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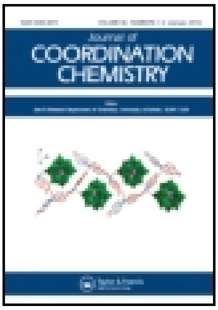

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Zengming Man [a] & Feng Guo [a]
[a] Key Laboratory of Coordination Chemistry and Functional Materials in Universities of Shandong , Dezhou University , Shandong , PR China

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# Coordination polymers constructed from an asymmetric biphenyl-dicarboxylate and N-donor ligands: structures and photoluminescent properties

ZENGMING MAN and FENG GUO*

*Key Laboratory of Coordination Chemistry and Functional Materials in Universities of Shandong, Dezhou University, Shandong, PR China*

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Two new metal coordination polymers, $[Zn_2(bpdc)_2(bip)(H_2O)]_n$ (**1**) and $[Cd(bpdc)(bip)]_n$ (**2**) [$H_2$bpdc = biphenyl-2,4′-dicarboxylic acid and bi*p* = 1,5-bis(2-methyl-imidazol-1-yl)pentane], have been synthesized and characterized by IR, elemental analysis, and X-ray diffraction. In **1** and **2**, the carboxylates adopt three coordination modes: bis-monodentate, monodentate, and bis-chelating. Compounds **1** and **2** are two-dimensional layer structures, but have different topological net: (3,6)-connected net with $(4^3)_2(4^6.6^6.8^3)$ and 4-connected net $(4^4.6^2)$, respectively. Luminescent properties of **1** and **2** are investigated.

*Keywords:* Crystal structure; Luminescence; Coordination polymer

## 1. Introduction

Construction of new coordination polymers has attracted attention due to their diverse structural topologies and potential applications as functional materials such as gas storage, magnetism, catalysis, and luminescence [1–14]. Different dimensional coordination polymers have been prepared with a variety of carboxylates; biphenyl dicarboxylates such as the biphenyl-2,2′-dicarboxylic acid and biphenyl-4,4′-dicarboxylic acid are good candidates for construction of coordination polymers [15–18]. Investigations of unsymmetrical biphenyl dicarboxylates such as biphenyl-2,4’-dicarboxylic acid are fewer than symmetrical biphenyl dicarboxylates [19–24]. N-imidazole ligands with –$CH_2$-spacers are good bridging ligands. The flexible nature of –$CH_2$-spacers allows the ligands to bend and rotate freely when coordinating to metal centers to conform to the coordination geometries of metal ions [25–27]. Mixture of carboxylates with N-donor auxiliary ligands is good for construction of new coordination polymers [28, 29].

In this paper, we have prepared two new coordination polymers using biphenyl-2,4′-dicarboxylic acid and 1,5-bis(2-methyl-imidazol-1-yl)pentane constructing $[Zn_2(bpdc)_2(bip)(H_2O)]_n$ (**1**) and $[Cd(bpdc)(bip)]_n$ (**2**). Their single crystal structures and luminescent properties have been investigated.

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*Corresponding author. Email: guofeng1510@yeah.net

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## 2. Experimental

### 2.1. *Materials and physical measurements*

All reagents and solvents were commercially available and used without purification. The bip was synthesized as scheme S1. Elemental analyses were carried out on a Carlo Erba 1106 full-automatic trace organic elemental analyzer. FT-IR spectra were recorded with a Bruker Equinox 55 FT-IR spectrometer as dry KBr pellets from 400 to 4000 $cm^{-1}$. Solid-state fluorescence spectra were recorded on a Hitachi F-4600 equipped with a xenon lamp and a quartz carrier at room temperature. X-ray powder diffraction (XRPD) measurements were performed on a Bruker D8 diffractometer operated at 40 kV and 40 mA using Cu-$K_\alpha$ radiation ($\lambda = 0.15418$ nm).

### 2.2. *Synthesis of* $[Zn_2(bpdc)_2(bip)(H_2O)]_n$ *(1)*

Hydrothermal reaction of $Zn(OAc)_2 \cdot 2H_2O$ (0.108 g, 0.5 mmol), $H_2bpdc$ (0.121 g, 0.5 mmol), NaOH (0.04 g, 1 mmol), bip (0.116 g, 0.5 mmol) and distilled water (18 mL) was heated to 160 °C for 96 h in a 25 mL stainless steel reactor with a Teflon liner. After cooling to room temperature, yellow block crystals were obtained and washed with alcohol several times (Yield: 38% based on Zn). Elemental Anal. Calcd for $C_{41}H_{38}Zn_2N_4O_9$ (%): C, 57.16; H, 4.45; N, 6.50. Found: C, 57.19; H, 4.42; N, 6.57. IR: 3215w, 1657s, 1524w, 1493s, 1402s, 1322m, 1240m, 1107w, 925m, 857m, 726w.

### 2.3. *Synthesis of* $Cd(bpdc)(bip)]_n$ *(2)*

Compound **2** was synthesized similar to **1** by using $Cd(OAc)_2 \cdot 2H_2O$ (0.133 g, 0.5 mmol) in place of $Zn(OAc)_2 \cdot 2H_2O$. The resulting product was yellow block crystals that were washed with alcohol to give pure samples (Yield: 43% based on Cd). Elemental Anal. Calcd for $C_{27}H_{28}CdN_4O_4$ (%): C, 55.44; H, 4.82; N, 9.58. Found: C, 55.36; H, 4.89; N, 9.55. IR: 1607s, 1564s, 1421w, 1293m, 1248m, 1104w, 963m, 891w, 778m, 642w.

### 2.4. *X-ray Crystallography*

Single crystal X-ray diffraction analyses of **1** and **2** were carried out on a Bruker SMART APEX II CCD diffractometer equipped with graphite monochromated Mo–$K_\alpha$ radiation ($\lambda = 0.71073$ Å) by using a $\omega$-scan mode. Empirical absorption correction was applied using SADABS [30]. All the structures were solved by direct methods and refined by full-matrix least-squares on $F^2$ using SHEXL 97 [31]. All non-hydrogen atoms were refined anisotropically; hydrogens were located by geometric calculations, and their positions and thermal parameters were fixed during the structure refinement. Crystallographic data and experimental details of structural analyses for coordination polymers are summarized in table 1. Selected bond length and angle parameters are listed in table 2.

Table 1. Crystallographic data and structure refinement for **1** and **2**.

| | | |
|---|---|---|
| Empirical formula | $C_{41}H_{38}Zn_2N_4O_9$ | $C_{27}H_{28}CdN_4O_4$ |
| Formula weight | 861.53 | 584.93 |
| Temperature [K] | 293(2) | 293(2) |
| Crystal system/space group | Triclinic, *P*-1 | Monoclinic, $P2_1/n$ |
| Unit cell dimensions | $a = 10.721(6)$ Å | $a = 11.844(5)$ Å |
| | $b = 10.792(6)$ Å | $b = 15.988(7)$ Å |
| | $c = 17.032(9)$ Å | $c = 13.610(6)$ Å |
| | $\alpha = 81.054(10)°$ | $\alpha = 90°$ |
| | $\beta = 81.628(10)°$ | $\beta = 91.583(5)°$ |
| | $\gamma = 76.719(10)°$ | $\gamma = 90°$ |
| Volume (Å$^3$) | 1882.11(18) | 2576.2(17) |
| *Z*, Calculated density (mg m$^{-3}$) | 2, 1.520 | 4, 1.508 |
| Measured reflections | 10,986 | 15,791 |
| Independent reflections | 7673 | 5855 |
| Independent reflections ($I > 2\sigma(I)$) | 6615 | 4767 |
| *F*(000) | 888 | 1192 |
| $\theta$ Range for data collection | 1.22–26.55 | 2.25–27.15 |
| Limiting indices | $-13 \leq h \leq 12$ | $-15 \leq h \leq 11$ |
| | $-13 \leq k \leq 11$ | $-20 \leq k \leq 20$ |
| | $-21 \leq l \leq 21$ | $-17 \leq l \leq 17$ |
| Goodness-of-fit on $F^2$ | 1.047 | 1.032 |
| $R_{\text{int}}$ | 0.014 | 0.024 |
| $R_1{}^a$, $wR_2{}^b$ [$I > 2\sigma$(I)] | $R_1 = 0.0321$, $wR_2 = 0.0941$ | $R_1 = 0.0296$, $wR_2 = 0.0702$ |
| $R_1{}^a$, $wR_2{}^b$ (all data) | $R_1 = 0.0384$, $wR_2 = 0.0985$ | $R_1 = 0.0403$, $wR_2 = 0.0752$ |
| Largest diff. peak and hole (e/Å$^3$) | 1.033 and −0.365 | 0.738 and −0.267 |

$^aR = \sum(||F_o| - |F_c||)/\sum|F_o|$. $^bwR = [\sum w(|F_0|^2 - |F_c|^2)^2/\sum w(F_0^2)]^{1/2}$.

## 3. Results and discussion

### 3.1. *Description of crystal structure*

**3.1.1. [$Zn_2(bpdc)_2(bip)(H_2O)]_n$ (1).** X-ray crystallography reveals **1** crystallizes in the triclinic *P*-1 space group. There are two independent Zn(II) (Zn1 and Zn2) in the asymmetric unit with square-pyramidal and tetrahedral geometry, respectively. As shown in figure 1(a), Zn1 is surrounded by four oxygens from four different bpdc and one nitrogen from one bip. Zn2 is coordinated by three oxygens from two carboxylates and coordinated water, and one nitrogen from bip. The two phenyl rings about the central bond have a twist with a dihedral angle of 46.45°. The carboxylates adopt bis-monodentate and monodentate coordination. Two symmetry-related Zn1 are bridged by four carboxylates into a dinuclear Zn(II) unit with short Zn···Zn distance of 3.036 Å [32].

For a simple framework, each Zn2 can be regarded as a 3-connected node and each dinuclear Zn1 can be defined as a 6-connected node, so the 2-D framework can be simplified as a binodal (3,6)-connected network with Schläfli symbol $(4^3)_2(4^6.6^6.8^3)$ (figure 1(b)). Hydrogen bonding exists [the distance of O(9)-H(9D)···O(4) is 2.649(3) Å and the angle of O(9)–H(9D)···O(4) is 172(4)° (symmetry code: $1-x$, $-y$, $-z$)], which leads to the 3-D supramolecule structure. There are also $\pi$–$\pi$ interactions between the phenyl rings of carboxylate and imidazole with a centroid-to-centroid distance of 3.888(2) Å (figure 1(c)).

**3.1.2. $Cd(bpdc)(bip)]_n$ (2).** The structure of **2** (figure 2(a)) shows each Cd1 surrounded by four oxygens from two carboxylates and two nitrogens from two bip in a distorted

Table 2. Selected bond lengths (Å) and angles (°) for **1** and **2**.

| | | | |
|---|---|---|---|
| Compound **1** | | | |
| Zn(1)–N(1) | 2.003(2) | Zn(1)–O(1) | 2.095(2) |
| Zn(1)–O(5) | 2.030(2) | Zn(1)–O(2)$^{i}$ | 2.118(2) |
| Zn(1)–O(6)$^{i}$ | 2.038(2) | Zn(2)–N(4)$^{iii}$ | 2.001(2) |
| Zn(2)–O(8) | 1.947(2) | Zn(2)–O(4)$^{ii}$ | 2.624(2) |
| Zn(2)–O(3)$^{ii}$ | 1.974(2) | Zn(2)–O(9) | 1.976(2) |
| N(1)–Zn(1)–O(5) | 106.12(7) | O(6)$^{i}$–Zn(1)–O(1) | 89.20(6) |
| N(1)–Zn(1)–O(6)$^{i}$ | 97.41(7) | N(1)–Zn(1)–O(2)$^{i}$ | 98.49(7) |
| O(5)–Zn(1)–O(6)$^{i}$ | 156.47(6) | O(5)–Zn(1)–O(2)$^{i}$ | 85.64(7) |
| N(1)–Zn(1)–O(1) | 104.43(7) | O(6)$^{i}$–Zn(1)–O(2)$^{i}$ | 90.68(7) |
| O(5)–Zn(1)–O(1) | 85.30(6) | O(1)–Zn(1)–O(2)$^{i}$ | 156.90(7) |
| O(8)–Zn(2)–O(3)$^{ii}$ | 106.23(7) | O(8)–Zn(2)–N(4)$^{iii}$ | 112.12(8) |
| O(8)–Zn(2)–O(9) | 99.59(8) | O(3)$^{ii}$–Zn(2)–N(4)$^{iii}$ | 108.39(8) |
| O(3)$^{ii}$–Zn(2)–O(9) | 111.80(9) | O(9)–Zn(2)–N(4)$^{iii}$ | 117.94(10) |
| O(8)–Zn(2)–O(3)$^{ii}$ | 106.23(7) | O(8)–Zn(2)–O(4)$^{ii}$ | 157.92(7) |
| Compound **2** | | | |
| Cd(1)–N(4)$^{i}$ | 2.253(2) | Cd(1)–O(3) | 2.344(2) |
| Cd(1)–N(1) | 2.294(2) | Cd(1)–O(2)$^{ii}$ | 2.378(2) |
| Cd(1)–O(1)$^{ii}$ | 2.338(2) | Cd(1)–O(4) | 2.396(2) |
| N(4)$^{i}$–Cd(1)–N(1) | 92.40(8) | O(1)$^{ii}$–Cd(1)–O(3) | 145.94(7) |
| N(4)$^{i}$–Cd(1)–O(1)$^{ii}$ | 111.10(8) | N(4)$^{i}$–Cd(1)–O(2)$^{ii}$ | 99.25(8) |
| N(1)–Cd(1)–O(1)$^{ii}$ | 89.07(7) | N(1)–Cd(1)–O(2)$^{ii}$ | 144.08(7) |
| N(4)$^{i}$–Cd(1)–O(3) | 91.08(7) | O(1)$^{ii}$–Cd(1)–O(2)$^{ii}$ | 55.04(7) |
| N(1)–Cd(1)–O(3) | 116.43(8) | O(3)–Cd(1)–O(2)$^{ii}$ | 97.31(7) |
| N(4)$^{i}$–Cd(1)–O(4) | 146.09(7) | O(1)$^{ii}$–Cd(1)–O(4) | 100.98(7) |
| N(1)–Cd(1)–O(4) | 98.97(8) | O(3)–Cd(1)–O(4) | 55.26(7) |

octahedral geometry [33, 34]. Carboxylates in **2** are bis-chelating and the two phenyl rings have a twist with a dihedral angle of 43.80°, leading to a 1-D zigzag chain. The 1-D chains are further connected by bip to form a 2-D (4,4)-connected layer with $(4^4.6^2)$ topological notation (figure 2(b)).

In **2**, intermolecular *π–π* interactions assemble the 2-D structure into a 3-D supramolecular framework. The interplanar Cg(1)–Cg(1) and Cg(3)–Cg(3) centroid distances are 3.882 (2) Å (symmetry code: $-x$, $1-y$, $1-z$) and 3.505(2) Å (symmetry code: $-x$, $1-y$, $-z$), which indicate the presence of face to face *π–π* stacking interactions that stabilize the crystal structure (figure 2(c)).

### 3.2. *XRPD and thermogravimetric analyzes (TGA)*

XRPD was used to confirm the phase purity of bulk materials of **1** and **2** at room temperature, indicating that the synthesized materials are homogeneous systems (figure S1). Thermogravimetric analyses (TGA) of **1** and **2** were performed on polycrystalline samples from room temperature to 600 °C under nitrogen. The TGA curve of **1** reveals that coordination water is gradually removed from room temperature to 150 °C (obsd 2.10%, calcd 2.09%). The whole framework begins to decompose at 450 °C. Complex **2** is stable up to 230 °C, above which the framework starts to decompose (figure S2) Supplementary material.

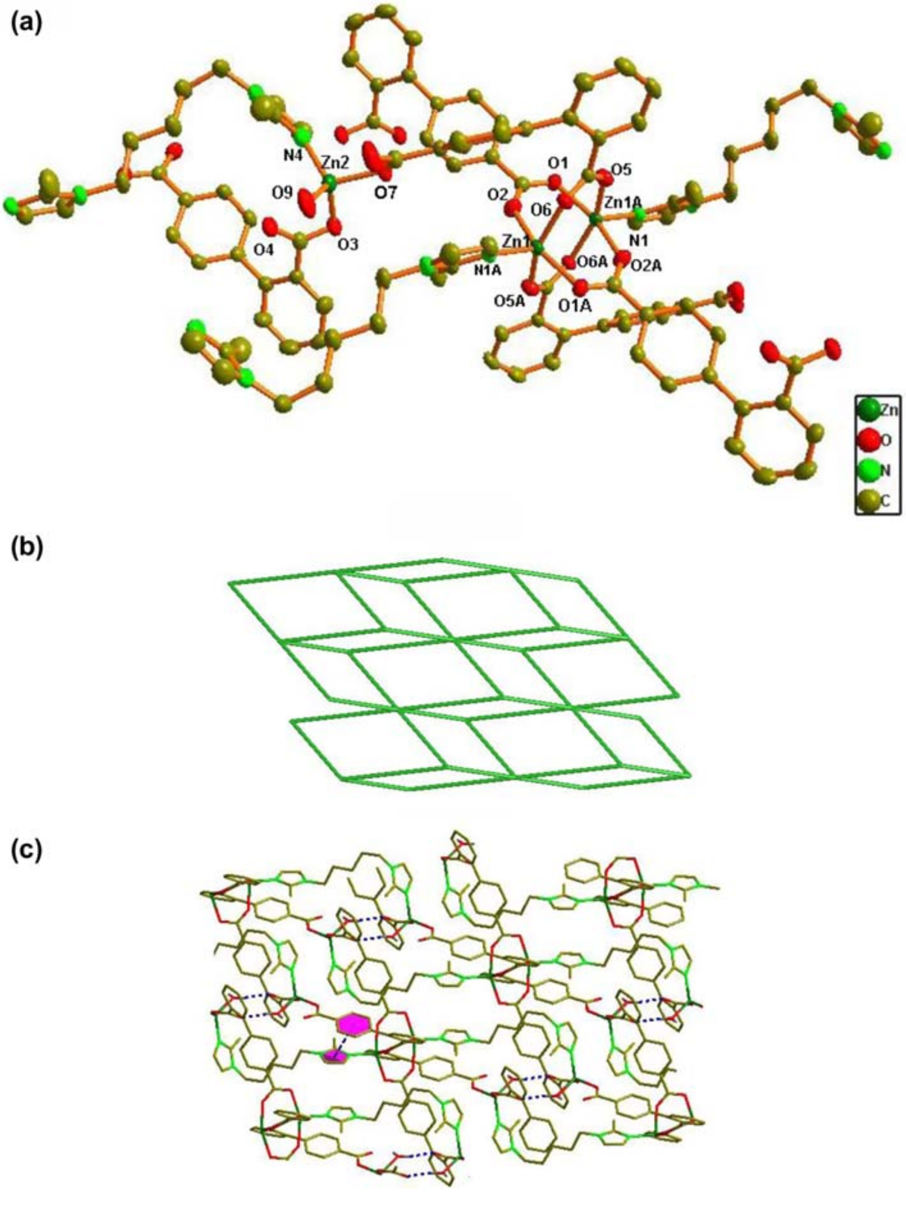

Figure 1. (a) Coordination environment of Zn(II) in **1**. Hydrogens are omitted for clarity. (b) The network, $(4^3)_2(4^6.6^6.8^3)$ topology notation, for the 2-D layer structure. (c) the supramolecular structure of **1** formed by hydrogen bonding and π–π interactions.

### 3.3. *Luminescent properties*

Emission spectra of **1** and **2** at room temperature are depicted in figure 3. $H_2$bpdc ligand exhibits a fluorescent emission at 354 nm ($\lambda_{ex} = 278$ nm) which may be attributed to $\pi^*$–π transition and bip has no obvious emission at 300–400 nm [22]. Intense luminescence emission bands are observed at 388 nm ($\lambda_{ex} = 324$ nm) for **1** and 385 nm ($\lambda_{ex} = 331$ nm) for

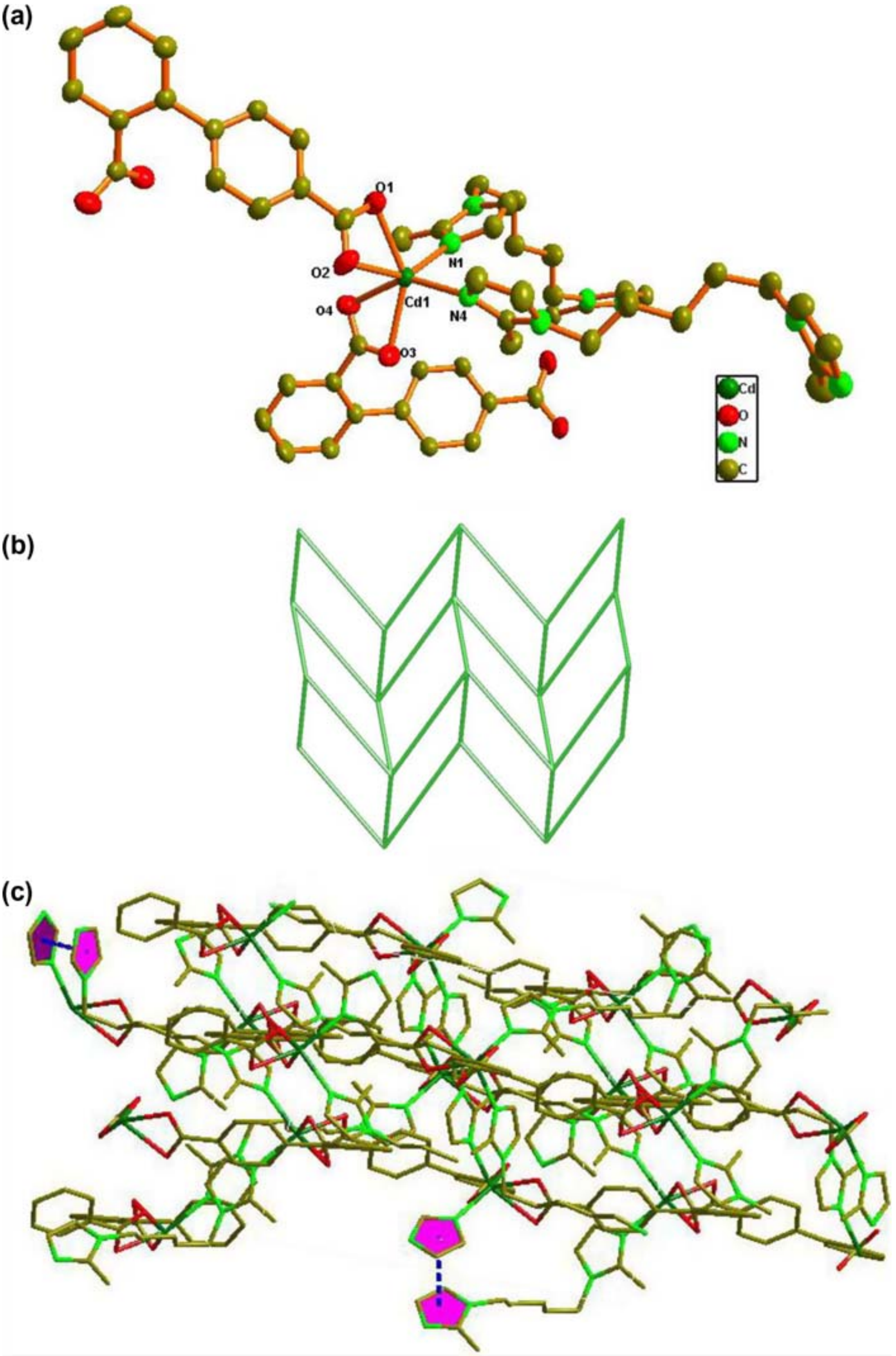

Figure 2. (a) Coordination environment of Cd(II) in **2**. Hydrogens and free water are omitted for clarity. (b) The $(4^4.6^2)$ topology notation for the 2-D layer structure. (c) The 3-D supramolecular structure formed by π–π interactions.

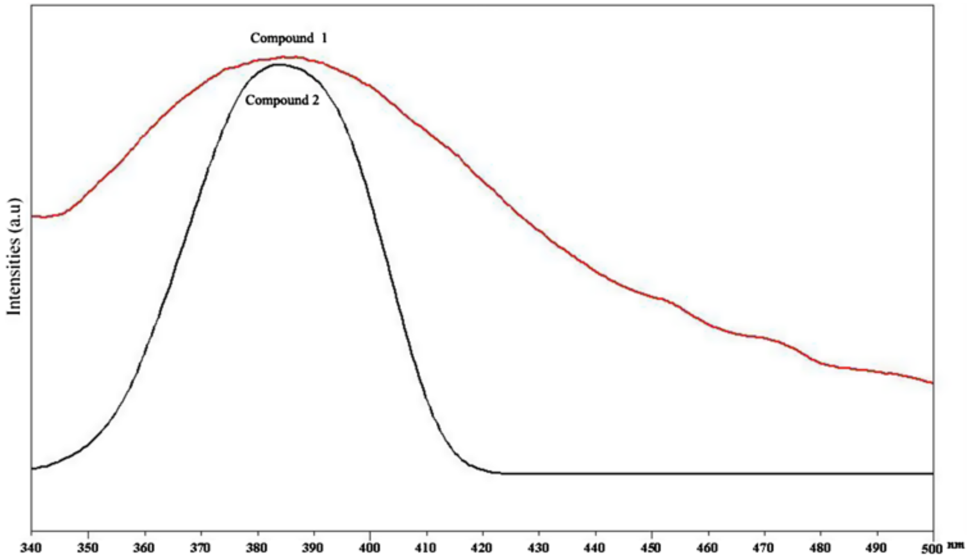

Figure 3. Solid-state emission spectra of **1** and **2** at room temperature.

**2**. Zn is difficult to oxidize or reduce due to its $d^{10}$ configuration; thus, fluorescent emissions of **1** and **2** are neither metal-to-ligand nor ligand-to-metal charge transfer, assigned to intraligand fluorescent emission [35–38]. The different emission positions and intensities of **1** and **2** may be due to the significant difference of their structures because enhancement of luminescence is associated with coordination of ligands to metal [39].

## 4. Conclusions

We synthesized and characterized $[Zn_2(bpdc)_2(bip)(H_2O)]_n$ (**1**) and $[Cd(bpdc)(bip)]_n$ (**2**). Compounds **1** and **2** have different topologies due to the different metal ions. We also investigated photoluminescence of **1** and **2**.

## Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC reference numbers: 862574 and 862575. These data can be obtained free of charge at http://www.ccdc.cam.ac.uk (or Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44 1223 336 033; E-mail: deposit@ccdc.cam.ac.uk).

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