From 2-D layer to 2-D → 3-D parallel interpenetration: syntheses, structures and luminescent properties

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1. Introduction

Coordination polymers are of interest and much effort has been invested in synthesis of coordination polymers for esthetic motifs, interesting structural topologies [1–3], and potential functionalities in energy or electron transfer, drug delivery, and even having an interesting relationship with biological systems [4–9]. Numerous 1-D chains and ladders, 2-D grids, 3-D porous motifs, interpenetrated modes, and helical staircase coordination polymers have been synthesized by judicious choice of appropriate...
metal ions and multifunctional organic ligands [10–13]. It is found that layered subunit is an increasingly ubiquitous structural element in cells and organisms [14–16].

Besides the simple 2-D layered networks, information on entanglements in 2-D species, showing a remarkable richness of types, has attracted our attention. Many reported species have interpenetration or other types of entanglements. Interpenetrated 2-D layers represent the most numerous group [17]. This kind of entanglement is generally constructed through the presence of identical 2-D motifs that are interlaced, sharing the same average plane. For increasing number of interpenetrating nets, they have been classified and elucidated in some excellent reviews [18–20]. Polycatenated and polythreaded coordination polymers formed by 2-D-layered motifs indicate that interpenetrating nets will be obtained more likely if the metal ions are linked by longer organic ligands, producing frameworks with larger cavities, accompanied by a network occupied by one or more other independent networks [21, 22].

We prepared \([\text{Co}_2(\text{HO-BDC})_2(\text{bbi})_2] \cdot \text{H}_2\text{O} \) (1) and \([\text{Zn(HO-BDC)(bbi)}] \) (2) by involving 1,1′-(1,4-butane diyl) bis(imidazole) (bbi) and 5-hydroxyisophthalic acid (OH-BDC) as starting materials. Complex 1 features a wavelike 2-D layer and 2 is an interesting 2-D → 3-D parallel interpenetrating network based on (4,4) motifs. Furthermore, the fluorescence properties of 1 and 2 have been studied at room temperature.

2. Experimental

2.1. Materials and physical measurements

Reagents were purchased commercially and used without purification. IR (KBr pellets) spectra were obtained from 4000 to 400 cm\(^{-1}\) using a Perkin-Elmer Spectrum One FT-IR spectrometer. Elemental analyses for C, H, and N were performed on a Perkin-Elmer 2400 Elemental Analyzer. Powder X-ray diffraction (PXRD) patterns were collected on a Rigaku D/Max 2000 X-ray diffractometer with graphite monochromated Cu-K\(_\alpha\) radiation (\(\lambda = 1.5418 \text{ Å}\)). Thermogravimetric analyses (TGA) were recorded on a Perkin-Elmer Thermal Analyzer at a heating rate of 10 °C min\(^{-1}\) (N\(_2\)). Luminescence spectra were performed on a Cary Eclipse spectrofluorometer (Varian) equipped with a xenon lamp and quartz carrier in the solid state at room temperature.

2.2. Synthesis of \([\text{Co}_2(\text{HO-BDC})_2(\text{bbi})_2] \cdot \text{H}_2\text{O} \) (1)

A mixture of CoCl\(_2\)·2.5H\(_2\)O (0.017 g, 0.1 mmol), OH-BDC (0.02 g, 0.1 mmol), bbi (0.02 g, 0.1 mmol), Na\(_2\)CO\(_3\) (0.011 g, 0.1 mmol), and H\(_2\)O (10 ml) were stirred. The pH of the mixture was adjusted to 6 with NaOH solution (0.05 mol/L), heated at 160 °C for 3 days under autogenous pressure, then cooled at a rate of 3 °C/h to room temperature. Purple flake crystals of 1 were separated, washed with distilled water, and dried at ambient temperature. Yield: 63%, based on Co for 1; Elemental analysis for 1: C\(_{36}\)H\(_{38}\)N\(_8\)O\(_{11}\)Co\(_2\) (876.59). Calcd %: C, 49.33; H, 4.37; N, 12.78. Found: C, 49.35; H, 4.34; N, 12.79. IR (KBr, cm\(^{-1}\)): 3424 (br), 3126 (w), 2936 (w), 1625 (m), 1563 (w), 1370 (s), 1274 (m), 1106 (m), 1035 (m), 974 (s), 897 (m), 726 (s), 655 (w).

2.3. Synthesis of \([\text{Zn(HO-BDC)(bbi)}] \) (2)

A mixture of Zn(NO\(_3\))\(_2\)·6H\(_2\)O (0.03 g, 0.1 mmol), OH-BDC (0.02 g, 0.1 mmol), bbi (0.02 g, 0.1 mmol), and H\(_2\)O (8 ml) was stirred. The pH of the mixture was adjusted to 6 with NaOH solution (0.05 mol/L). The resulting mixture was sealed in a 25-mL Teflon-lined stainless steel vessel under autogenous pressure. After heating for 3 days at 160 °C and cooling at a rate of 3 °C/h to room temperature, colorless block crystals of 2 were separated, washed with distilled water, and dried at ambient temperature. Yield: 55%, based on Zn for 2; Elemental analysis for 2: C\(_{18}\)H\(_{18}\)N\(_4\)O\(_5\)Zn (435.73). Calcd %: C, 49.62; H, 4.16; N, 12.86. Found: C, 49.64; H, 4.13; N, 12.88. IR (KBr, cm\(^{-1}\)): 3432 (br), 3130 (w), 2955 (w), 1623 (w), 1415 (s), 1241 (m), 1104 (m), 1035 (m), 974 (s), 897 (m), 726 (s), 655 (w).
2.4. X-ray crystallography

Single-crystal X-ray diffraction data for 1 and 2 were collected on a Bruker SMART APEX CCD diffractometer with Mo-Kα radiation (λ = 0.71073 Å) at 293(2) K. The absorption correction was performed empirically. The crystal structures were solved by direct methods and refined employing full-matrix least squares on F² (SHELXTL-97) [23]. All non-hydrogen atoms were refined anisotropically; the hydrogens of organic ligands were located geometrically. Due to the small crystallographic disorder for 1, O9 in HO-BDC and O1W are disordered and occupied two sites, with total occupancy of 1. The crystal data

Table 1. Crystal data and structure refinements for 1 and 2.

|          | 1                  | 2                  |
|----------|--------------------|--------------------|
| Complex  |                    |                    |
| Empirical formula | C₃₆H₃₈N₈O₁₁Co₂       | C₁₈H₁₈N₄O₅Zn       |
| Formula weight | 876.61             | 435.73             |
| T (K)     | 293(2)             | 293(2)             |
| Wavelength λ (Å) | 0.71073            | 0.71073            |
| Crystal system | P                 | P2(1)/n            |
| Space group | Triclinic          | Monoclinic         |
| a (Å)     | 9.6984(5)          | 9.5493(12)         |
| b (Å)     | 13.8962(7)         | 14.5411(17)        |
| c (Å)     | 14.5986(8)         | 13.7193(16)        |
| α (°)     | 92.856(4)          | 90                |
| β (°)     | 95.764(5)          | 91.110(2)          |
| γ (°)     | 92.509(4)          | 90                |
| V (Å³)    | 1952.73(18)        | 1904.7(4)          |
| Z         | 2                  | 4                 |
| D_calcd (Mg m⁻³) | 1.487             | 1.520             |
| Absorption coefficient (mm⁻¹) | 0.919             | 1.327             |
| F(0 0 0)  | 900                | 896               |
| R_int    | 0.0453             | 0.0192            |
| R_a, wR_b [I > 2σ(I)] | 0.0634, 0.1392 | 0.0274, 0.0670 |
| R_a, wR_b (all data) | 0.1106, 0.1597 | 0.0337, 0.0698 |

Table 2. Bond lengths (Å) and angles (°) for 1 and 2.

|          | 1                  | 2                  |
|----------|--------------------|--------------------|
| Co(1)–O(1) | 1.982(3)          | Co(2)–O(8)#1     | 1.934(4)          |
| Co(1)–N(1) | 2.015(4)          | Co(2)–O(6)       | 1.944(3)          |
| Co(1)–N(3) | 2.031(4)          | Co(2)–N(5)       | 2.026(4)          |
| Co(1)–O(4)#1 | 2.185(5)          | Co(2)–N(7)       | 2.035(4)          |
| Co(1)–O(3)#1 | 2.281(5)          | O(6)–Co(2)–N(7) | 110.07(17)        |
| O(1)–Co(1)–N(1) | 111.66(16)   | N(5)–Co(2)–N(7) | 106.54(17)        |
| O(1)–Co(1)–N(3) | 110.06(16)   | O(8)#1–Co(2)–N(7) | 98.59(18)       |
| N(1)–Co(1)–N(3) | 114.14(18)   | O(8)#1–Co(2)–O(6) | 120.51(16)      |
| O(1)–Co(1)–O(4)#1 | 84.21(16)   | O(4)#1–Co(2)–N(5) | 109.57(19)       |
| N(1)–Co(1)–O(4)#1 | 113.4(2)    | O(6)–Co(2)–N(5) | 114.49(16)       |
| N(3)–Co(1)–O(4)#1 | 119.44(19)   | N(3)–Co(1)–O(3)#1 | 90.52(17)       |
| O(1)–Co(1)–O(3)#1 | 140.72(15)   | O(4)#1–Co(1)–O(3)#1 | 56.52(16)     |
| N(1)–Co(1)–O(3)#1 | 87.70(16)    |                    |                  |
| Zn(1)–O(1) | 1.9454(13)        | Zn(1)–N(1)       | 2.0035(17)        |
| Zn(1)–O(3) | 1.9645(14)        | Zn(1)–N(3)       | 2.0096(18)        |
| O(1)–Zn(1)–O(3) | 112.57(6)   | N(1)–Zn(1)–N(3) | 109.86(7)         |
| O(1)–Zn(1)–N(1) | 116.99(7)    | O(1)–Zn(1)–N(3) | 108.81(7)         |
| O(3)–Zn(1)–N(1) | 96.75(7)     | O(3)–Zn(1)–N(3) | 111.44(7)         |

Notes: Symmetry codes for 1: #1, x + 1, y, z, #2, x − 1, y, z.

2.4. X-ray crystallography

Single-crystal X-ray diffraction data for 1 and 2 were collected on a Bruker SMART APEX CCD diffractometer with Mo-Kα radiation (λ = 0.71073 Å) at 293(2) K. The absorption correction was performed empirically. The crystal structures were solved by direct methods and refined employing full-matrix least squares on F² (SHELXTL-97) [23]. All non-hydrogen atoms were refined anisotropically; the hydrogens of organic ligands were located geometrically. Due to the small crystallographic disorder for 1, O9 in HO-BDC and O1W are disordered and occupied two sites, with total occupancy of 1. The crystal data
and structure refinements of 1 and 2 are summarized in Table 1 and selected bond lengths and angles are listed in Table 2.

3. Results and discussion

3.1. Crystal structure of [Co2(HO-BDC)2(bbi)2]·H2O (1)

X-ray crystallographic analysis indicated that 1 crystallizes in triclinic space group Pī. The structure of 1 contains two kinds of Co(II) ions. As shown in Figure 1, Co1 displays a distorted tetragonal pyramidal coordination geometry, two nitrogens from two different bbi ligands (Co(1)–N(1) = 2.015(4) Å, Co(1)–N(3) = 2.031(4) Å) and three oxygens from two HO-BDC ligands (Co(1)–O(1) = 1.982(3) Å, Co(1)–O(3)#1 = 2.281(5) Å, Co(1)–O(4)#1 = 2.185(5) Å; symmetric code: #1, 1 + x, y, z). The carboxylic groups of HO-BDC are bidentate chelate and monodentate, respectively. Co2 has a disordered tetrahedral coordination sphere, coordinated by two nitrogens from two bbi ligands (Co(2)–N(5) = 2.026(4) Å, Co(2)–N(7) = 2.035(4) Å) and two oxygens from two HO-BDC ligands (Co(2)–O(6) = 1.944(3) Å, Co(2)–O(8)#1 = 1.934(4) Å; symmetric code: #1, 1 + x, y, z). Co(II) may be oxidized to Co(III) upon coordination. To make sure of the oxidation state of cobalt centers, we did valence bond calculation, showing no Co(III) in this structure with the results Co(1) ~ 2.144, Co(2) ~ 2.221. In addition, the difference...
in bond lengths between Co(III) and Co(II) is quite large and the higher oxidation state prone to bind “softer” N-donor groups, while the lower oxidation state binds exclusively to O-donors [24, 25], yet in the structure, these phenomena are not found. Divalent cobalt centers can meet the charge balance in the structure. The carboxylic groups of HO-BDC ligands are monodentate. Two Co(II) ions with different coordination modes form the similar deeply undulating layers. Investigation of the crystal packing showed that all the 2-D layers stack layer by layer in an ABAB pattern along the c axis (Figure 2).

### 3.2. Crystal structure of [Zn(HO-BDC)(bbi)] (2)

X-ray crystallographic analysis indicated that 2 crystallizes in the monoclinic space group P2(1)/n with intriguing features. As indicated in Figure 3, the fundamental building unit of 2 consists of one Zn(II), one OH-BDC ligand, and two halves of bbi ligands. Zn(II) has a slightly distorted tetrahedral coordination geometry, coordinated by two carboxyl oxygens from two OH-BDC ligands (Zn(1)–O(1) = 1.9454(13) and Zn(1)–O(3) = 1.9645(14) Å) and two nitrogens from two bbi ligands (Zn(1)–N(1) = 2.0035(17) and Zn(1)–N(3) = 2.0096(18) Å). The Zn–O and Zn–N bond distances are all within the normal ranges [26, 27]. The OH-BDC in 2 performs as a bis(monodentate) ligand to join Zn(II) ions to form uniform chains. These chains are further propagated into a highly undulating or corrugated 2-D (4,4) layer which is common in the structure of the coordination polymer based on isophthalate and its derivatives [28] by μ₂-bridged bbi ligands (Figure 4(a) and (b)). Additional investigation for this structure indicates that rectangular voids within each layer with dimensions of 13.4 × 9.5 Å² are spacious enough to allow the formation of catenation between adjacent layers. Each sheet is penetrated by two others (one above
and the other below) which have parallel but not coincident mean planes, leading to an overall 2-D → 3-D interpenetration (Figure 4(c)). In general, stacking layer by layer is normal for connecting parallel 2-D layers. But this stacking is always without direct interaction or interpenetration not to mention dimension increase just like 1. In a few cases, the single-layer motif is highly undulating and “thick” enough to obtain parallel polycatenation networks with dimension increase via interpenetration [29]. Previous examples of 2-D → 3-D entangled structures, which have been observed for interpenetration systems in parallel fashion based on (4,4) networks, are fewer than the models of 2-D → 2-D parallel interpenetration or 2-D → 3-D inclined polycatenation [30, 31].

3.3. Comparison of the structures of 1 and 2

From the structural descriptions above, we can see that the O-donor ligands (HO-BDC) are all deprotonated. It can adopt diverse coordination modes, but two modes are found in the two frameworks. In 1, HO-BDC connects Co1 and Co2 by $\mu_2$-$\eta^1$:$\eta^2$ and $\mu_2$-$\eta^1$:$\eta^1$, respectively. For 2, it only adopts the $\mu_2$-$\eta^1$:$\eta^1$ mode; the bond lengths of Co–O are from 1.934(4) to 2.281(5) Å, while the bond lengths of Zn–O are from 1.9454(13) to 1.9645(14) Å. The N-donor bbi links two mental centers through the trans configuration in the two complexes; the bond lengths of Co–N are from 2.015(4) to 2.035(4) Å, while the bond lengths of Zn–N are from 2.0035(17) to 2.0096(18) Å. Similar undee 2-D (4,4) layers are formed based on the coordination modes of ligands and metal centers. Finally, 1 shows stacking layers, yet 2 has 2-D → 3-D interpenetration since it possesses enough space and “thick” layers. The divergence between the final structures of the two complexes can be considered from ligands coordinated with the distinct metal centers.

3.4. IR spectra of 1 and 2

IR spectra of carboxylate complexes can provide information to distinguish the coordination modes of the carboxyl groups [32, 33] (Figure S1). The broad band centered at 3424 and 3432 cm$^{-1}$ reveals the O–H characteristic stretching vibration from the not coordinated hydroxyl of HO-BDC. Complete deprotonation of the dicarboxyl ligands is indicated in the complexes for the absence of $\nu$(COOH) absorption bands of the ligands at 1700 cm$^{-1}$ in spectra. For 1, bands at 1625, 1563, and 1370 cm$^{-1}$ can be, respectively,
assigned to the antisymmetric and symmetric stretching vibrations of the deprotonated carboxylic groups; \( \Delta \nu = \nu_{as}(COO^-) - \nu_{s}(COO^-) = 255 \text{ cm}^{-1} (1625–1370 \text{ cm}^{-1}) \), and 193 \text{ m}^{-1} (1563–1370 \text{ cm}^{-1}) \) indicate that the coordination modes of the carboxyls are monodentate and bidentate. For 2, the bands at 1623 and 1415 \text{ cm}^{-1} can be, respectively, assigned to the antisymmetric and symmetric stretching vibrations of the deprotonated carboxylic groups; \( \Delta \nu = 208 \text{ cm}^{-1} \) shows the coordinate modes of the carboxyls in 2 are monodentate. The results are consistent with the coordination of the carboxylate in the compounds.

### 3.5. Photoluminescent properties

Luminescent properties of d^{10} transition metal complexes (such as Zn(II), Cd(II) and Hg(II)) have attracted interest because of their various applications in chemical sensors, photochemistry, and electroluminescent displays [34, 35]. Luminescent properties of 1 and 2 are investigated in the solid state at room temperature (Figure 5). To further analyze the nature of the emission band, the photoluminescent properties of HO-BDC and bbi have also been investigated under the same experimental conditions. Complex 2 exhibits green light with one luminescent emission band at \( \lambda_{\text{max}} = 545 \text{ nm} \) upon photoexcitation at 360 nm. HO-BDC and bbi ligands display luminescence with emission maxima at 409 and 436 nm (\( \lambda_{\text{ex}} = 360 \) and 357 nm), respectively (Figure S2 and Figure S3), which can be attributed to the \( \pi^* \rightarrow n \) or \( \pi^* \rightarrow \pi \) transitions. The emission of 2 is neither metal-to-ligand charge transfer nor ligand-to-metal transfer since Zn^{2+} is difficult to oxidize or reduce, but rather assigned to intraligand emission [36–40]. Comparing with the maximum emission peak of free HO-BDC and bbi ligands and the maximum emission peak of 2, the emission band of 2 is red-shifted. This could be assigned to intraligand luminescent emission enhanced by the coordination of the Zn^{2+} ion to the ligand. Similar red shifts have been observed before [41]. Different from 2, 1 exhibits purple emissions at 420 nm when excited at 360 nm [42].

### 3.6. Thermal analyses

To characterize the compound more fully in terms of thermal stability, the thermal behaviors of 1 and 2 were examined by TGA on polycrystalline sample (Figure S4). The TGA diagram of 1 indicates two main weight losses. The first begins at 90 °C and is completed at 120 °C. The observed weight loss of 1.96% corresponds to loss of the one crystallization water molecule (Calcd 2.10%). The second weight loss begins at 365 °C and completes at 550 °C. The observed weight loss of 82.10% corresponds to loss of bbi and HO-BDC (Calcd 83.09%). We speculate that the final products of the thermal decomposition are metal oxide with the final weight of 15.94% (Calcd 17.10%) for 1. For 2, TGA shows no weight loss up to 360 °C. The weight loss from 360 to 560 °C is due to the organic groups decomposing gradually in the range. The remaining weight (18.23%) indicates that the final product should be ZnO (Calcd 18.68%).

### 3.7. Powder X-ray diffraction

Powder X-ray diffraction (PXRD) has been used to check phase purity of these complexes in the solid state (Figure S5 and Figure S6). The peak positions of the measured PXRD patterns are in agreement with the simulated patterns, which indicate that the synthesized materials and the measured single crystals are the same compound.

### 4. Conclusion

Two new coordination polymers have been synthesized under hydrothermal conditions. Complex 1 has a 2-D wavelike net, while 2 has 2-D \( \rightarrow \) 3-D parallel interpenetration of highly undulating layer networks. The present research further proves that layered coordination nets can be efficient structural elements to construct high-dimensional nets. This entangled system of 2-D \( \rightarrow \) 3-D parallel interpenetration with
(4,4) nets draws more attention to layered organization. We also report fluorescence properties of the two compounds.

**Supplementary material**

Crystallographic data for the structural analyses have been deposited with the Cambridge Crystallographic Data Center (CCDC) Nos. 900445 and 882921 for 1 and 2, respectively. The data can be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; E-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

**Disclosure statement**

No potential conflict of interest was reported by the authors.

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