec (0.1 mmol, 25.4 mg), and H₂O (12 mL) was stirred for 1 h, and then, the pH was adjusted to 6.5 by the addition of 0.1 mol/dm³ NaOH solution. The resulting mixture was sealed in a 25-mL Teflon-lined stainless steel vessel, which was heated at 140 °C for 72 h, and then cooled to room temperature at a rate of 5 °C/h. Purple block-shape crystals of complex **1** suitable for single-crystal X-ray diffraction were obtained in 45 % yield (based on Co(OAc)₂·4H₂O). Calcd. for C₂₉H₂₃CoN₄O₄ (*Fw* = 550.46): C 63.3, H 4.2, N 10.2 %; found: C 63.0, H 4.0, N 9.9 %. FTIR (KBr pellet, cm⁻¹): 3,426 m, 1,654 m, 1,515 m, 1,403 s, 1,284 m, 1,157 m, 989 m, 855 m, 764 s, 433 m.

Synthesis of complex 2

Complex **2** was synthesized by a similar procedure to that for complex **1**, replacing L1 and H₄btec with L2 (0.1 mmol, 33.8 mg) and H₂bdc (0.1 mmol, 16.6 mg), respectively.



Scheme 1 All ligands in this paper

Pink crystals suitable for single-crystal X-ray diffraction were obtained in 53 % yield (based on Co(OAc)₂·4H₂O). Calcd. for C₃₀H₂₄CoN₄O₅ (*Fw* = 579.46): C 62.2, H 4.2, N 9.7 %; found: C 61.5, H 4.1, N 9.4 %. FTIR (KBr pellet, cm⁻¹): 3,462 w, 1,605 m, 1,542 m, 1,430 m, 1,390 s, 1,259 m, 1,190 m, 836 m, 753 s, 421 s.

X-ray crystallography

X-ray diffraction data for complexes **1** and **2** were obtained at room temperature on a Bruker Smart 1000 CCD diffractometer with graphite-monochromated Mo–Ka radiation ($\lambda = 0.71073$ Å) and $\omega - 2\theta$ scan mode at 293 K. Semi-empirical absorption corrections were applied using the SADABS program [21]. Both the structures were solved by direct methods, and all non-hydrogen atoms were refined anisotropically with the SHELXTL package using a full-matrix least-squares procedure based on F^2 values

Table 1 Crystallographic data and structure refinement for 1 and 2

Complex	1	2
Empirical formula	C ₂₉ H ₂₃ CoN ₄ O ₄	C30H24CoN4O5
Formula weight	550.44	579.46
Temperature	293(2) K	293(2) K
Wavelength/Å	0.71073	0.71073
Crystal system	Monoclinic	Orthorhombic
Space group	P21/c	Pnna
Unit cell dimensions		
a/Å	12.020(3)	17.2366(18)
b/Å	9.571(3)	12.5117(13)
c/Å	21.090(6)	11.8458(13)
β/°	92.244(4)	
$V/Å^3$	2,424.4(11)	2,554.7(5)
Ζ	4	4
$Dc/g \text{ cm}^{-3}$	1.508	1.507
<i>F</i> (000)	1,136	1,236
Crystal size/mm	$0.28\times0.22\times0.22$	$0.26 \times 0.22 \times 0.21$
θ Range/°	1.70-27.50	2.09-25.02
Index range h, k, l	-9/15, -12/12, -27/26	-15/20, -14/13, -14/12
Reflections collected	14,198	12,160
R _{int}	0.0688	0.0455
Goodness of fit on F^2	0.884	0.864
Final <i>R</i> indices $[I > 2\sigma(I)]^{[a,b]}$	$R_1 = 0.0599,$ $wR_2 = 0.1715$	$R_1 = 0.0358,$ $wR_2 = 0.1078$
R (all data) ^[a,b]	$R_1 = 0.1171,$ $wR_2 = 0.2112$	$R_1 = 0.0505,$ $wR_2 = 0.1234$
Largest diff. peak and hole	0.607, -0.492	0.230, -0.381

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

Table 2 The selected bond lengths (Å) and angles (°) for 1 and 2

Parameter	Value	Parameter	Value				
$[Co(L1)(btec)_{0.5}]_n$ (1)							
Co1–O2	1.942(3)	Co1–N1	2.052(4)				
Co1-O3	1.951(3)	Co1–N4	2.048(4)				
O2-Co1-O3	103.20(15)	O2-Co1-N1	117.28(14)				
O2-Co1-N4	110.64(14)	O3-Co1-N1	101.13(14)				
O3-Co1-N4	122.85(15)	N4-Co1-N1	102.28(15)				
N2-C24-C28	111.8(4)	N3-C17-C15	112.1(4)				
$\{[Co(L2)(bdc)] \cdot H_2O\}_n$ (2)							
Co1–N1	2.083(2)	Co1-O2	2.1228(19)				
Co1–O1	2.215(2)	01-Co1-O1C	83.97(11)				
N1–Co1–N1C	100.63(12)	O2-Co1-O1	60.35(7)				
N1-Co1-O2	101.32(8)	O2-Co1-O1C	94.98(7)				
N1C-Co1-O1	157.98(8)	O2C-Co1-O2	148.15(10)				

Symmetry transformation used to generate equivalent atoms: C = -x + 1, y, -z + 3/2

[22]. All non-hydrogen atoms were refined anisotropically, and hydrogen atoms bonded to carbon atoms were placed in geometrically calculated positions. H atoms of one water molecule for 1 were not located due to their disorder and high thermal parameters of O1W, whose occupation factor is 0.5. Crystallographic data and experimental details for structural analysis of the complexes are summarized in Table 1, and selected bond lengths and angles are listed in Table 2.

Results and discussion

IR spectra

In the IR spectra of the complexes, bands at $1,515 \text{ cm}^{-1}$ for **1** and $1,542 \text{ cm}^{-1}$ for **2** are assigned to the $v_{C=N}$ absorption of the benzimidazole rings of the ligands L1 and L2, respectively. A broadband around 3,426 and $3,462 \text{ cm}^{-1}$ for **1** and **2**, respectively, can be attributed to the stretching vibrations of lattice water molecules or the existence of hydrogen bonds. The asymmetric and symmetric stretching vibrations of the carboxyl groups are observed at 1,654 and 1,403 cm⁻¹ for complex **1**, and at 1,605 and 1,430 cm⁻¹ for complex **2**. The separations ($\Delta v[v_{as}(COO)-v_{s}(COO)]$) between these bands indicate the presence of monodentate (251 cm^{-1} for **1**) and chelating (175 cm^{-1} for **2**) carboxylate groups [23]. There is no characteristic band at around 1,700 cm⁻¹, indicating that all carboxyl groups in both complexes are completely deprotonated.



Fig. 1 a Coordination environment of the Co atom in 1, and hydrogen atoms were omitted for clarity; b 2D (4,4) supramolecular structure in 1; c The uninodal 4-connected 2D net with a point symbol of $\{4^2 \cdot 6^2\}$ topology network in 1

Structural analysis of the complexes

Single-crystal X-ray diffraction analysis reveals that complex **1** crystallizes in the monoclinic space group $P2_1/c$. The asymmetric unit contains one Co(II) center, one L1 ligand, and half a btec^{4–} ligand. As shown in Fig. 1a, each Co atom is four-coordinated by two nitrogen atoms (N1, N4) from two different L1 ligands and two oxygen atoms (O2, O3) belonging to two different btec^{4–} ligands to give a trigonal pyramidal geometry, with a τ_4 value of 0.85 [24]. The angle of O3–N1–N4 is 67.93(11)°. The bond lengths of Co–N1, Co–N4, Co–O2, and Co–O3 are 2.052(4), 2.048(4), 1.942(3), and 1.951(3) Å, respectively. The bond angles range from 101.13(14) to 122.85(15)°, which are comparable to values observed for similar cobalt(II) complexes [25].

In the structure of complex 1, the L1 ligand adopts cisconformation, with the dihedral angle between the mean planes of the two benzimidazole rings being 89.64(2)°. These L1 ligands act in a bis-monodentate mode, bridging neighboring Co atoms into infinite 1D $[CoL]_n$ zigzag chains with a distance between adjacent Co atoms of 11.290(1) Å. Furthermore, four carboxyl groups of the btec⁴⁻ ligands assume monodentate coordination modes to extend the $[CoL]_n$ chains into a 2D (4,4) layer (Fig. 1b). In order to understand the architecture of complex 1 more fully, the topological method has been used to simplify and analyze the 2D structure by TOPOS 4.0 software [26]. The L1 ligands can be simplified to be linear connectors; the cobalt center should be considered as a 4-connected node, connected by two L2 and btec⁴⁻ ligands. The btec⁴⁻ ligands linking four Co atoms should also be viewed as 4-connected nodes. The network of 1 shows a uninodal 2D 4-connected sql topology with point symbol of $\{4^4.6^2\}$ (Fig. 1c). As is shown in this figure, the grid-like layer is made of rhomboids and irregular squares with a throughligand Co-Co distance of 5.5438(6) Å for anionic btec⁴⁻ ligands, and 11.2859(5) Å for L1 ligand, providing a distorted (4,4) structure (the red nodes represent btec⁴⁻ ligands, and the blue nodes are Co atoms). In addition, the crystal packing is enhanced by two kinds of weak C-H--O hydrogen bonds (C19-H19...O4A: 3.244(6) Å, 154°; C27-H27...O4B: 3.296(6) Å, 164°, symmetry codes: A = 2-x, 2-y, -z; B = x, 5/2-y, 1/2 + z) between L1 and atom O4 of the $btec^{4-}$ ligands.

Complex 2 crystallizes in the orthorhombic space group *Pnna*. X-ray crystal structural determination reveals that complex 2 has an infinite 1D ladder-like chain structure. The asymmetric unit consists of one Co(II) center, one anionic bdc²⁻ ligand, one L2 ligand, and one lattice water molecules. The Co atom is coordinated by four oxygens (O1, O2, O1C, O2C) from two different bdc²⁻ ligands and two nitrogen atoms (N1, N1C) (symmetry code: C = -x + 1, y, -z + 3/2) from two L2 ligands in a distorted octahedral configuration (Fig. 2a). The bond lengths of Co1–O1, Co1–O2, and Co–N are 2.215(2), 2.124(2), and 2.1228(19) Å, respectively. The coordination angles range from 60.35(7) to 157.98(8)°.

In complex 2, each bdc^{2-} ligand adopts a chelating coordination mode connecting two neighboring Co atoms



Fig. 2 a Coordination environment of the Co atom in **2**, hydrogen atoms were omitted for clarity, and symmetry transformation was used to generating equivalent atoms: C = -x + 1, y, -z + 3/2; D = 2.5-x, 1-y, z; E = x, 1 + y, z; **b** 1D zigzag chain connected by Co atoms and bdc^{2–} ligands in **2**; **c** 1D ladder-like chain connected by L2 and bdc^{2–} ligands in **2**

to form a 1D $[Co_2(bdc)_2]$ zigzag chain (Fig. 2b). Each L2 ligand adopts a *cis*-conformation to link $[Co_2(bdc)_2]$ units, developing a 1D ladder-like chain (Fig. 2c). The adjacent non-bonding Co…Co distances are 9.391(9) (Co1…Co1D) and 12.512(8) Å (Co1…Co1E) (symmetry codes: D = 2.5-x, 1-y, z; E = x, 1 + y, z). In addition, these 1D chains are further extended into a 3D supramolecular architecture by five kinds of C–H… π hydrogen bonds (Table 3). The distances of H…Cg are from 2.73(3) to 2.93(3) Å, and the angles of C–H…Cg are in the range of 112(4)–155(4)°. These weak interactions play an important role in strengthening the stability in such metal–organic coordination polymers.

Table 3 The C–H··· π hydrogen bond distances (Å) and angles (°) in **2**

d(X–H)	d(H···Cg)	$d(X \cdots Cg)$	∠XHCg
C5–H5…Cg4	2.75(3)	3.509(4)	139
C8–H8A…Cg1	2.93(3)	3.422(4)	112
C8–H8A…Cg2	2.73(3)	3.627(4)	155
C10-H10Cg2	2.90(2)	3.648(4)	138
C15-H15Cg3	2.91(3)	3.612(4)	134

Symmetry transformation used to generate equivalent atoms: F = x, -0.5-y, 0.5-z, G = x, 0.5-y, 0.5-z (Cg1: N1–C1–N2–C2–C7, Cg2: C2–C3–C4–C5–C6–C7, Cg3: C9–C10–C11–C10F–C9F–C12, Cg4: C13–C14–C13G–C15G–C16–C15)

The diversity of structures obtained in this and other studies results from factors including the conformation of the ligands and the choice of anions [27]. Complexes related to 1 and 2 were reported previously, namely $[Co(L3)(btec)_{0.5}]_n$ (3), $[CoCl_2(L2)]_n(DMF)_{n/2}(CH_3OH)_{n/4}$ (4), and $[CoCl_2(L2)]_2(DMF)_2$ (5) (L3 = 1,4-bis(2-methylbenzimidazol-1-ylmethyl)benzene, DMF = N,N-dimethylformamide) [28, 29]. In all five complexes 1–5, the flexible bis(benzimidazole) ligands adopt cis- or trans-conformations to connect the Co centers, but exhibit different torsional arrangements, leading to a variety of different nonbonding Co…Co distances and distinct geometries. Table 4 compares the geometries obtained in these complexes. Complex 2 displays a 1D ladder-like chain structure, while 4 features 1D linear chain, and 5 is a dinuclear complex. The different structures can be attributed to distinct conformations of the L2 in complexes 4 and 5.

In complexes 1 and 3, the Co atom displays four-coordinated environment connecting $btec^{4-}$ and different flexible bis(benzimidazole) ligands (L1 or L3) to perform two kinds of 2D layer structures. While 1 and 3 show different configurations (trigonal pyramid for 1, tetrahedron for 3), which may result from the different conformations of the flexible bis(benzimidazole) ligands (*cis* for L1, *trans* for

Table 4 The comparison of the related complexes of complex 1 and 2



Fig. 3 TG curves of compounds 1 and 2

L3), these results reveal that the conformations of such flexible bis(benzimidazole) ligands have an important effect on the final structures of the complexes.

Thermal properties and XRPD analysis

Thermogravimetric analysis (TGA) experiments were carried out to measure the thermal stabilities of complexes 1 and 2, as shown in Fig. 3. There is only one weight-loss stage for complex 1, but a two-step weight-loss process was observed for complex 2. For complex 1, a rapid weight loss in the range of 390–565 °C corresponds to the decomposition of all organic ligand, and the remaining residue is CoO (calcd: 13.6 %; found: 14.3 %). The TGA curve of complex 2 shows that the compound begins to lose weight above 20 °C. Water is lost steadily, giving an overall loss of 2.8 % at 160 °C (calcd: 3.1 %). The weight loss continues from 351 to 502 °C, and the final residue is CoO (calcd: 12.9 %; found: 13.2 %).

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The related complexes	Coordination number	Conformation	Crystal structure	Co…Co separation (Å)	Geometry	References		
$[Co(L1)(btec)_{0.5}]_n(1)$	4	cis	2D layer	11.290(1)	Trigonal pyramidal	This work		
$\{[Co(L2)(bdc)]\cdot H_2O\}_n(2)$	6	cis	1D ladder-like chain	12.512(8)	Octahedral	This work		
$[Co(L3)(btec)_{0.5}]_n(3)$	4	trans	2D layer	12.5255(3)	Tetrahedron	[28]		
$[CoCl_2(L2)]_n(DMF)_{n/2}(CH_3OH)_{n/4}(4)$	4	trans	1D linear chain	13.1252(6)	Tetrahedral	[<mark>29</mark>]		
$[CoCl_2(L2)]_2(DMF)_2(5)$	4	cis	0D loop	10.4243(7)	Tetrahedral	[29]		

where L1 = 1,3-bis(2-methylbenzimidazol-1-ylmethyl)benzene, L2 = 1,3-bis(benzimidazol-1-ylmethyl)benzene, L3 = 1,4-bis(2-methylbenzimidazol-1-ylmethyl)benzene, btc = 1,2,4,5-benzenetetracarboxylate, bdc = 1,3-benzenedicarboxylate, DMF = N,N-dimethylformamide



Fig. 4 a X-ray powder diffraction patterns of 1. b X-ray powder diffraction patterns of 2

Complexes 1 and 2 were also characterized by X-ray powder diffraction (XRPD) to confirm the phase purity of the bulk materials. The experimental and computer-simulated XRPD patterns of the complexes are shown in Fig. 4. The XRPD patterns of both as-synthesized products closely match the simulated ones from the single-crystal data, indicating that both complexes are in a pure phase.

Photoluminescence properties

The photoluminescence properties of complexes 1 and 2, and the free ligands L1 and L2 have been recorded in the solid state at room temperature (Fig. 5). The free ligands L1 and L2 display emission maxima at 305 and 345 nm, respectively ($\lambda_{ex} = 290$ nm for L1, and $\lambda_{ex} = 280$ nm for L2), which can be assigned to $\pi \rightarrow \pi^*$ transitions [30–32]. An emission peak can be observed at about 360 nm for complex 1 and 316 nm for 2, when both complexes are excited at



Fig. 5 a Solid-state photoluminescence spectra of free L1 ligand and 1. b Solid-state photoluminescence spectra of free L2 ligand and 2

300 nm. For complex **1**, the red shift of the emission peak, by 55 nm compared to free ligand L1, probably arises from ligand-centered $\pi - \pi^*$ electronic transitions within the benzimidazole rings of the L1 ligand [33]. Compared with the free ligand L2, the emission maximum of complex **2** is blue-shifted, which may be assigned to a ligand-to-metal charge transfer (LMCT) transition.

Conclusion

In summary, two Co(II) coordination polymers have been hydrothermally synthesized and structurally characterized. The structural differences between complexes 1 and 2 probably arise from the different conformations (*cis* or *trans*) of the flexible bis(benzimidazole) ligand and the role played by the different anionic ligands in the assembly of the resulting frameworks.

Supplementary materials

CCDC 981056 and 981057 contain the supplementary crystallographic data for the complexes **1** and **2**. These data can be obtained free of charge via http://www.ccdc.cam.ac. uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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