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Coordination polymers constructed from d^{10} ions, saturated or unsaturated aliphatic carboxylic acids, and bidentate nitrogen ligands: synthesis, characterization, and fluorescence

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Coordination polymers constructed from d^{10} ions, saturated or unsaturated aliphatic carboxylic acids, and bidentate nitrogen ligands: synthesis, characterization, and fluorescence

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Five new coordination polymers synthesized from d^{10} metal ions, aliphatic carboxylic acids, and bidentate nitrogen ligands have been characterized with single crystallographic X-ray analysis, IR, and TG. Compound **1** has a twofold 3-D network. During the synthesis of **1**, fumaric acid reacts with water giving malic acid, which coordinates Cd forming **2**. If we use Zn instead of Cd, 1-D chain-like **3** is formed. With hydrogen bonds and π - π interactions, the 1-D covalent chains form a 3-D supramolecular network. The 3-D supramolecular networks connect together and form a twofold interpenetration structure. Compound **4** shows a 2-D layer structure. Compound **5** exhibits 1-D double-chain structure composed by hydrogen bonds. We studied the fluorescence of **1**, **2**, **3**, and **5**.

Keywords: Coordination polymer; Mixed ligands; Fluorescence

1. Introduction

Coordination polymers have attracted attention for structural variety, intriguing physical properties, and promising applications in catalysis, gas absorption, optics, electronics, and magnetism [1–9]. Much effort has been devoted to the synthesis of 1-, 2-, and 3-D coordination polymers [10–15] with studies on coordination mode, functionality, flexibility, and electronic property of organic ligands [16]. Many organic ligands have been used to construct coordination polymers [17–19] with carboxylic acids playing an important role [20–22]. Aromatic carboxylic acids have been widely used in the construction of coordination polymers with interesting structural features and physical properties [23–25]. Saturated and unsaturated carboxylic acids such as 4-cyclohexene-1,2-dicarboxylic, fumaric, and aconitic acids have also been used to construct coordination polymers [26–30]. Furthermore, they possess some obvious merits that aromatic carboxylic acids do not possess: (1) saturated aliphatic dicarboxylates exhibit conformational and coordination versatility due to the

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single-bonded carbon chains; (2) for unsaturated aliphatic dicarboxylates, the C=C double bond is an active point, which can lead to reactions such as conformational transformation, adding with water, giving further structures [31,32]. Coordination polymers constructed from carboxylates and pyridines can incorporate interesting properties of different functional groups [33]. The rational combination of saturated or unsaturated aliphatic acid and bidentate nitrogen ligands can lead to interesting structures with intriguing physical properties.

Coordination polymers constructed from d^{10} metal ions Zn(II), Cd(II), Cu(I), Ag(I), and Au(I) have attracted interest because they exhibit appealing structural features and show fluorescence [34–40]. As Lewis acids, d^{10} metal ions catalyze organic reactions under hydrothermal conditions [41]. Interesting coordination polymers constructed from d^{10} metal ions and mixed ligands have been synthesized [42–46].

On the basis of the aforementioned considerations with saturated or unsaturated aliphatic carboxylic acids, rigid bidentate nitrogen ligands, and d^{10} metal ions, we synthesized five new coordination polymers, $[\text{Cd}(\text{fuma})(4,4'\text{-bipy})]_n$ (**1**), $[\text{Cd}(\text{mal})(4,4'\text{-bipy})(\text{H}_2\text{O})]_n$ (**2**), $[\text{Zn}(\text{fuma})(4,4'\text{-bipy})_2]_n$ (**3**), $[\text{Zn}_3(\text{cit})_2(\text{H}_2\text{O})_2]_n$ (**4**), and $\{[\text{Zn}(1,2\text{-cy})(2,2'\text{-bipy})(\text{H}_2\text{O})_2] \cdot 2\text{H}_2\text{O}\}_n$ (**5**) (fuma = fumaric acid, mal = malic acid, cit = citric acid, 1,2-cy = 4-cyclohexene-1,2-dicarboxylic acid, 2,2'-bipy = 2,2'-bipyridine, 4,4'-bipy = 4,4'-bipyridine). Compounds **1**, **2**, **3**, and **5** exhibit fluorescence at room temperature.

2. Experimental

2.1. Materials and methods

All chemicals purchased were of reagent grade and used without purification. Elemental analyses (C, H, N) were performed on a Perkin-Elmer 2400 CHN elemental analyzer. FT/IR spectra were recorded from 4000 to 400 cm^{-1} on an Alpha Centaur FTIR spectrophotometer using KBr pellets. TG analyses were performed on a Perkin-Elmer TGA7 instrument in flowing N_2 with a heating rate of 10 $^\circ\text{C min}^{-1}$. Photoluminescence spectra were measured using a FL-2T2 instrument (SPEX, USA) with 450 W xenon lamp monochromated by double grating (1200 gr mu^{-1}). All measurements were performed at room temperature.

2.2. Preparation of **1** and **2**

The mixture of $\text{CdCl}_2 \cdot 2.5\text{H}_2\text{O}$ (0.057 g, 0.25 mmol), fumaric acid (0.058 g, 0.50 mmol), 4,4'-bipy (0.078 g, 0.50 mmol) and 8 mL H_2O was stirred for 20 min and the pH was adjusted to 6 with 1 M NaOH. After stirring for another 20 min, the mixture was transferred to a 23 mL Teflon-lined stainless steel bomb and kept at 150 $^\circ\text{C}$ under autogenous pressure for 5 days. The reaction system was cooled to room temperature during 24 h. A large amount of yellow block crystals of **1** were obtained. Yield: 51% (based on Cd); at the same time, colorless crystals of **2** were obtained. Yield: 27% (based on Cd). Elemental Anal. Calcd (%): C, 43.92; H, 2.63; N, 7.32; Found (%): C, 43.86; H, 2.72; N, 7.39 (for **1**). Elemental Anal. Calcd (%): C, 40.15; H, 3.37; N, 6.69; Found (%): C, 40.09; H, 3.32; N, 6.72 (for **2**). IR(cm^{-1}): 3430(vw), 1582(s), 1536(s),

1516(m), 1439(s), 1376(m), 1086(w), 751(m), 713(m), 618(w) (for **1**). IR (cm⁻¹): 3419(s), 1654(s), 1574(s), 1521(s), 1434(s), 1367(s), 719(s), 616(s), 556(w) (for **2**).

Parallel reactions were carried out to discuss the influence of reaction conditions on the synthesis of **1** and **2**. Under acid conditions (pH from 5 to 6), we can get **1** and **2** at the same time. However, under basic conditions (pH from 7 to 8), only **1** was obtained. We also carried out the above reactions under different temperature (from 140°C to 170°C) and found the temperature did not affect **1** and **2**.

2.3. Preparation of **3**

Compound **3** was prepared from a mixture of ZnCl₂·6H₂O (0.061 g, 0.25 mmol), fumaric acid (0.057 g, 0.50 mmol), 4,4'-bipy (0.078 g, 0.50 mmol), and 8 mL H₂O with the pH adjusted to 5 with 1 M NaOH. The mixture was stirred for 20 min then placed in a Teflon-lined stainless steel bomb and heated to 140°C under autogenous pressure for 3 days. The reaction system was then cooled to room temperature during 24 h. Yellow crystals of **3** were obtained. Yield: 62% (based on Zn). Elemental Anal. Calcd (%): C, 58.37; H, 4.08; N, 11.36; Found (%): C, 58.41; H, 3.91; N, 11.44. IR (cm⁻¹): 3152(w), 1618(w), 1521(s), 1471(s), 1441(m), 1344(m), 853(s), 726(s), 642(m), 523(w).

Parallel reactions were carried out to examine the influence of reaction conditions on the synthesis of **3**, which can only be obtained under acidic condition; under basic condition nothing was obtained. This can be attributed to different activity of fumaric acid under various acidities. We also carried out the above reactions under different temperatures, but found temperature did not affect the structure of **3**.

2.4. Preparation of **4**

Compound **4** was prepared from a mixture of ZnCl₂·4H₂O (0.052 g, 0.25 mmol), aconitic acid (0.087 g, 0.50 mmol), 1,10'-phen (0.090 g, 0.50 mmol), and 8 mL H₂O; the pH was adjusted to 5 with 1 M NaOH. The mixture was stirred for 25 min then placed in a Teflon-lined stainless steel bomb and heated to 150°C under autogenous pressure for 4 days. The reaction system was then cooled to room temperature over 24 h. Yellow crystals of **4** were obtained. Yield: 51% (based on Zn). Elemental Anal. Calcd (%): C, 23.69; H, 1.99; Found (%): C, 23.63; H, 1.94. IR (cm⁻¹): 3620(s), 3244(s), 1526(s), 1376(s), 1330(s), 1211(m), 955(w), 844(s), 731(s), 601(m), 528(w), 431(w).

Parallel reactions were carried out to assess the influence of reaction conditions on the synthesis of **4**. The change in reaction time and temperature did not affect the yield of **4**. Under acidic conditions, **4** could be synthesized, but when pH value rose, the yield of **4** decreased.

2.5. Preparation of **5**

Compound **5** was prepared from a mixture of ZnCl₂·4H₂O (0.052 g, 0.25 mmol), 4-cyclohexene-1,2-dicarboxylic acid (0.085 g, 0.50 mmol), 2,2'-bipy (0.078 g, 0.50 mmol), and 8 mL H₂O with the pH adjusted to 4 with 1 M NaOH while stirring. The mixture was placed in a Teflon-lined stainless steel bomb and heated to 160°C under autogenous pressure for 4 days and cooled to room temperature during 24 h. Colorless crystals of **5**

Table 1. Crystal data and structure refinements for 1–5.

	1	2	3	4	5
Empirical formula	C ₁₄ H ₁₀ N ₂ O ₄ Cd	C ₁₄ H ₁₄ N ₂ O ₆ Cd	C ₂₄ H ₂₀ N ₄ O ₄ Zn	C ₆ H ₆ O ₈ Zn _{1.5}	C ₁₈ H ₂₆ N ₂ O ₉ Zn
Formula weight	382.64	426.67	493.81	304.16	479.78
Crystal system	Orthorhombic	Orthorhombic	Monoclinic	Triclinic	Orthorhombic
Space group	<i>C mca</i>	<i>I bma</i>	<i>C 2/c</i>	<i>P-1</i>	<i>P bcn</i>
Unit cell dimensions (Å, °)					
<i>a</i>	11.720(2)	8.4193(17)	18.801(4)	6.4633(13)	15.598(3)
<i>b</i>	15.463(3)	21.862(4)	9.833(2)	7.3145(15)	13.943(3)
<i>c</i>	15.897(3)	22.993(5)	14.744(3)	9.6962(19)	21.272(4)
α	90	90	90	85.20(3)	90
β	90	90	125.93(3)	77.32(3)	90
γ	90	90	90	81.02(3)	90
Volume (Å ³), <i>Z</i>	2880.9(10), 8	4232.2(15), 8	2207.1(8), 4	441.15(15), 2	4626.5(16), 8
Calculated density (g cm ⁻³)	1.764	1.312	1.486	2.290	1.378
Goodness-of-fit on <i>F</i> ²	1.189	1.187	0.994	1.045	1.075
<i>R</i> (int)	0.0384	0.0451	0.0373	0.0290	0.0753
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0396, <i>wR</i> ₂ = 0.1596	<i>R</i> ₁ = 0.0454, <i>wR</i> ₂ = 0.1519	<i>R</i> ₁ = 0.0386, <i>wR</i> ₂ = 0.1193	<i>R</i> ₁ = 0.0286, <i>wR</i> ₂ = 0.0847	<i>R</i> ₁ = 0.0653, <i>wR</i> ₂ = 0.1782

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

were obtained. Yield: 59% (based on Zn). Elemental Anal. Calcd (%): C, 45.06; H, 5.46; N, 5.84; Found (%): C, 45.12; H, 5.52; N, 5.78. IR (cm⁻¹): 3346(s), 3251(s), 1541(s), 1349(s), 1262(m), 1118(m), 817(s), 728(s), 655(w), 583(m), 457(w).

2.6. X-ray crystallography

Single crystals of 1–5 were glued on glass fibers. 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 SHELXTL97 crystallographic software package [47, 48]. Anisotropic thermal parameters were used to refine all non-hydrogen atoms. Carbon-bound hydrogens were placed in calculated positions. Oxygen-bound hydrogens were located in the difference Fourier maps and kept in that position. Further details of the X-ray structural analysis are given in table 1. Selected bond lengths are listed in table 2.

3. Results and discussion

3.1. Structural description

Compound 1 exhibits a 3-D network. In the fundamental unit of 1, there are one Cd, three fumarates, and two 4,4'-bipy, as shown in figure 1(a). The carboxylates from fumarate adopt two coordination modes, one chelating, and the other bridging. Cd(1) is a distorted octahedron coordinated to two nitrogens from 4,4'-bipy ligands, two oxygens from a chelating carboxylate, and another two oxygens from two different bridging carboxylates. Two bridging carboxylates connect two Cds forming a dimer. Each Cd dimer connects with four fumarates and fumarates connect with Cd with covalent bonds forming a 2-D grid-like network with large pores (9.289 × 12.081 Å) as

Table 2. Selected bond lengths [Å] for **1**–**5**.

1			
Cd(1)–O(1)	2.305(7)	Cd(1)–O(2)	2.457(8)
Cd(1)–O(3)#1	2.272(6)	Cd(1)–O(4)#2	2.305(8)
Cd(1)–N(1)	2.307(7)	Cd(1)–N(1)#3	2.307(7)
2			
Cd(1)–O(1)	2.228(5)	Cd(1)–N(1)	2.318(5)
Cd(1)–N(1)#1	2.318(5)	Cd(1)–O(4)	2.331(5)
Cd(1)–O(2)	2.382(4)	Cd(1)–O(6)#2	2.386(5)
3			
Zn(1)–O(1)	2.053(2)	Zn(1)–O(1)#1	2.053(2)
Zn(1)–O(2)	2.308(2)	Zn(1)–O(2)#1	2.308(2)
Zn(1)–N(1)	2.098(2)	Zn(1)–N(1)#1	2.098(2)
4			
Zn(1)–O(3)	2.071(19)	Zn(2)–O(1)#1	1.951(2)
Zn(1)–O(3)#3	2.071(19)	Zn(2)–O(4)#2	1.955(19)
Zn(1)–O(5)#3	2.078(19)	Zn(2)–O(6)	2.010(2)
Zn(1)–O(7)#3	2.117(18)	Zn(2)–O(8)	2.016(2)
Zn(1)–O(7)	2.117(18)		
5			
Zn(1)–O(1)	2.063(3)	Zn(1)–O(3)	2.033(3)
Zn(1)–O(5)	2.137(3)	Zn(1)–O(6)	2.167(4)
Zn(1)–N(1)	2.104(3)	Zn(1)–N(2)	2.107(3)

Symmetry transformations used to generate equivalent atoms for **1**: #1 $x, y-1/2, -z+3/2$; #2 $-x+1, -y+1/2, z-1/2$; #3 $-x+1, y, z$; For **2**: #1 $x, y, -z+1$; #2 $x+1/2, -y+1/2, z$; For **3**: #1 $-x, y, -z+1/2$; For **4**: #1 $-x-1, -y, -z+2$; #2 $-x, -y, -z+1$; #3 $-x-1, -y, -z+1$.

shown in figure 1(b). The most interesting feature of **1** is that two such individual 2-D grid networks are connected by 4,4'-bipy ligands to form a 3-D twofold interpenetration framework as shown in figure 1(c) and 1(d).

During the synthesis of **1**, the fumaric acid reacted with water and produced malic acid, which produced another coordination polymer, [Cd(mal)(4,4'-bipy)(H₂O)]_n (**2**). Single crystal X-ray analysis reveals that **2** also exhibits a 2-D network with one Cd in the fundamental unit, connecting with five oxygens, three coming from two different carboxylates, one from the hydroxyl group, the other from water at distances of 2.229–2.482 Å. Two nitrogens from different 4,4'-bipy ligands occupy the other two coordination sites as shown in figure 2(a). With carboxylate and hydroxyl, the malate links adjacent Cds forming a 1-D chain-like structure. The 4,4'-bipy ligands connect the neighboring 1-D chains forming a 2-D layer structure as shown in figure 2(b).

Unlike **1** and **2**, **3** has a 3-D twofold interpenetrated supramolecular structure. In the fundamental unit there is one octahedral Zn bound to four oxygens (2.053 Å and 2.306 Å) and two nitrogens (2.099 Å) as shown in figure 3(a). In **3**, the fumarate is bis-chelating forming a 1-D chain-like structure. Adjacent chains connect with π – π and hydrogen bond interactions giving the 3-D supramolecular network as shown in figure 3(b). Two such 3-D supramolecular networks connect forming a twofold interpenetration supramolecular network as shown in figure 3(c) and 3(d).

During the synthesis of **4**, we added aconitic acid at the beginning, but in the product, citrate coordinates to Zn. Aconitic acid must react with water-producing citric acid. As shown in figure 4(a), there exist two crystallographically independent Zn's in the fundamental unit. Zn(1) connects two hydroxyl oxygens and four

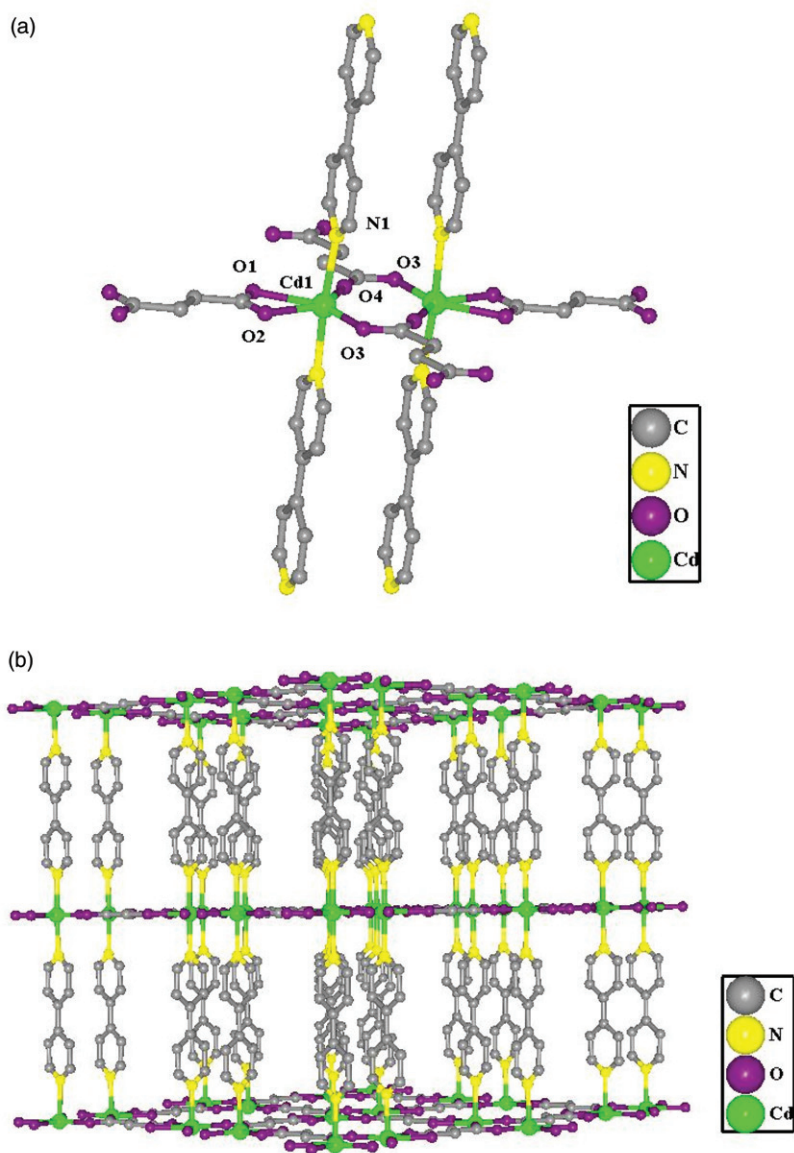


Figure 1. (a) The fundamental unit of **1**; (b) the 3-D network of **1**; (c) the space-filling mode of 3-D network of **1**; and (d) the scheme representation of interpenetrating network of **1**.

carboxylate oxygens from two different citrates. The bond distances range from 2.071 to 2.118 Å and bond angles are from 78.72° to 180°, resulting in a distorted octahedral coordination. Unlike Zn(1), Zn(2) connects with three carboxylate oxygens from three citrates with bond distances from 1.951 to 2.010 Å. The last coordination site is occupied by water at 2.016 Å giving a distorted tetrahedron. In citrate, three carboxylates and one hydroxyl are deprotonated; one carboxylate is monodentate and the other two are bridging. With this kind of linking, the citrate acts as a

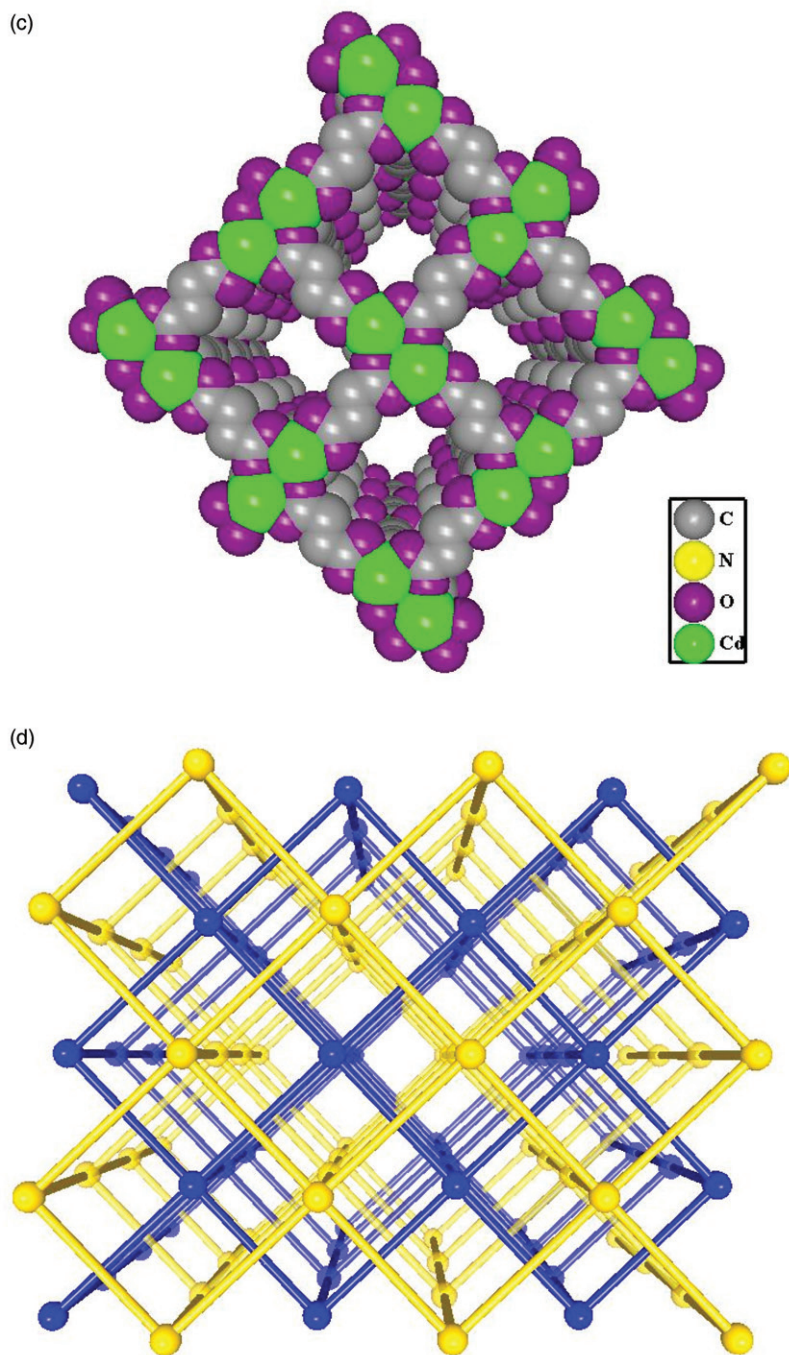


Figure 1. Continued.

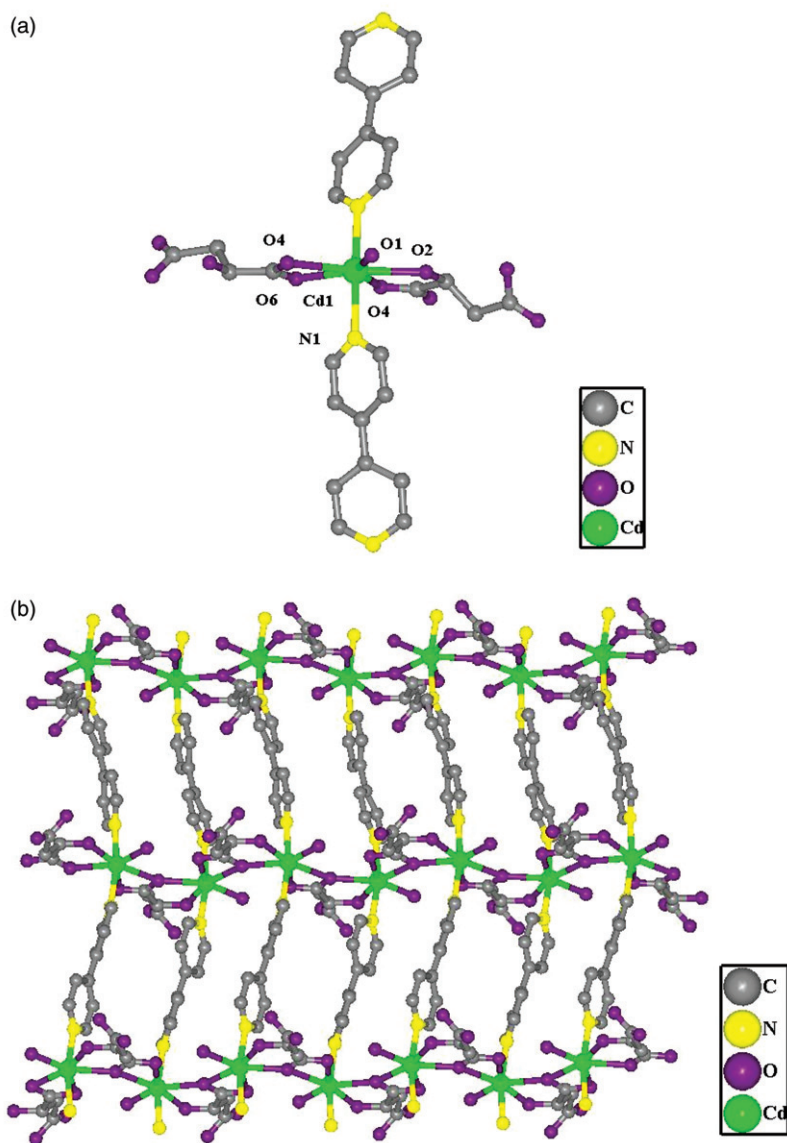


Figure 2. (a) The fundamental unit of **2** and (b) the 2-D layer structure of **2**.

four-connection node, connecting with four Zn's. Citrate connects adjacent Zn's together forming a 1-D ladder, then carboxylates link neighboring ladders together giving a 2-D network as shown in figure 4(b).

Compound **5** has a 1-D chain-like structure with only one crystallographically distinct Zn in the fundamental unit. Zn binds with two carboxylate oxygens from two 1,2-cy ligands, two nitrogens from 2,2'-bipy and two waters as shown in figure 5(a). This results in a distorted octahedral coordination of Zn. Two carboxylates are

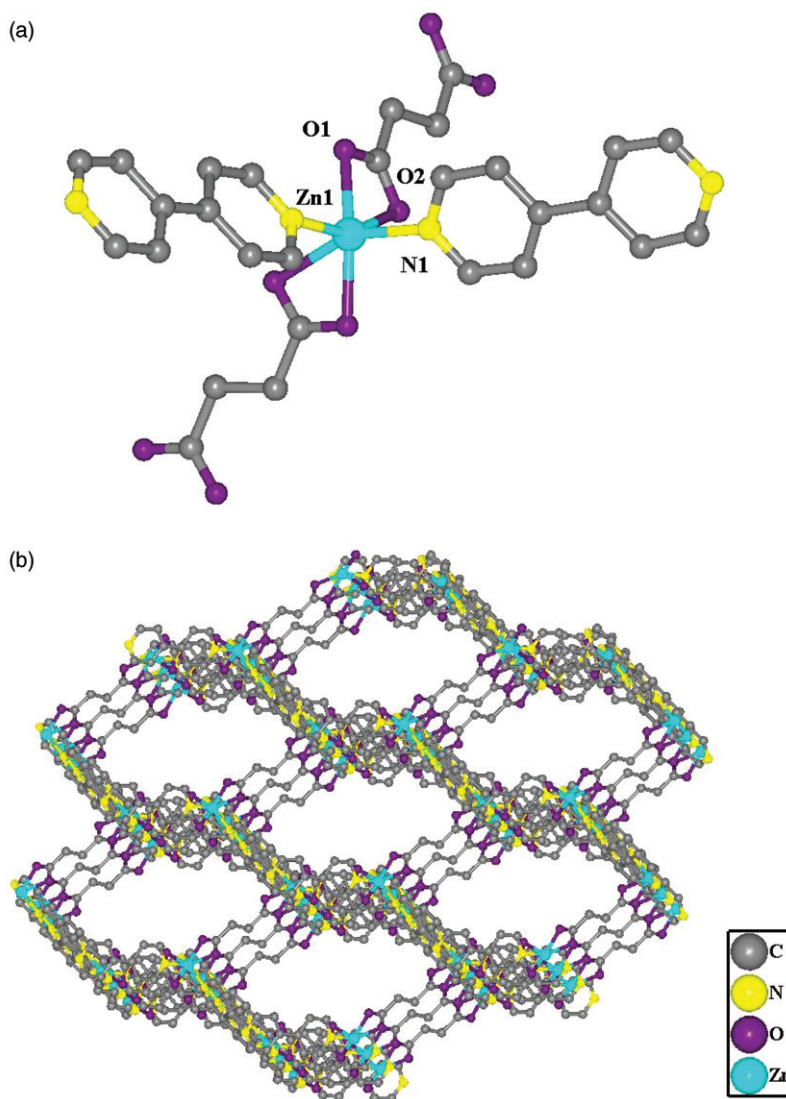


Figure 3. (a) The fundamental unit of **3**; (b) the 3-D supramolecular structure of **3**; (c) the two-fold interpenetration network of **3**; and (d) the interpenetration network of **3**.

monodentate. Adjacent Zn's link forming a 1-D chain-like structure as shown in figure 5(b). Lattice water and coordination water construct hexagon water clusters, connecting adjacent chains together to form a double chain-like structure as shown in figure 5(c).

3.2. Thermal stability

In order to examine thermal stabilities, thermal gravimetric analyses (TGA) were carried out. Compound **1** shows weight loss from 312°C to 407°C with weight loss

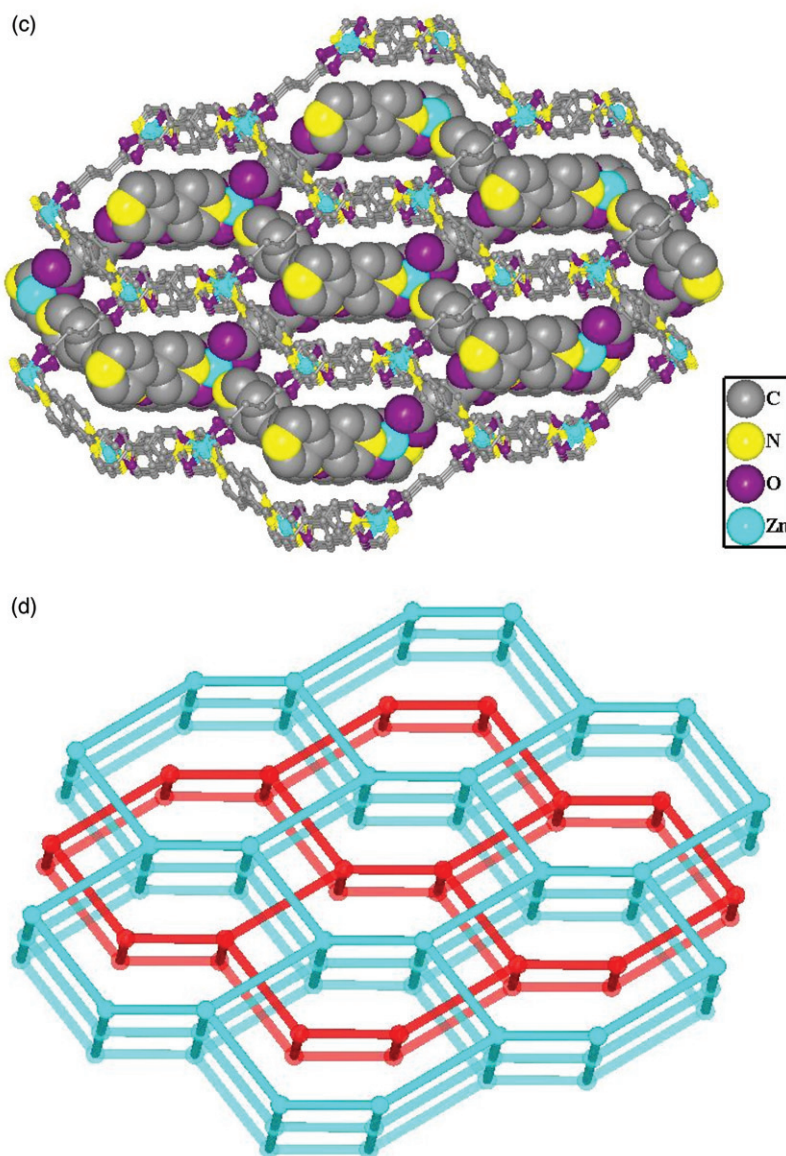


Figure 3. Continued.

of 70.49% equivalent to decomposition of ligands (Calcd 70.63%). Compound **2** displays two-step weight loss, the first weight loss about 4.26% from 113°C to 156°C shows the release of water, and the second about 67.73% from 305°C to 411°C exhibits the loss of organic ligands (Calcd 4.30% and 67.56%). Compound **3** displays one-step weight loss from 326°C to 419°C of 86.51%, equivalent to the loss of organic ligands (Calcd 86.76%). Compound **4** exhibits two weight losses. The first weight of 5.84%

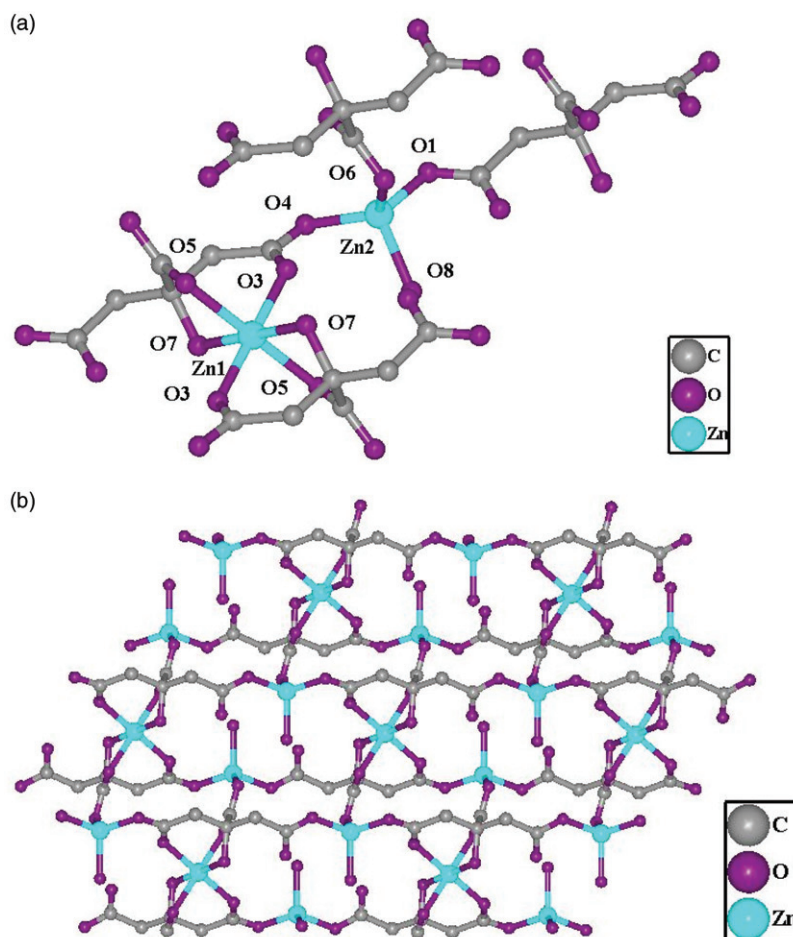


Figure 4. (a) The fundamental unit of **4** and (b) the 2-D covalent network of **4**.

occurs at 106–168°C, corresponding to the removal of water (Calcd 5.92%) and the second of 61.77% from 297°C to 424°C is assigned to the loss of organic ligands (Calcd 61.84%). Compound **5** exhibits continuous weight loss in the range 62–91°C (7.56%), 111–149°C (7.41%), and 285–371°C (67.39%), corresponding to the release of lattice water (Calcd 7.50%), coordinated water (Calcd 7.50%), and organic ligands (Calcd 67.60%).

3.3. Fluorescent property

The fluorescence of **1**, **2**, **3**, and **5** are shown in “Supplementary Material”. The compounds exhibit fluorescence with emission maxima at *ca.* 442, 431, 433, and 412 nm (excitation at 353, 338, 324, and 309 nm), respectively. According to previous reports, these emission bands can be assigned to ligand-to-metal charge transfer (LMCT) emission [49–52].

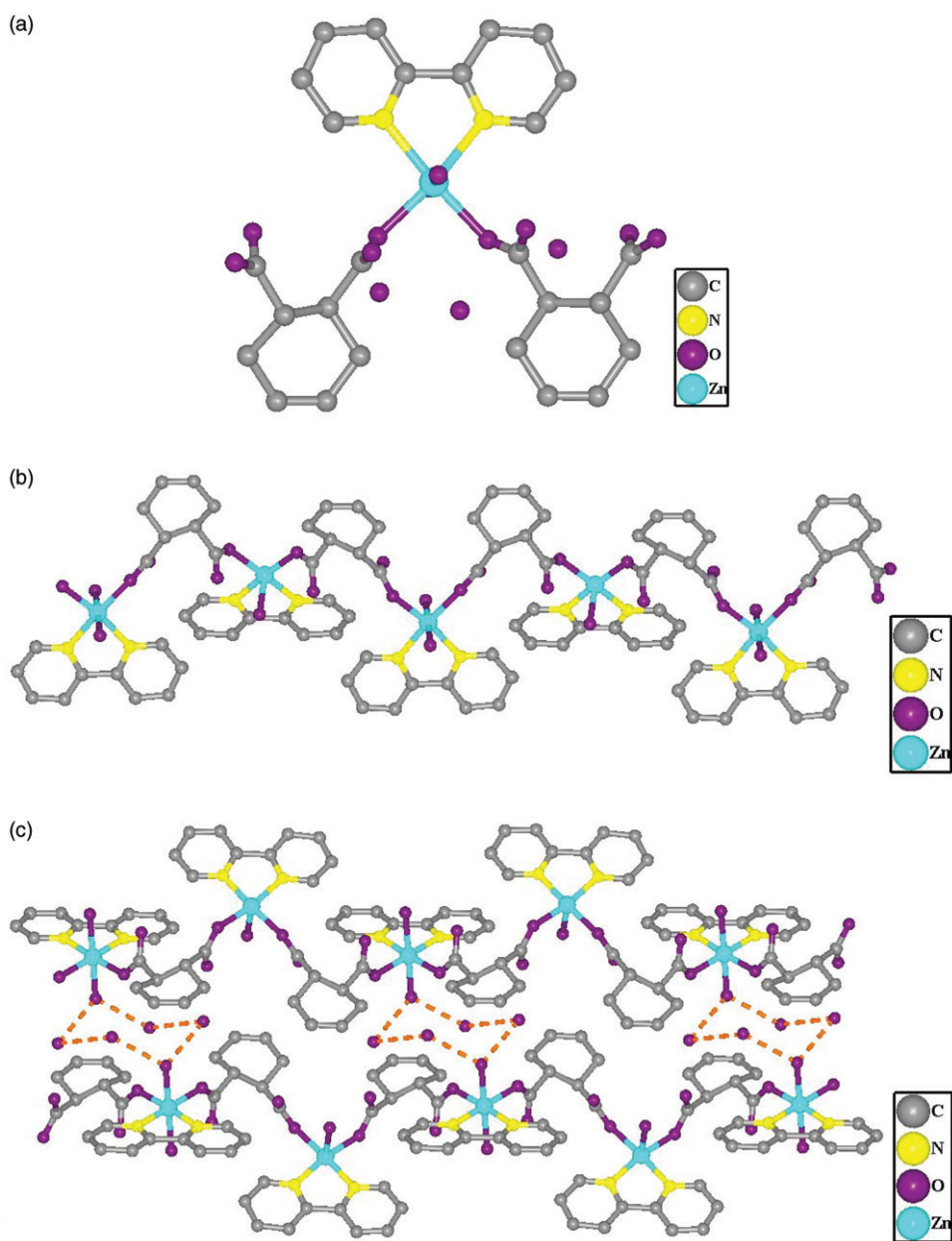


Figure 5. (a) The fundamental unit of **5**; (b) the 1-D covalent network of **5**; and (c) the 1-D double chain structure of **5**.

4. Conclusion

In this article, we combined the merits of d^{10} metal ions, saturated or unsaturated aliphatic carboxylic acids, and bidentate nitrogen ligands and synthesized five new coordination polymers with various structural features.

Supplementary materials

The crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC 734145, 611148, 734146, 721340, and 734147 for **1–5**. These data can be obtained free of charge at <http://www.ccdc.cam.ac.uk/conts/retrieving.html>.

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