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Two novel 2-fold interpenetration frameworks constructed by 4,4'-bis(1H-imidazol-1-yl-methyl)biphenyl cobalt(II) coordination polymer and the effect of dicarboxylic acid



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ABSTRACT

To investigate the construction of interpenetrating frameworks, two novel coordination polymers $[Co(bimb)_3](NO_3)_2\cdot 2H_2O$ (1) and $[Co(bimb)(bdc)]\cdot H_2O$ (2) [bimb = 4,4'-bis(1H-imidazol-1-yl-methyl) biphenyl; $H_2bdc = 1,2$ -benzenedicarboxylic acid] were hydrothermally synthesized and characterized by elemental analysis, IR, PXRD and TGA. Complex 1 shows a 2-fold pcu interpenetrating framework. However, complex 2 exhibits a rare 2-fold uoc interpenetrating framework due to the introduction of bdc anion. The results indicate that the presence of the second ligand could tune the product structures. Photoluminescence properties of complexes 1 and 2 reveal the cobalt ion disturbed ligand emission. © 2013 Elsevier B.V. All rights reserved.

1. Introduction

Metal organic frameworks (MOFs) have gained increasing interest in their intriguing architectures and versatile potential application in gas storage, catalysis, optics, microelectronics, biology, and so on [1-8]. As an important subject in the design and synthesis of MOFs, the study of network topology is not only a powerful tool for simplifying complicated structure but also plays an instructive role in the rational design of certain functional materials with desirable properties [9–14]. Many topologically interesting MOFs have been discussed in recent years [15,16]. Among those structures, one particularly intriguing phenomenon is interpenetration, whose aesthetic appeal is presently one of the most fascinating aspects of this field [17–21]. The occurrence of interpenetrating polymeric networks, once a rarity, is now becoming increasingly common, aided by the rapid growth of network-based crystal engineering. In the past two decades, a number of interpenetrating structures have been reported, however, most of which belong to dia nets and **pcu** nets [22,23]. The length of the ligand could dominate the interpenetration structures, nevertheless, counterions, number and type of the solvated molecules, coordination geometry and

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kinetic factors are also very important for the outcome of the connectivity and topology of the coordination polymers in the crystallization process [24–25].

As a classical flexible long spacer ligand first used for coordination polymers synthesis in 2000 [25], 4,4'-bis(1H-imidazol-1-ylmethyl)biphenyl (bimb) was also found to be promising candidate for constructing high dimensional interpenetrating structures. However, up to date, few 3D networks based on bimb ligand are reported [26]. Most of the bimb complexes are metallocycles [27,28], 1D polymers [29,30] and 2D layers [31]. Recently, a number of complexes with interesting structures were obtained by the combination of bimb and other auxiliary ligands or other building blocks, generally aromatic carboxylic acids [32–38] and polyoxometalates (POMs) [39,40].

In this work, we sought to investigate the construction of high dimensional interpenetrating structures by bimb ligand. Complex **1** was hydrothermally synthesized by reacting bimb with cobalt nitrate, which shows a 2-fold pcu interpenetrating 3D framework. Further study indicates that the auxiliary ligand plays an important role in the formation of complex structure. Complex **2** was synthesized following that of **1** except that 1,2-benzenedicarboxylic acid (H₂dpc) was used as the mixed ligand. However, complex **2** exhibits a rare 2-fold uoc interpenetrating 3D framework. The structures of bimb and H₂dpc are shown in Scheme 1. In addition, their thermal stability and photoluminescence property have been investigated and described.

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Scheme 1. Structures of bimb and H₂dpc.

2. Experimental

2.1. Materials and measurements

Cobalt nitrate, ethanol, imidazole, 1,2-benzenedicarboxylic acid are from Kermel and 4,4'-bis(chloromethyl)-1,1'-biphenyl from J & K Chemicals. All chemicals purchased are of reagent grade and were used as received without further purification. Elemental analyses for C, H, and N were carried out on a Vario EL III elemental analyzer. IR spectrum was recorded on a Bruker tensor 27 FT-IR spectrophotometer in the 4000–400 cm⁻¹ region by using KBr pellets. The luminescent spectrum was taken on a Perkin Elemer Corporation Model Fluorescence Spectrometer LS 55 PL. PL spectra were performed in solid samples after the crystals were crushed and put between quartz plates. Thermogravimetric analyses were carried out with a Perkin-Elmer DTA-1700 with a heating rate of 10 °C/min under atmosphere from 25 to 800 °C. X-ray powder diffraction measurement, whose range of 2θ is from 5° to 50°, was performed on a Rigaku D/Max-IIIB X-ray diffractometer Cu Ka $(\lambda = 1.5406 \text{ Å})$ radiation (40 kV and 200 mA) and Ni filter.

2.2. Synthesis

2.2.1. Synthesis of $[Co(bimb)_3](NO_3)_2 \cdot 2H_2O(1)$

4,4'-Bis(1H-imidazol-1-yl-methyl)biphenyl (bimb) was prepared following the literature [41]. Co(NO₃)₂·6H₂O (0.12 g, 0.40 mmol), bimb (0.13 g, 0.40 mmol), H₂O (4 mL) and ethanol (4 mL) were stirred for 10 min in air, before the mixture was transferred and sealed in a 20 mL Teflon-lined stainless autoclave, which was heated at 140 °C for 3 days and then cooled to room temperature. Pink crystals were obtained in 57% yield based on Co^{II} cation after washed with deionized water and ethanol. *Anal.* Calc. for C₆₀H₅₈CoN₁₄O₈ (1162.12): C, 62.01; H, 5.03; N, 16.87. Found: C, 62.09; H, 5.12; N, 16.72%. IR (KBr pellet, cm⁻¹): 3104 (w), 1502 (s), 1436 (m), 1359 (m), 1282 (m), 1223 (m), 1110 (m), 1081 (m), 1031 (w), 905 (m), 823 (m), 744 (s), 696 (w), 664 (m), 628 (w), 490 (m).

2.2.2. Synthesis of $[Co(bimb)(bdc)] \cdot H_2O(2)$

The preparation of **2** was similar to that of **1** except that H_2 dpc (0.07 g, 0.40 mmol) was used as auxiliary ligand, and then the pH was adjust to 6–7 by 1 M NaOH solution. Brown block crystals were filtered and washed with distilled water and ethanol (32% yield based on Co^{II} cation). Elemental *Anal.* Calc. for C₂₈H₂₄N₄O₅Co (555.44): C, 60.55; H, 4.36; N, 10.09. Found: C, 60.62; H, 4.42; N, 10.01%. IR (KBr, cm⁻¹): 3435 (w), 3123 (w), 1700 (w), 1610 (m), 1581 (m), 1449 (m), 1396 (m), 1338 (w), 1281 (m), 1230 (m), 1187 (w), 1097 (s), 1005 (w), 952 (m), 830 (s), 803 (s), 754 (s), 691 (w), 626 (w), 505 (w).

Table 1

Crystal data and structure refinements for complexes 1 and 2.

	1	2
Empirical formula	C60H58CoN14O8	C28H24C0N4O5
Formula weight	1162.13	555.44
Crystal system	monoclinic	tetragonal
Space group	P2/c	I41/a
a (Å)	15.600(3)	19.904(3)
b (Å)	9.654(2)	19.904(3)
c (Å)	20.651(8)	26.180(5)
α (°)	90	90
β(°)	112.25(2)	90
γ (°)	90	90
$V(Å^3)$	2878.5(14)	10372(3)
Ζ	2	16
$D_{\text{calc}} (\text{mg m}^{-3})$	1.341	1.423
$\mu (\mathrm{mm}^{-1})$	0.366	0.708
F(000)	1214	4591
Collected/unique	26420/6494	46385/5926
R _{int}	0.0315	0.0827
Goodness-of-fit GOF on F ²	1.030	1.043
$R_{l}^{a}[I > 2\sigma(I)]; wR_{2}^{b}[I > \sigma(I)]$	0.0443; 0.1360	0.0495; 0.1044
R_{l}^{a} (all); wR_{2}^{b} (all)	0.0650; 0.1505	0.0850; 0.1167

^a $R_1 = \sum ||Fo| - |Fc|| / \sum |Fo|$.

^b $wR_2 = \{\sum [w(F_o^2 - F_c^2)^2 / \sum w(F_o^2)^2]\}^{1/2}.$

2.3. X-ray crystallography

Single-crystal X-ray diffraction data for **1** and **2** were collected on a Rigaku R-AXIS RAPID imaging plate diffractometer with graphite-monochromated Mo K α (λ = 0.71073 Å) at 291 K. Empirical absorption corrections based on equivalent reflections were applied. The structures of **1** and **2** were solved by direct methods and refined by full-matrix least-squares methods on F^2 using SHELXS-97 crystallographic software package [42]. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms of the bimb and bdc molecules were placed in calculated positions and treated as riding on their parent. The hydrogen atoms of water molecules were located in the difference Fourier map and were refined as riding on their parent O atom. The crystal parameters, data collection and refinement results for **1** and **2** are summarized in Table 1. Selected bond lengths and angles are listed in Table S1 (see the Supporting information).

3. Results and discussion

3.1. Synthesis

The crystals of complexes **1** and **2** were synthesized by hydrothermal reactions. The influences of the reaction conditions were investigated. pH values have significant impacts on the quality of the crystals and structure of the final products. For the synthesis of complex **1** and **2**, the suitable pH are 7–8 and 6–7, respectively. The different products with uncertain structures would be obtained as powder or microcrystal in other pH values conditions. In addition, the influence of the solutions was also investigated by changing the ratio of water and ethanol. However, the same products were obtained with slight difference in the yields.

3.2. Structure description

Single-crystal X-ray diffraction analysis showed that the asymmetric unit of **1** consists of three half bimb molecules, half of Co^{II} cation, one uncoordinated nitrate anion and one lattice water molecule (Fig. 1a). In **1**, Co^{II} cation lies on an inversion center, which is six-coordinated in a octahedral environment defined by six N atoms from six bimb molecules with the Co–N distances in range



Fig. 1. (a) Stick-ball representation of the molecule structure of **1** with thermal ellipsoids at 50% probability, where hydrogen atoms are omitted for clarity; (b) Schematic illustration of 3D 6-connected honeycomb framework (When the bimb molecule is seen as straight line, the framework would be reduced to pcu α -Po net with the Schläfli symbol of (4¹².6³)); (c) 2-fold interpenetrated pcu net. Symmetry code: (I) 1 – x, –2 – y, –z; (II) – x, y, –0.5 – z; (III) – x, –1 – y, –z; (IV) 2 – x, y, 0.5 – z.

of 2.137(2)–2.219(2) Å. As shown in Fig. 1, bimb (N1) and bimb (N3) molecules exhibit *trans*-conformation, however, bimb (N5) molecule bears *cis*-conformation. *trans*-Bimb (N1) and *cis*-bimb (N5) molecules link the Co^{II} cation to form a double chain structure along [201] direction, furthermore, the *trans*-bimb(3) molecules connect these chains to build up a 3D honeycomb framework containing channels with large dimension of 13.772(6) Å × 19.308(4) Å parallel to [201] direction (Fig. 1b). Topological analysis indicates that this framework can be reduced to a pcu α -Po framework with the Schläfli symbol of (4¹²·6³) [43]. The large channels provide appropriate environment for interpenetration occurrence, which lead to a novel 2-fold interpenetrated pcu framework with the uncoordinated nitrate anions and water molecules filling into the interspaces (Fig. 1c).

Single-crystal X-ray diffraction analysis showed that the asymmetric unit of **2** consists of two half bimb molecules, one Co^{II} cation, one bdc anion and one lattice water molecule (Fig. 2a). The Co^{II} cation is four-coordinated in a tetrahedral environment defined by two N atoms from two bimb molecules and two O atoms from two bdc anions. In contrast to the octahedral coordinated Co^{II} cation in 1, the bonds (Co–N and Co–O) in 2 are shorter with distances in range of 1.997–2.015(2) Å. As shown in Fig. 2a, both bimb molecules show *cis*-conformation, which bridge the Co^{II} cations to form two helical chains with a same inner diameter of 6.431(1) Å along [100] and [010] directions, respectively. Meanwhile, bdc anions link the Co^{II} cations to build up another helical chain with a relatively small inner diameter of 2.624(1) Å along [001] direction. These helical chains bear different rotation directions, in which the bdc chains connect with the bimb chains to construct an unusual 3D uoc type topology framework with a Schlafli symbol of $(4^2 \cdot 8^4)$ (Fig. 2b) [44]. Similar to complex **1**, the uoc type framework of **2** also has a large cavity with the dimension of $12.616(2) \times 13.606(4)$ Å along [010] direction, which tends to form a 2-fold interpenetrated unusual uoc framework with the uncoordinated water molecules filling into the interspaces (Fig. 2c).

How to get oriented structures has been of great interest and an increasing effort has been devoted to this area. Therefore, it is very interesting to compare these two 2-fold interpenetrating frameworks to know the effect of introducing bdc ligand. Both complexes 1 and 2 can be seen as consisting of three coordination chains, zigzag conformation for 1 and helical conformation for 2 respectively. In 1 and 2, these chains interlink each other to form the skeletons possessing honeycomb-like cavities with large dimensions, which makes interpenetration occurrence possible. However, the cobalt coordination sphere is changed from six-coordinated octahedral geometry to four-coordinated tetrahedral geometry by introducing the bdc ligand, which tuned the framework from pcu to uoc types. Previous studies also show that cobalt is usually six-coordinated when only coordinating with the imidazole containing ligand, but if carboxylic acid is introduced as the mixed ligand, the cobalt tends to take four-coordinated tetrahedral geometry [28,45,46].

3.3. PXRD and TGA

In order to characterize the phase purity of other crystalline materials, PXRD patterns for **1** and **2** were performed at room temperature (Fig. S2 ESI†). The location and intensity of diffraction peaks of calculated and experimental patterns match well, indicating that these two coordination polymers were synthesized as a single phase.



Fig. 2. (a) Stick-ball representation of the molecule structure of **2** with thermal ellipsoids at 50% probability, where hydrogen atoms are omitted for clarity; (b) Schematic illustration of 3D 4-connected uoc type topology framework with a Schlafli symbol of $(4^2 \cdot 8^4)$; (c) 2-fold interpenetrated uoc net. Symmetry code: (1) –*x*, 0.5 – *y*, *z*; (II) –*x*, 1.5 – *y*, *z*; (III) –0.25 + *y*, 0.75 – *x*, -0.25 + *z*.

The TG measurements of **1** and **2** were carried out under atmosphere in the temperature range 30–800 °C (Fig. S3, ESI†). The TG curve of **1** shows that the weight loss of 3.24% appearing from 50 to 178 °C corresponds to the loss of two lattice water molecules (calc. 3.10%). The framework collapses starting at 278 °C, which decomposed orderly resulting in the residue CoO at 525 °C (calc. 6.45%, found 5.95%). For **2**, the first weight loss of 3.42% at 50–243 °C was consistent with the loss of one lattice water molecule (calc. 3.24%). The framework of **2** collapses starting at 262 °C, then loses its weight of 84.67% between 262 and 502 °C, corresponding to the decomposition of all bimb and bdc molecules (calc. 86.14%) with the resulting residue of CoO.

The results of *PXRD* and TGA are consistent with the structures of the two complexes.

3.4. Photoluminescence properties

Photoluminescence properties of metal-organic complexes have been widely investigated for their potential applications as fluorescence-emitting materials, owing to their higher thermal stability than pure organic materials and the ability to tune the emission wavelength by adopting different metal cations [47].



Fig. 3. Solid-state photoluminescence spectra of bimb, complexes 1 and 2.

The photoluminescence properties of bimb, **1** and **2** were studied in solid state at room temperature. As shown in Fig. 3, bimb molecule exhibits two emission bands at 428 and 464 nm ($\lambda_{ex} = 350$ nm), which is assigned to $\pi^* \rightarrow \pi$ intraligand fluorescence. In complex **1**, the two emission bands at 422 and 458 nm ($\lambda_{ex} = 350$ nm) both show blue shifts of 6 and 8 nm, respectively comparison with bimb molecule. Interestingly, the emission intensity of the band at 422 nm exhibits an obvious increase in contrast to the other one. Complex **2** shows three emission bands at 412, 430 nm ($\lambda_{ex} = 350$ nm), in which the bands of 412 nm arises from the bimb molecule with blue shifts of 16 nm (Fig. S4). The emissions of complexes **1** and **2** may be attributed to a joint contribution of the $\pi^* \rightarrow \pi$ intraligand transitions and charge transfer transitions between the bimb and/or bdc molecules and Co^{II} cations [48,49].

4. Conclusions

In summary, the present investigation demonstrated the structures and properties of two bimb-cobalt coordination polymers. The results indicate that the coordination polymers based on long spacer bimb ligand are inclined to form interpenetrating structures, which can be tuned by mixed carboxylic ligand. Complex **1** shows a 2-fold pcu interpenetrating framework. However, a rare 2-fold uoc interpenetrating framework is formed in complex **2** owing to the introduction of bdc molecule causing the cobalt coordination sphere changing from six-coordinated octahedral geometry to four-coordinated tetrahedral geometry. Luminescent analyses of complexes **1** and **2** reveal that the Co^{II} cations are able to tune the luminescent emission of ligands.

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Appendix A. Supplementary material

CCDC 901311 and 905892 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at http:// dx.doi.org/10.1016/j.ica.2013.03.046.

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