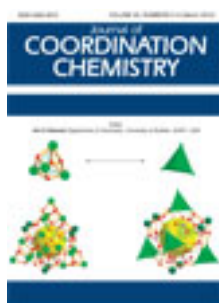


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### Synthesis and structural characterization of three supramolecular coordination polymers constructed from different metal ions and 2,2'-bipyridyl-3,3'-dicarboxylic acid

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## Synthesis and structural characterization of three supramolecular coordination polymers constructed from different metal ions and 2,2'-bipyridyl-3,3'-dicarboxylic acid

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Three new supramolecular coordination polymers based on 2,2'-bipyridyl-3,3'-dicarboxylic acid (H<sub>2</sub>BPDC) and Mn(II), Fe(II), and Zn(II) were synthesized under hydrothermal conditions and characterized with single-crystallographic X-ray analysis and IR spectrum. Complex **1** exhibits a 1-D, chain-like structure, which is further connected to 2-D supramolecular layer structure through hydrogen bonds. Complex **2** exhibits a 3-D supramolecular structure constructed from 1-D chains through hydrogen bonds and  $\pi$ - $\pi$  interactions. Like **1**, **3** also shows 2-D supramolecular layer structure based on 1-D chains. Furthermore, the fluorescence of **3** was studied.

**Keywords:** Supramolecular coordination polymer; Hydrogen bond;  $\pi$ - $\pi$  Interaction; Fluorescence property

### 1. Introduction

Supramolecular coordination polymers based on metal and organic building blocks has been rapidly expanding for their diverse topologies and potential applications in catalysis, gas sorption, and magnetism [1–4]. During formation of supramolecular coordination polymers, weak forces, such as hydrogen bonds and  $\pi$ - $\pi$  interactions induce intra-/inter-molecular interactions in the self-assembly process and direct the formation of the final structures [5–8]. During construction of supramolecular coordination polymers, pyridine-carboxylic acid ligands are multi-functional units [9]; not only can they form supramolecular architectures through  $\pi$ - $\pi$  interactions engendered by aromatic–aromatic stacking but they are also capable of functioning as hydrogen-bond donors as well as hydrogen-bond acceptors [10, 11].

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In the family of pyridine-carboxylic acid ligands, 2,2'-bipyridyl-3,3'-dicarboxylic acid (H<sub>2</sub>BPDC) is a good candidate for preparation of supramolecular coordination polymers for several reasons: first, during the construction of supramolecular networks, H<sub>2</sub>BPDC can act as hydrogen-bond donors and acceptors [12, 13]; second, owing to the rich coordination modes of chelating bipyridine and two carboxylic groups, H<sub>2</sub>BPDC can adopt various connections in formation of supramolecular coordination polymers [14–18]; third, distortion of bipyridine from the central bond endows H<sub>2</sub>BPDC a peculiar ability to link metal ions into supramolecular complexes with interesting structural features [19, 20].

Hydrothermal synthesis is a powerful method for preparation of supramolecular coordination polymers; superheated reaction systems provide ideal conditions for the crystal growth, owing to the enhanced transporting ability of water [21–24]. In such temperature and pressure environment, problems of different solubilities for organic ligands and metal salts are minimized [25–27].

Herein, under hydrothermal conditions, we synthesize three supramolecular coordination polymers [Mn(BPDC)(H<sub>2</sub>O)<sub>3</sub>·H<sub>2</sub>O]<sub>n</sub> (**1**), [Fe(BPDC)(H<sub>2</sub>O)<sub>2</sub>]<sub>n</sub> (**2**), and [Zn(BPDC)(H<sub>2</sub>O)]<sub>n</sub> (**3**) from H<sub>2</sub>BPDC and different metal ions. Complexes **1** and **3** both exhibit 2-D supramolecular layer structures constructed from 1-D chains with hydrogen bonds. Complex **2** shows a 3-D supramolecular structure composed of 1-D chains with hydrogen bonds and  $\pi$ - $\pi$  interactions. The fluorescence of **3** was studied.

## 2. Experimental

### 2.1. Materials and methods

H<sub>2</sub>BPDC was synthesized by literature methods [28]. All other chemicals purchased were of reagent grade and used without purification. Elemental analyses (C, H, and N) were performed on a Perkin-Elmer 2400 CHN elemental analyzer. FT-IR spectra were recorded from 4000 to 400 cm<sup>-1</sup> on an Alpha Centaur FTIR spectrophotometer using KBr pellets. Thermal gravimetric analyses (TGA) were performed on a Perkin-Elmer TGA7 instrument in flowing N<sub>2</sub> with a heating rate of 10°C min<sup>-1</sup>. All measurements were performed at room temperature.

### 2.2. Preparation of **1**

Complex **1** was prepared from a mixture of Mn(OAc)<sub>2</sub>·4H<sub>2</sub>O (0.061 g, 0.25 mmol), H<sub>2</sub>BPDC (0.122 g, 0.50 mmol), and 7 mL H<sub>2</sub>O with pH adjusted to 6 with 1 mol L<sup>-1</sup> NaOH. The mixture was stirred, then placed in a Teflon-lined stainless steel bomb and heated to 160°C under autogenous pressure for 4 days. The reaction system was then cooled to room temperature during 24 h. Yellow crystals of **1** were obtained. Yield: 58% (based on Mn). Elemental Anal. Calcd (%): C, 39.04; H, 3.82; N, 7.59. Found (%): C, 39.06; H, 4.02; N, 7.46. IR (cm<sup>-1</sup>): 3342(m), 2925(s), 1615(s), 1592(s), 1396(s), 1169(m), 810(m).

### 2.3. Preparation of **2**

Complex **2** was prepared from a mixture of  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$  (0.050 g, 0.25 mmol),  $\text{H}_2\text{BPDC}$  (0.122 g, 0.50 mmol), and 8 mL  $\text{H}_2\text{O}$  with pH adjusted to 7 with  $1 \text{ mol L}^{-1}$  NaOH. The mixture was stirred then placed in a Teflon-lined stainless steel bomb and heated to  $150^\circ\text{C}$  under autogenous pressure for 5 days. The reaction system was then cooled to room temperature during 24 h. Green crystals of **2** were obtained. Yield: 63% (based on Fe). Elemental Anal. Calcd (%): C, 43.14; H, 3.02; N, 8.39. Found (%): C, 43.25; H, 3.08; N, 8.45. IR ( $\text{cm}^{-1}$ ): 3286(m), 1664(m), 1585(s), 1391(s), 1066(m), 841(m).

### 2.4. Preparation of **3**

Complex **3** was prepared from a mixture of  $\text{Zn}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$  (0.055 g, 0.25 mmol),  $\text{H}_2\text{BPDC}$  (0.122 g, 0.50 mmol), and 10 mL  $\text{H}_2\text{O}$  with pH adjusted to 5 with  $1 \text{ mol L}^{-1}$  NaOH. The mixture was stirred then placed in a Teflon-lined stainless steel bomb and heated to  $120^\circ\text{C}$  under autogenous pressure for 3 days. The reaction system was then cooled to room temperature during 24 h. Colorless crystals of **3** were obtained. Yield: 71% (based on Zn). Elemental Anal. Calcd (%): C, 44.27; H, 2.48; N, 8.60. Found (%): C, 44.38; H, 2.61; N, 8.52. IR ( $\text{cm}^{-1}$ ): 3315(m), 1591(s), 1445(s), 1408(s), 1311(w), 1159(w), 989(w), 712(m), 541(w).

### 2.5. X-ray crystallography

Single crystals of **1–3** were glued on glass fibers, respectively. Data were collected on a Bruker AXS SMART APEX II CCD diffractometer at 293 K. The structures were solved by direct methods and refined by full-matrix least-squares on  $F^2$  using the SHELXTL-97 crystallographic software package [29, 30]. Anisotropic thermal parameters were used to refine all non-hydrogen atoms. Carbon-bound hydrogen atoms were placed in calculated positions; oxygen-bound hydrogen atoms were located in the difference Fourier maps and kept in that position. Further details of the X-ray structural analyses are given in table 1. Selected bond lengths and angles are listed in table 2.

## 3. Results and discussion

### 3.1. Structure description

Single-crystal X-ray analysis reveals that **1** crystallizes in the monoclinic system with  $P2_1/n$  space group. There exists only one crystallographic independent Mn in the fundamental unit as shown in figure 1(a). Mn binds with one carboxylate oxygen atom and two nitrogen atoms from  $\text{HBPDC}^-$  with Mn–O3 distance of 2.053 Å, Mn–N1 distance of 2.055 Å, and Mn–N2 distance of 2.096 Å. The other three coordination sites are occupied by three water molecules with Mn–O distances from 2.035 to 2.092 Å. This results in a distorted octahedral coordination mode of Mn. Two nitrogen atoms and one carboxylate oxygen atom of  $\text{HBPDC}^-$  bind to neighboring Mn atoms, giving a 1-D, chain-like structure as shown in figure 2(a). Adjacent chains are connected through

Table 1. Crystal data and structure refinements for **1**–**3**.

	<b>1</b>	<b>2</b>	<b>3</b>
Empirical formula	C <sub>12</sub> H <sub>14</sub> N <sub>2</sub> O <sub>8</sub> Mn	C <sub>12</sub> H <sub>10</sub> N <sub>2</sub> O <sub>6</sub> Fe	C <sub>12</sub> H <sub>8</sub> N <sub>2</sub> O <sub>5</sub> Zn
Formula weight	369.19	334.07	325.57
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	<i>P</i> 2 <sub>1</sub> / <i>n</i>	<i>C</i> 2/ <i>c</i>	<i>P</i> 2 <sub>1</sub> / <i>c</i>
Unit cell dimensions (Å, °)			
<i>a</i>	9.938(11)	11.618(3)	12.831(3)
<i>b</i>	9.174(10)	8.0409(19)	6.0875(12)
<i>c</i>	15.982(18)	12.965(3)	17.284(4)
$\beta$	96.705(2)	100.191(3)	104.08(3)
Volume (Å <sup>3</sup> ), <i>Z</i>	1447.1(3), 4	1192.1(5), 4	1309.5(5), 4
Calculated density (g cm <sup>-3</sup> )	1.695	1.861	1.651
<i>F</i> (000)	756	680	656
Reflections collected	8965	3527	11,815
Reflections unique	2845	1144	2949
<i>R</i> (int)	0.0213	0.0126	0.0457
<i>S</i>	1.143	1.050	1.048
<i>R</i> <sub>1</sub> [ <i>I</i> > 2σ( <i>I</i> )]	0.0436	0.0346	0.0414
<i>wR</i> <sub>2</sub> [ <i>I</i> > 2σ( <i>I</i> )]	0.1559	0.1297	0.1359
<i>R</i> <sub>1</sub> (all data)	0.0505	0.0355	0.0467
<i>wR</i> <sub>2</sub> (all data)	0.1649	0.1313	0.1421

$$R_1 = \sum ||F_o| - |F_c|| / \sum |F_o|; wR_2 = \sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]^{1/2}.$$

hydrogen bonds between carboxylate oxygen atoms O1 and O4, and coordinated water molecules O5 and O6 with H1–O5 = 1.891 Å, O1–H1–O5 = 150.49°; O4–H4 = 2.599 Å, O4–H4–O6 = 136.38°, resulting in a 2-D supramolecular layer structure as shown in figure 3(a). Furthermore, there exists a hydrogen bond between free water O1w and O7 with H5–O1w = 1.828 Å, O1w–H5–O7 = 161.35°. An interesting chiral coordination polymer composed of Mn and BPDC has been reported, but this complex exhibits completely different structural features from **1** [14]. Another two coordination polymers with isostructural features constructed from Ni or Co and BPDC have also been reported [13].

The structure of **2** has also been established. There exists only one crystallographic independent Fe in the fundamental unit. Fe(1) binds two carboxylate oxygen atoms and two nitrogen atoms from BPDC<sup>2-</sup>; the last two coordination sites are occupied by water molecules resulting in a distorted octahedron with Fe1–O1 = 2.159 Å, Fe1–O3 = 2.209 Å, Fe1–N1 = 2.242 Å as shown in figure 1(b). Two carboxylates of BPDC<sup>2-</sup> both adopt monodentate coordination and nitrogen atoms chelate, connecting adjacent Fe's together forming a 1-D, chain-like structure as shown in figure 2(b). Adjacent chains are connected by hydrogen bonds and form a 2-D supramolecular layer structure with O2–H2 = 1.909 Å and O2–H2–O3 = 171.98° as shown in figure 3(b). These layers are connected and form a 3-D supramolecular network by  $\pi$ – $\pi$  interactions with distances of neighboring pyridine rings from 3.54 Å to 3.65 Å as shown in figure 4. These hydrogen bonds and  $\pi$ – $\pi$  interactions stabilize the framework.

Coordination polymer **3** crystallizes in a monoclinic system and *P*2<sub>1</sub>/*c* space group. There exists one crystallographic independent Zn in the fundamental unit. Zn(1) is tetrahedral with three carboxylate oxygen atoms from BPDC<sup>2-</sup> and one water molecule. Zn–O bond distances range from 1.932(2) to 1.980(2) Å as shown

Table 2. Selected bond lengths (Å) and angles (°) for **1**–**3**.

<b>1</b>			
Mn(1)–O(3)#1	2.051(2)	Mn(1)–O(5)	2.053(3)
Mn(1)–O(6)	2.094(3)	Mn(1)–O(7)	2.038(3)
Mn(1)–N(1)	2.055(3)	Mn(1)–N(2)	2.096(3)
O(3)#1–Mn(1)–O(5)	90.73(9)	O(3)#1–Mn(1)–O(6)	178.89(10)
O(3)#1–Mn(1)–N(1)	88.13(10)	O(3)#1–Mn(1)–N(2)	84.58(10)
O(5)–Mn(1)–O(6)	89.78(10)	O(5)–Mn(1)–N(2)	92.82(11)
O(7)–Mn(1)–O(3)#1	88.85(10)	O(7)–Mn(1)–O(5)	95.72(14)
O(7)–Mn(1)–O(6)	90.12(11)	O(7)–Mn(1)–N(1)	92.75(13)
O(7)–Mn(1)–N(2)	169.29(13)	N(1)–Mn(1)–O(5)	171.43(11)
N(1)–Mn(1)–O(6)	91.52(11)	N(1)–Mn(1)–N(2)	78.62(10)
N(2)–Mn(1)–O(6)	96.38(10)		
<b>2</b>			
Fe(1)–O(1)#1	2.1589(14)	Fe(1)–O(1)#2	2.1589(14)
Fe(1)–O(3)	2.2094(18)	Fe(1)–O(3)#3	2.2094(18)
Fe(1)–N(1)	2.2415(17)	Fe(1)–N(1)#3	2.2415(17)
O(1)#1–Fe(1)–O(1)#2	87.43(8)	O(1)#1–Fe(1)–O(3)	91.78(6)
O(1)#1–Fe(1)–O(3)#3	82.80(6)	O(1)#2–Fe(1)–O(3)	82.80(6)
O(1)#2–Fe(1)–O(3)#3	91.78(6)	O(1)#1–Fe(1)–N(1)	162.62(6)
O(1)#1–Fe(1)–N(1)#3	102.38(6)	O(1)#2–Fe(1)–N(1)	102.38(6)
O(1)#2–Fe(1)–N(1)#3	162.62(6)	O(3)–Fe(1)–O(3)#3	172.52(8)
O(3)–Fe(1)–N(1)	103.55(6)	O(3)#3–Fe(1)–N(1)	82.60(7)
O(3)–Fe(1)–N(1)#3	82.60(6)	O(3)#3–Fe(1)–N(1)#3	103.55(6)
N(1)–Fe(1)–N(1)#3	72.10(9)		
<b>3</b>			
Zn(1)–O(1)	1.946(2)	Zn(1)–O(3)#2	1.978(2)
Zn(1)–O(4)#1	1.932(2)	Zn(1)–O(5)	1.980(2)
O(1)–Zn(1)–O(5)	113.87(9)	O(1)–Zn(1)–O(3)#2	106.76(9)
O(3)#2–Zn(1)–O(5)	96.80(9)	O(4)#1–Zn(1)–O(1)	112.68(9)
O(4)#1–Zn(1)–O(3)#2	123.22(9)	O(4)#1–Zn(1)–O(5)	102.49(9)

Symmetry transformations used to generate equivalent atoms for **1**: #1:  $-x+5/2, y+1/2, -z+3/2$ ; for **2**: #1:  $-x, y+1, -z+3/2$ ; #2:  $x, y+1, z$ ; #3:  $-x, y, -z+3/2$ ; for **3**: #1:  $-x+1, -y+1, -z$ ; #2:  $x, y-1, z$ .

in figure 3(a). For BPDC<sup>2-</sup>, one carboxylate is monodentate while another adopts bridging coordination. With this connection, adjacent Zn atoms are linked into a 1-D, chain-like structure as shown in figure 3(b). Adjacent chains are connected and form a 2-D layer by hydrogen-bond interactions with O2–H2 = 1.830 Å and O2–H2–O5 = 162.28° as shown in figure 3(c).

### 3.2. IR spectroscopy

For **1**, absorptions at 1592 and 1396 cm<sup>-1</sup> display asymmetric and symmetric vibrations, respectively; the separation ( $\Delta$ ) between  $\gamma_{\text{asym}}$  (CO<sub>2</sub>) and  $\gamma_{\text{sym}}$  (CO<sub>2</sub>) of 196 cm<sup>-1</sup> indicates the presence of monodentate coordination. The IR spectrum of **2** shows the absorptions at 1585 and 1391 cm<sup>-1</sup> and  $\Delta$  of 194 cm<sup>-1</sup>, also indicating monodentate coordination, in agreement with the result of single-crystal analysis. Complex **3** exhibits strong bands at 1591, 1445, and 1408 cm<sup>-1</sup>, which can be attributed to the antisymmetric and symmetric stretches, respectively. The  $\Delta$  values are 146 and 188 cm<sup>-1</sup>, in agreement with the coordination of the carboxylate.

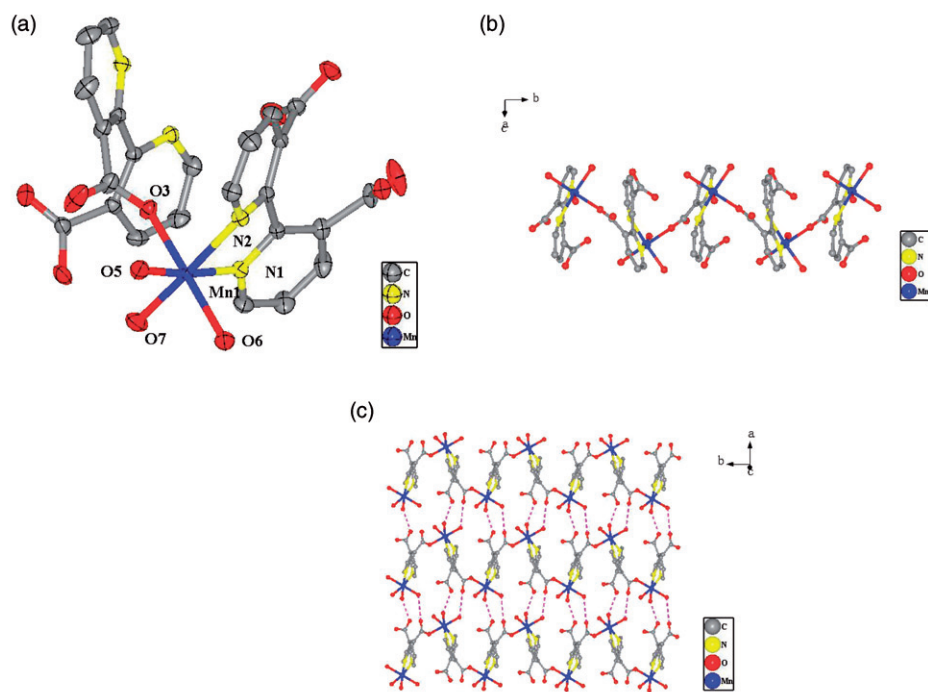


Figure 1. (a) The fundamental unit of **1**, (b) 1-D chain structure of **1**, and (c) 2-D supramolecular layer structure of **1**.

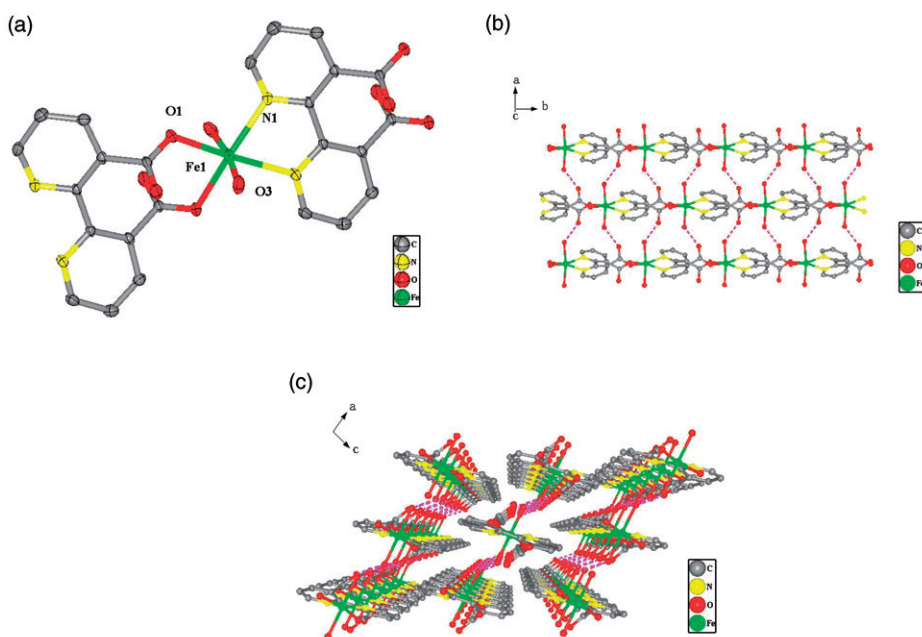


Figure 2. (a) The fundamental unit of **2**, (b) 2-D supramolecular layer structure of **2**, and (c) 3-D supramolecular network of **2**.



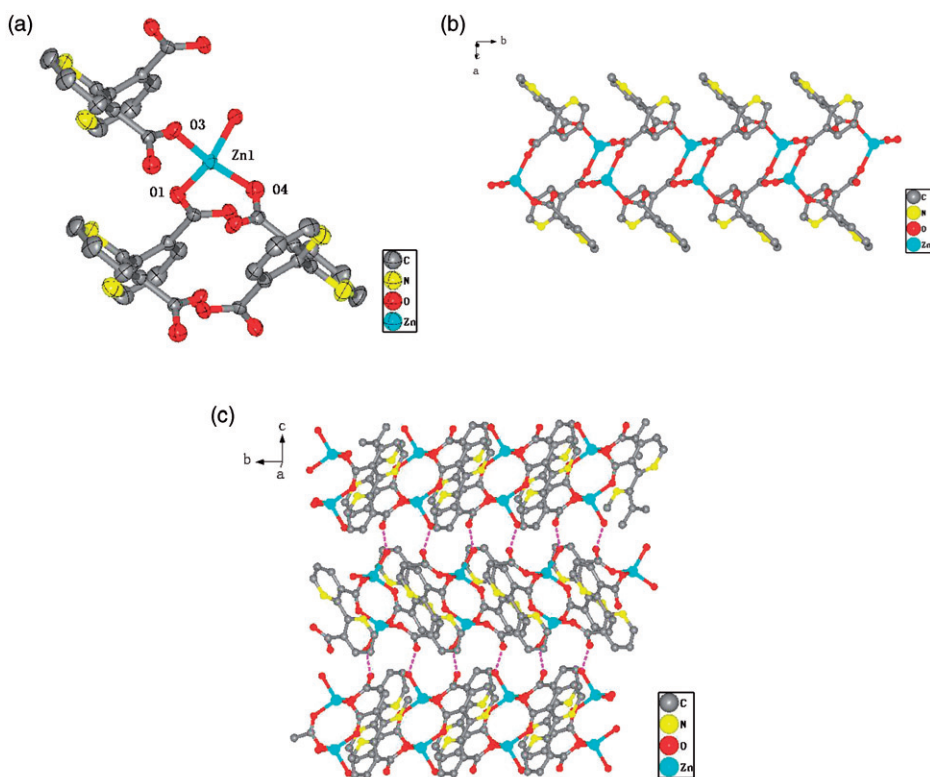


Figure 3. (a) The fundamental unit of **3**, (b) 1-D chain structure of **3**, and (c) 2-D supramolecular layer structure of **3**.

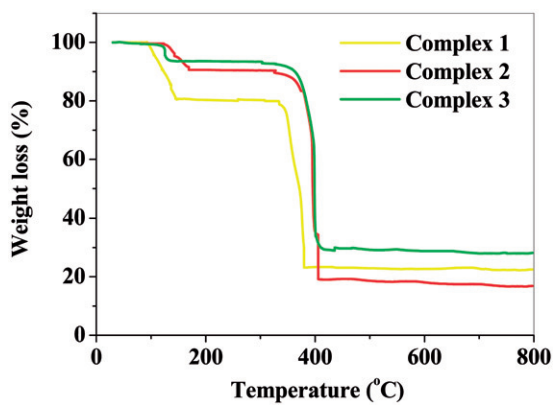


Figure 4. The thermal analysis of **1–3**.

### 3.3. Thermal analysis

In order to examine the stabilities of **1–3**, TGA were carried out in nitrogen from 30°C to 800°C (figure 4). For **1**, the TG curve shows two-step weight loss. The first weight loss of 19.56% from 92 to 150°C corresponds to the loss of one lattice and

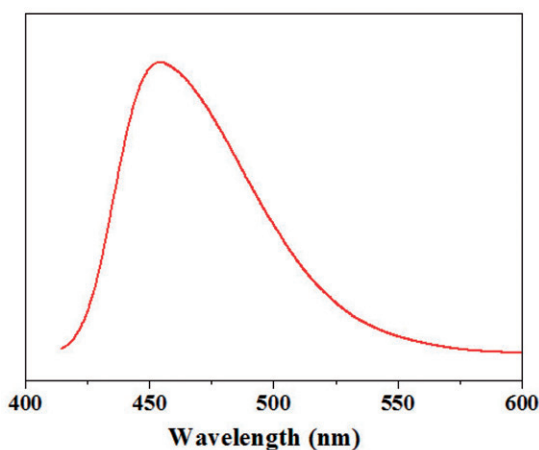


Figure 5. The fluorescence property of **3**.

three-coordinated water molecules (Calcd 19.50%). The second weight loss of 65.54% from 311°C to 380°C corresponds to decomposition of organic ligands (Calcd 65.15%). For **2**, the TG curve shows that the first weight loss of 10.61% from 120°C to 175°C was due to loss of water molecule (Calcd 10.78%). Over the range 323–410°C, the weight loss of 72.55% corresponds to the decomposition of organic ligands (Calcd 72.49%). Complex **3** also displays two-step weight loss of 5.65% from 114°C to 156°C and 74.86% from 338°C to 423°C, respectively, which can be attributed to the loss of guest water molecule (Calcd 5.53%) and organic ligands (Calcd 75.01%), respectively.

### 3.4. Fluorescence

During the past few years, coordination polymers composed of  $d^{10}$  metal centers have been investigated for fluorescence properties with potential applications in photochemistry, chemical sensors, and other aspects. The fluorescence spectrum of **3** was measured at room temperature in the solid state (figure 5). The main emission peak is observed at 452 nm (with  $\lambda_{\text{ex}} = 310$  nm). The emission of **3** can be ascribed to  $\pi-\pi^*$  or  $\pi-n$  transitions of organic ligands [12].

## 4. Conclusions

Three new supramolecular coordination polymers have been synthesized from  $\text{H}_2\text{BPDC}$  and  $\text{Mn(II)}$ ,  $\text{Fe(II)}$ , and  $\text{Zn(II)}$  under hydrothermal conditions. These coordination polymers exhibit 2- and 3-D supramolecular structures.

## Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC reference number 827136 (1), 827137 (2), and 855761 (3).

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