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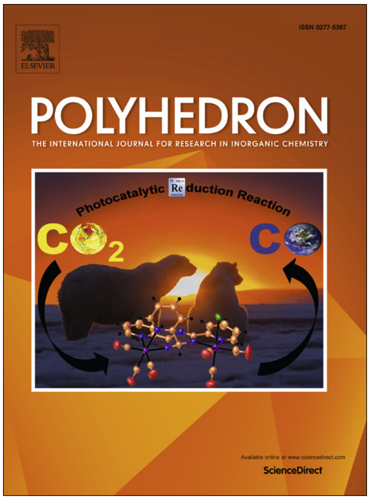

Synthesis, structure and properties of four new coordination polymers based on 2-(4-Chloro-phenoxy)-N-pyridin-3-ylacetamide

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Synthesis, structure and properties of four new coordination polymers based on 2-(4-Chloro-phenoxy)-N-pyridin-3-ylacetamide

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Abstract: A series of coordination polymers, namely, [Zn(L)(HIP)(H_2O)] (**1**), [Cd(L)(HIP)]·1/2H_2O (**2**), [$Co_{1.5}$(L)(BTC)(CH_3OH)(H_2O)$_3$]·2H_2O (**3**), [$Ni_{1.5}$(L)(BTC

)(H_2O)$_4$]·3H_2O (**4**), where L = 2-4-(Chloro-phenoxy)-N-pyridin-3-yl-acetamide, H_2HIP = 5-hydroxyisophthalic acid, H_3BTC = 1,3,5-benzenetricarboxylic acid, have been synthesized under hydrothermal conditions. Single crystal X-ray diffraction analysis reveals that the complexes **1**, **3** and **4** display 1D chain structure. In complex **2**, each Cd_2 unit is linked into BTC form two dimensional layer structures, which are further connected by μ_2-L ligand to construct 3D architecture with $\{4^{24};6^4\}$-**bcu** topology. Moreover, their power X-ray diffraction analysis, thermostability and luminescence properties are explored.

Keywords: Coordination polymers Acylamide Multicarboxylate

Introduction

The construction of coordination polymers has attracted considerable interest during the past decades not only due to their intriguing network topologies but also their potential applications in catalysis[1,2], luminescence[3,4], magnetism[5], gas

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adsorption[6,7], electrochemistry[8,9]. A large number of studies showed that the synthesis of coordination frame works depend on reaction temperature, systematic pH reactants, molar ratio, central metal ion, and the nature of organic ligands[10-14]. Especially properties of organic ligands, such as angle, distance, flexibility, and functional groups, are the key factors for the synthesis of framework of coordination polymers[15-18]. Therefore, utilizing multidentate organic ligands including N or O donors is considered to be an effective strategy to form coordination polymers[19-20].

Acylamide functional ligands as N-donor ligands have been received widespread attention because not only their has the abilities to provide two types of hydrogen bonds sites, the -NH moiety acting as an electron acceptor and the -C=O group acting as an electron donor, which is contribute to the construction of supramolecular structure but also obtained product has a potential biological activity[21-25]. The study of amide complexes including mono-pyridyl-mono-amide[26,27], pyridyl mono-amide[27,28], bis-pyridyl-bis-amide[29-30] and tripyridyl tri-amide[31] has attracted much attentions in recent years.

Multicarboxylate ligands as multidentate O-donor ligands such as 1,4-benzenedicarboxylic acid (1,4-H_2BDC), 1,3,5-Benzenetricarboxylic (1,3,5-H_3BTC) are often selected as multifunctional organic linkers to form the structures of higher dimensions because of their abundant coordination modes and strong coordination ability[32-34]. A great number of 1D-, 2D-, 3D-framework, constructed by combining the carboxylate ligands and N-donor ligands under hydrothermal conditions, were reported[35]. For example, Wang's group reported a

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series bis-pyridyl-bis-amide coordination polymers and their properties including luminescence, electrochemistry and photocatalytic properties in recent years[36-38].

In this study, we chose the 2-(4-Chloro-phenoxy)-N-pyridin-3-yl-acetamide (L) (Scheme **1**) and 5-hydroxyisophthalic acid (H_2HIP), 1,3,5-benzenetricarboxylic acid (H_3BTC) (Scheme **2**) as mixed ligands to react with the transition metal salts and as a result, four new complexes $[Zn(L)(HIP)(H_2O)]$ (**1**), $[Cd(L)(HIP)]\cdot 1/2H_2O$ (**2**), $[Co_{1.5}(L)(BTC)(CH_3OH)(H_2O)_3]\cdot 2H_2O$ (**3**), $[Ni_{1.5}(L)(BTC)(H_2O)_4]\cdot 3H_2O$ (**4**). Their solid state structures, power X-ray diffraction analysis, thermal stability and luminescent properties have been studied.

Experimental

All the reagents and the solvents were purchased commercially and were used directly without further purification. Elemental analysis for C, H and N was carried out on Perkin-Elmer 2400II elemental analyzer. The FT-IR spectrometer fourier transform infrared spectroscopy in the 4000-400 cm^{-1} regions, using KBr pellets. Perkin-Elmer Diamond TG/DTA thermal analyzer was used under atmosphere at a heating rate of 10 K min^{-1} in the temperature range 25-800□ using platinum crucibles. Fluorescence spectra were recorded with F-2500 FL Spectrophotometer analyzer. Powder X-ray diffraction patterns were obtained by using a pinhole camera (Anton Paar) operating with a point focused Ni-filtered Cu Kα radiation in the 2θ range from 5° to 50° with a scan rate of 0.08° per second.

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Synthesis of L

The L ligand was prepared according to the literature method[39]. Elemental Anal. Calc. for $C_{13}H_{11}ClN_2O_2$: C 59.39, H 4.19, N 10.66. Found: C 59.37, H 4.20, N 10.67 %. **IR** (cm^{-1}): 3445 (s), 1684 (s), 1650 (m), 1592 (m), 1537 (s), 1508 (w), 1489 (s), 1434 (m), 1408 (m), 1284 (m), 1235 (s), 1172 (w), 1089 (w), 1067 (m), 822 (m), 710 (m), 664 (m).

Synthesis of [Zn(L)(HIP)(H_2O)] (1)

A mixture of $Zn(NO_3)_2·6H_2O$ (0.0297 g, 0.1 mmol), L (0.0263 g, 0.1 mmol), H_2HIP (0.0182 g, 0.1mmol), water (10 mL), and NaOH (0.010 g, 0.25 mmol) was placed in a 25mL Teflon reactor. The mixture was heated at 120 for 3d, followed by slow cooling to room temperature. The resulting orange crystalline materials were collected. Yield: 42% (based on Zn). Elemental Anal. Calc. for $C_{21}H_{17}ClN_2O_8Zn$: C 47.89, H 3.23, N 5.32; found: C 47.87, H 3.21, N 5.35 %. **IR** (cm^{-1}): 3382 (s), 3256 (s), 3065 (s), 1706 (m), 1629 (m), 1600 (w), 1566 (s), 1489 (s), 1446 (w), 1416 (m), 1364 (m), 1334 (m), 1311 (w), 1292 (w), 1243 (w), 1224 (m), 1127 (w), 1089 (m), 1003 (m), 820 (w), 782 (m), 735 (m) 665 (w).

Synthesis of [Cd(L)(HIP)]·1/2H2O (2)

A similar synthetic procedure was followed as that for **1**. Here, a salt of $3CdSO_4·8H_2O$ (0.0256 g, 0.1mmol) was used. Orange crystalline material **2** were gained. Yield: 40% (based on Cd). Elemental Anal. Calc. for $C_{42}H_{32}Cl_2N_4O_{15}Cd_2$: C 44.66, H 2.84, N 4.96; found: C 44.65, H 2.85, N 4.98 %. **IR** (cm^{-1}): 3440 (s), 1669

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(m), 1609(w), 1541 (s), 1488 (s), 1442 (w), 1421 (w), 1380 (m), 1269(m), 1241(m), 1191 (w), 1128 (w), 1063 (m), 808 (w), 783 (m), 735 (m), 694 (w).

Synthesis of $[Co_{1.5}(L)(BTC)(CH_3OH)(H_2O)_3]\cdot 2H_2O$ (3)

A mixture of $Co(NO_3)_2\cdot 6H_2O$ (0.0291 g, 0.1 mmol), L (0.0263 g, 0.1 mmol), 1,3,5-H_3BTC (0.0210 g, 0.1 mmol), H_2O (2 mL), CH_3OH (2 ml) and NaOH (0.010 g, 0.25 mmol) was heated in a sealed glass tube at 85 ℃ for 3 days, then, the resulting solution was cooled to room temperature, red prism-shaped crystalline material was collected by filtration. Yield: 45% (based on Co). Elemental Anal. Calc. For $C_{46}H_{56}Cl_2N_4O_{28}Co_3$: C 40.57 , H 4.12 , N 4.12; found: C 40.56, H 4.14, N 4.10 %. **IR** (cm^{-1}): 3286 (s), 1668 (m), 1610 (s), 1547 (s), 1489 (m), 1460 (w), 1431 (m), 1372 (s), 1285 (w), 1247 (m), 1168 (w), 1094 (w), 1069 (w), 1010 (m), 827 (w), 808 (w), 761 (m), 715 (m).

Synthesis of $[Ni_{1.5}(L)(BTC)(H_2O)_4]\cdot 3H_2O$ (4)

A similar synthetic procedure was followed as that for **1**. Here, a salt of $Ni(CH_3COO)_2\cdot 4H_2O$ (0.0249 g, 0.1 mmol) was used and H_2HIP was replaced by H_3BTC (0.0210 g, 0.1 mmol). The resulting green crystalline material were collected. Yield: 38% (based on Ni). Elemental Anal. Calc. For $C_{44}H_{42}Cl_2N_4O_{30}Ni_3$: C 38.60, H 4.09, N 4.09; found: C 38.63, H 4.07, N 4.12 %. **IR** (cm^{-1}): 3384 (s), 1666 (m), 1611 (s), 1548 (s), 1530 (s), 1489 (s), 1461 (w), 1430 (s), 1369 (s), 1284 (w), 1246 (m), 1169 (w), 1095 (w), 1070 (w), 1009 (w), 827 (w), 812 (w), 761 (m), 718 (m).

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Description of crystal structures

[Zn(L)(HIP)(H_2O)] **(1)**

Single-crystal X-ray diffraction analysis reveals that complex **1** crystallized in the triclinic space group *P*-1. The crystal structure of complex **1** contains a Zn ion, one L ligands and two HIP anions and one coordinated water. As shown in Fig. 1a, each ion adopts a distorted {$ZnNO_3$} tetrahedral geometry to coordinate with one nitrogen atom from one L ligand with a Zn-N2 distance of 2.047(3) Å, Three oxygen atoms from the two HIP ligands and one coordinated water molecule, with the Zn-O distances of 1.952(3) Å, 2.000(3) Å and 2.011(3) Å, respectively. In **1**, the HIP anion shows a similar “V” shaped bis(monodentate) bridging coordination mode to connect the adjacent Zn(1) ions to form a chain with a Zn···Zn 9.467(5) distance of Å. The adjacent 1D chains are also linked together by the hydrogen bonding interaction between the oxygen atoms of coordinated water molecule and HIP [O(8)-H(8A)···O(2)#4], this forms a ladder-like 2D supramolecular net (Fig. 1b). The final 3D supramolecular framework of **1** is constructed through the hydrogen bonding interactions (O(8)-H(8B)...O(7)#3 = 2.649(4) Å, N(1)-H(1)...O(3)#3 = 2.851(4) Å, O(5)-H(5)...O(4)#5 = 2.800(4) Å) (fig. 1c).

[Cd(L)(HIP)]·1/2H_2O **(2)**

Single crystal X-ray crystallography reveals that complex **2** is a 3D metal -organic coordination polymer. The coordination environment of Cd ion is depicted in Fig. 2a. The Cd ion is coordinated by four oxygen atoms [Cd(1)-O, 2.2086(17) -2.4537(18) Å] from three HIP ligands, which occupy equatorial positions and one

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nitrogen atom (N2A) as well as oxygen atom (O6) from two L ligands [Cd(1)-N(2)#3 = 2.311(2) Å, Cd(1)-O(6) = 2.451(2) Å] , which belong to axial positions, which construct a six-coordinated octahedron environment. As for the HIP ligands, all the hydroxyl groups do not participate in the coordination with Cd ions but the two carboxylate groups of the HIP ligands adopt different coordination fashion to connect Cd ions. One carboxylate group connects to Cd(II) ion, and another carboxylate group adopts a chelate / bridge tridentate coordination mode connecting two Cd(II) ions (Fig. 2b). Two Cd (II) ions are linked by two chelate / bridge carboxylate groups from two HIP to form a Cd_2 unit with a Cd ⋯ Cd separation of 3.738 Å. Different Cd_2 units are connected by μ_2 - HIP resulting in a 2D layer with 4^4-**sql**. Different 2D layers are linked by μ_2 - L ligands to form a 3D architecture (fig. 2c).

Each Cd_2 unit is linked by four ligands and four HIP anions, which can be defined as an 8-connected node. Topologiacal analysis reveals that it is a uninodal 8-connected noninterpenetrated network with $\{4^{24};6^4\}$-**bcu** topology (fig. 2d).

$[Co_{1.5}(L)(BTC)(CH_3OH)(H_2O)_3]\cdot 2H_2O$ **(3)** and $[Ni_{1.5}(L)(BTC)(H_2O)_4]\cdot 3H_2O$ **(4)**

Complex **4** show a similar coordination environment as complex **3**, complex **3** contains one coordinated methanol molecule and there is one coordinated water molecules on the structure of complex **4**. complex **3** and complex **4** crystallizes in the triclinic space group *P*-1, The asymmetric unit of **3** contains one and a half Co(II) atoms, one L ligand, one BTC ligand, three coordinated water molecules, one coordinated methanol molecule and two lattice water molecules. As show in Fig. 3a, there are two crystallographically independent Co(II) centers with same coordination

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environments, the Co1 is six-coordinated by two oxygen atoms from carboxylate groups of two BTC ligand in the apical positions and four coordinated water molecules in the equatorial positions showing a CoO_6 distorted octahedral environment. The Co2 is surrounded by five oxygen atoms of two carboxyl groups from one BTC ligand, one coordinated methanol molecule and one coordinated water molecules as well as one nitrogen atom from L. In **3**, The BTC ligands adopt a μ_3-bridging mode connect with three Co(II) ions to generate a ladder-like chain with the Co1 on the rungs (Co1···Co1 10.352 Å), and the Co2 on the siderails (Co2···Co2 10.352 Å). The ladder-like chain are further connected by hydrogen bonding interaction (O(2W)-H(2W1)...O(8)#5), resulting in a 2D network of complex **3** (Fig. 3b). Finally, coordinated water molecules connect the adjacent layers into 3D supramolecular framework by hydrogen bonding interactions (O(2W)-H(2W2)...O(3)#4, O(2W)-H(2W1)...O(8)#5) (Fig. 3c).

IR spectrum

The strong peaks at 1629 and 1334 cm^{-1} for **1**, 1609 and 1380 cm^{-1} for **2**, 1610 and 1372 cm^{-1} for **3**, 1611 and 1369 cm^{-1} for **4**, may be attributed to the asymmetric and symmetric vibrations of carboxyl groups. The bands around 1706 cm^{-1} for **1** and 1669 cm^{-1} for **2**, 1668 cm^{-1} for **3**, 1666 cm^{-1} for **4**, are characteristic of the carbonyl groups. The presence of the characteristic bands at 1566, 1489, 1446, 1364 cm^{-1} for **1**, 1541, 1488, 1442, 1421 cm^{-1} for **2**, 1547, 1489, 1460, 1431 cm^{-1} for **3**, 1548, 1489, 1461, 1430 cm^{-1} for **4**, suggest the stretching vibrations of the pyridyl rings the

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organic ligands. The bands at 3382 3440 3286 3384 are assigned to stretching and bending vibrations of -OH groups of water molecules for complex **1**-**4**.

Fluorescent properties

The samples for solid-state luminescence of complexes **1-2** and the free ligand **L** have been investigated at room temperature because of their potential photoactive applications[40]. As show in Fig. 5, the L ligand displays an emission band at 312 nm (λ_{ex} = 276 nm), which may be ascribed to the $\pi \rightarrow \pi^*$ or $n \rightarrow \pi^*$ transitions[41]. Under the same excitation conditions, the complex **1** displays a broad and strong emission at 368 nm upon excitation at 273 nm. For complex **2**, it can be observed that emission occurs at 344 nm (λ_{ex} = 271 nm). Thus, compared to L, the emission maxima of **1** and **2** are red-shifted. The most likely emission mechanism is ligand-to-metal charge transfer (LMCT)[42]. The fluorescent intensity of complex **1** is stronger than that of the free ligand, because of the ligand coordinated to the metal center, which enhances its rigidity and thus reduces the loss of energy through radiationless pathways[43]. The strong fluorescence emission of complex **1** show a potential application value in photosensitive material.

Thermogravimetric analyses

Thermal gravimetric analyses (TGA) of coordination complexes **1-4** investigated to exhibit their thermal stabilities. As shown in Fig. 6, the test for complexes **1-4** are carried out at temperature range from 30 to 800 ℃ under an air atmosphere, For 1, the first weight loss, 5.08 %, at 130-175 ℃ is ascribed to a loss of coordinated water molecules (Calc. 3.42 %). The overall framework of **1** begins to collapse at 280 ℃ and

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ends 600 □ with the ZnO residue of 16.13 (Calcd: 15.47%). A first weight loss of 2.11 % for **2** at a region of 140-280 □ is equivalent to the elimination of the one water molecule (Calcd: 1.60 %). The second stage occurs between 350 and 520 □ and corresponds to the release of the organic ligand. The corresponding weight losses are 75.28 % (calcd. 75.64 %). After 520 □, there is no further weight loss. The residues (22.61 %) is CdO (Calcd. 22.76 %). For complex **3**, the first weight loss step from 90-210 □ is attributed to the loss of the lattice water, coordinated water molecules and methanol molecules. The weight loss is about 17.20 % (Calcd. 17.94 %). A second weight loss was observed in a range of 210-550 □, corresponding to the decomposition of organic ligands L and BTC. The CoO residue of **3** of 17.73 % (calcd: 16.52 %). The TG curves of complex **4** exhibit two obvious weight loss steps. The first weight loss of 14.72 % at timeperatures between 80 and 200 □ is assigned to the elimination of the water molecules and coordinated waters (calcd.: 17.76 %). The second weight loss occurred at 270 □. The NiO residue of **4** is 16.68 % (calcd: 16.38 %).

XRD Patterns

The observed and simulated power XRD patterns of the complexes **1-4** are depicted in Fig. 6. It shows that the measured power XRD patterns are in good agreement with the pattern simulated from the X-ray single-crystal data, indicating phase purities of the sample.

Conclusion

In summary, a series of mono-pyridyl-mono-amide coordination polymers were

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prepared under solvothermal conditions and were characterized by IR spectrum, elemental analysis, fluorescent properties, thermogravimetric analysis and Powder X-ray diffraction patterns analysis. The title complexes **1**,**3**,**4** display 1-D coordinated polymer while complex 2 displays 3-D mof structure. The results show that mono-pyridyl-mono-amide is a good candidate for construction coordination polymers with stable and intriguing structures. Moreover, the polycarboxylates also greatly contribute to the structural diversities of the final frameworks. The fluorescent behaviors **1-2** imply that complexes **1** may be good candidates for photoactive materials.

Appendix A. Supplementary data

CCDC 1486621 148662 1486624 1486625 contains the supplementary crystallographic data for **1-4**. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail:deposit@ccdc.cam.ac.uk.

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Table 1 Crystallographic data for polymers **1-4**

Polymer	**1**	**2**	**3**	**4**
Formula	$C_{21}H_{17}ClN_2O_8Zn$	$C_{42}H_{32}Cl_2N_4O_{15}Cd_2$	$C_{46}H_{56}Cl_2N_4O_{28}Co_3$	$C_{44}H_{42}Cl_2N_4O_{30}Ni_3$
Formula weight	526.19	1128.42	1360.64	1353.85
Crystal system	Triclinic	Orthorhombic	Triclinic	Triclinic
Space group	*P-1*	*Pbcn*	*P-1*	*P-1*
a (Å)	9.467(5)	13.3967(19)	10.352(5)	10.218(5)
b (Å)	10.054(6)	18.103(3)	10.788(5)	10.777(5)
c (Å)	12.908(7)	17.019(2)	13.633(7)	13.625(6)
α (°)	68.421(7)	90	112.299(6)	112.644(5)
β (°)	86.363(7)	90	100.196(6)	100.317(5)
γ (°)	69.873(7)	90	93.932(5)	94.339(5)
$D_{calc.}$ (Mg·m^{-3})	1.633	1.816	1.638	1.672
Z	2	4	1	1
Absorption coefficient (mm^{-1})	1.325	1.239	1.090	1.238
F(000)	536	2248	699	692
Crystal size (mm^3)	0.19 x 0.18 x 0.17	0.21 x 0.20 x 0.18	0.14 x 0.13 x 0.12	0.12 x 0.11 x 0.10
θ range (°)	1.70 to 26.00	1.89 to 28.56	1.65 to 25.00	1.66 to 25.00
Reflections collected	5964	25230	7323	7310
Unique reflections	4070 [R(int) = 0.0406]	4960 [R(int) = 0.0249]	4746 [R(int) = 0.0173]	4679 [R(int) = 0.0187]
Completeness to *θ* = 25.00	96.7 %	94.2 %	98.2 %	98.7 %
Max. and min. transmission	0.8061 and 0.7868	0.8078 and 0.7809	0.8804 and 0.8624	0.8862 and 0.8656
Data / restraints / parameters	4070 / 0 / 307	4960 / 0 / 299	4746 / 17 / 413	4679 / 2 / 384
GOF on F^2	1.066	1.106	1.138	1.099
R indices [$I > 2\sigma(I)$]	R1 = 0.0652, wR2 = 0.1683	R1 = 0.0278 , wR2 = 0.0743	R1 = 0.0316, wR2 = 0.0945	R1 = 0.0319, wR2 = 0.0941
R indices (all data)	R1 = 0.0758, wR2 = 0.1761	R1 = 0.0339 , wR2 = 0.0776	R1 =0.0357 , wR2 = 0.0966	R1 = 0.0350, wR2 = 0.1011
Largest diff. peak and hole (e.Å^{-3})	1.797 and -1.476	0.880 and-0.317	0.388 and -0.306	0.751 and -0.389

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Table 2 Selected bond lengths (Å) and angles (°) for **1-4**

1			
Zn(1)-O(6)#1	1.952(3)	O(6)#1-Zn(1)-C(21)#1	28.45(11)
Zn(1)-O(3)	2.000(3)	O(3)-Zn(1)-C(21)#1	137.07(11)
Zn(1)-O(8)	2.011(3)	O(8)-Zn(1)-C(21)#1	105.73(11)
Zn(1)-N(2)	2.047(3)	N(2)-Zn(1)-C(21)#1	111.08(12)
O(6)-Zn(1)#2	1.952(3)	C(14)-O(3)-Zn(1)	114.6(2)
O(6)#1-Zn(1)-O(3)	108.77(11)	C(21)-O(6)-Zn(1)#2	104.4(2)
O(6)#1-Zn(1)-O(8)	113.69(13)	C(13)-N(2)-Zn(1)	116.2(3)
O(3)-Zn(1)-O(8)	96.25(11)	C(12)-N(2)-Zn(1)	124.8(3)
O(6)#1-Zn(1)-N(2)	131.12(13)	O(7)-C(21)-Zn(1)#2	74.0(2)
O(3)-Zn(1)-N(2)	99.85(12)	O(6)-C(21)-Zn(1)#2	47.11(17)
O(8)-Zn(1)-N(2)	101.19(13)		
2			
Cd(1)-O(3)#1	2.2086(17)	O(4)-Cd(1)-O(6)	80.82(8)
Cd(1)-O(4)	2.2870(17)	O(5)#2-Cd(1)-O(6)	100.55(8)
Cd(1)-O(5)#2	2.2895(16)	N(2)#3-Cd(1)-O(6)	167.49(8)
Cd(1)-N(2)#3	2.311(2)	O(3)#1-Cd(1)-O(5)	158.69(7)
Cd(1)-O(6)	2.451(2)	O(4)-Cd(1)-O(5)	54.73(6)
Cd(1)-O(5)	2.4537(18)	O(5)#2-Cd(1)-O(5)	76.04(6)
O(3)-Cd(1)#4	2.2086(17)	N(2)#3-Cd(1)-O(5)	99.97(7)
O(5)-Cd(1)#2	2.2895(16)	O(6)-Cd(1)-O(5)	80.55(7)
N(2)-Cd(1)#5	2.311(2)	C(21)-O(3)-Cd(1)#4	102.30(14)
O(3)#1-Cd(1)-O(4)	136.85(6)	C(14)-O(4)-Cd(1)	96.03(14)
O(3)#1-Cd(1)-O(5)#2	92.35(6)	C(14)-O(5)-Cd(1)#2	167.74(16)
O(4)-Cd(1)-O(5)#2	130.06(6)	C(14)-O(5)-Cd(1)	87.94(13)
O(3)#1-Cd(1)-N(2)#3	98.15(8)	Cd(1)#2-O(5)-Cd(1)	103.95(6)
O(4)-Cd(1)-N(2)#3	89.25(7)	C(8)-O(6)-Cd(1)	155.1(2)
O(5)#2-Cd(1)-N(2)#3	91.66(7)	C(13)-N(2)-Cd(1)#5	114.14(17)
O(3)#1-Cd(1)-O(6)	84.19(7)	C(12)-N(2)-Cd(1)#5	127.49(18)
3			
Co(1)-O(5)	2.0751(18)	O(5)#1-Co(1)-O(2W)	89.11(7)
Co(1)-O(5)#1	2.0751(18)	O(1W)-Co(1)-O(2W)	93.77(8)
Co(1)-O(1W)	2.0849(18)	O(1W)#1-Co(1)-O(2W)	86.23(8)
Co(1)-O(1W)#1	2.0849(19)	O(5)-Co(1)-O(2W)#1	89.11(7)
Co(1)-O(2W)	2.1400(19)	O(5)#1-Co(1)-O(2W)#1	90.89(7)
Co(1)-O(2W)#1	2.1400(19)	O(1W)-Co(1)-O(2W)#1	86.23(8)
Co(2)-O(1)#2	2.0615(17)	O(1W)#1-Co(1)-O(2W)#1	93.77(8)
Co(2)-O(3W)	2.064(2)	O(2W)-Co(1)-O(2W)#1	180.0
Co(2)-O(7)	2.0912(18)	O(1)#2-Co(2)-O(3W)	89.75(7)
Co(2)-O(4)	2.1343(17)	O(1)#2-Co(2)-O(7)	94.57(7)
Co(2)-N(2)	2.151(2)	O(3W)-Co(2)-O(7)	89.98(7)
Co(2)-O(3)	2.2151(18)	O(1)#2-Co(2)-O(4)	104.63(7)
O(1)-Co(2)#3	2.0615(17)	O(3W)-Co(2)-O(4)	95.02(7)

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O(5)-Co(1)-O(5)#1	180.0	O(7)-Co(2)-O(4)	160.15(6)
O(5)-Co(1)-O(1W)	89.85(7)	O(1)#2-Co(2)-N(2)	90.34(7)
O(5)#1-Co(1)-O(1W)	90.15(7)	O(3W)-Co(2)-N(2)	178.42(7)
O(5)-Co(1)-O(1W)#1	90.15(7)	O(7)-Co(2)-N(2)	91.59(7)
O(5)#1-Co(1)-O(1W)#1	89.85(7)	O(4)-Co(2)-N(2)	83.43(7)
O(1W)-Co(1)-O(1W)#1	180.0	O(1)#2-Co(2)-O(3)	164.46(6)
O(5)-Co(1)-O(2W)	90.89(7)	O(3W)-Co(2)-O(3)	87.59(7)
O(4)-Co(2)-O(3)	60.41(6)	O(7)-Co(2)-O(3)	100.74(6)
		N(2)-Co(2)-O(3)	91.91(7)
4			
Ni(1)-O(7)#1	2.0285(19)	O(3)-Ni(2)-O(9)	89.70(8)
Ni(1)-O(7)	2.0285(19)	O(10)-Ni(2)-O(9)	88.81(8)
Ni(1)-O(11)	2.0645(19)	O(3)-Ni(2)-N(2)	91.53(8)
Ni(1)-O(11)#1	2.0645(19)	O(10)-Ni(2)-N(2)	91.91(8)
Ni(1)-O(12)#1	2.099(2)	O(9)-Ni(2)-N(2)	178.50(8)
Ni(1)-O(12)	2.099(2)	O(3)-Ni(2)-O(6)#2	101.70(8)
Ni(2)-O(3)	2.0237(18)	O(10)-Ni(2)-O(6)#2	161.96(7)
Ni(2)-O(10)	2.045(2)	O(9)-Ni(2)-O(6)#2	93.42(8)
Ni(2)-O(9)	2.053(2)	N(2)-Ni(2)-O(6)#2	85.49(8)
Ni(2)-N(2)	2.078(2)	O(3)-Ni(2)-O(5)#2	162.44(7)
Ni(2)-O(6)#2	2.0957(19)	O(10)-Ni(2)-O(5)#2	101.01(7)
Ni(2)-O(5)#2	2.1791(19)	O(9)-Ni(2)-O(5)#2	87.20(7)
O(5)-Ni(2)#3	2.1791(19)	N(2)-Ni(2)-O(5)#2	91.36(7)
O(6)-Ni(2)#3	2.0957(19)	O(6)#2-Ni(2)-O(5)#2	61.29(7)
O(7)#1-Ni(1)-O(7)	180.0	O(3)-Ni(2)-C(21)#2	132.38(8)
O(7)#1-Ni(1)-O(11)	89.33(7)	O(10)-Ni(2)-C(21)#2	131.39(8)
O(7)-Ni(1)-O(11)	90.67(7)	O(9)-Ni(2)-C(21)#2	92.48(8)
O(7)#1-Ni(1)-O(11)#1	90.67(7)	N(2)-Ni(2)-C(21)#2	86.06(8)
O(7)-Ni(1)-O(11)#1	89.33(7)	O(6)#2-Ni(2)-C(21)#2	30.68(7)
O(11)-Ni(1)-O(11)#1	180.000(1)	O(5)#2-Ni(2)-C(21)#2	30.74(7)
O(7)#1-Ni(1)-O(12)#1	90.75(8)	C(14)-O(3)-Ni(2)	126.48(15)
O(7)-Ni(1)-O(12)#1	89.25(8)	C(21)-O(5)-Ni(2)#3	87.50(13)
O(11)-Ni(1)-O(12)#1	86.64(9)	C(21)-O(6)-Ni(2)#3	91.35(15)
O(11)#1-Ni(1)-O(12)#1	93.36(9)	C(22)-O(7)-Ni(1)	132.10(16)
O(7)#1-Ni(1)-O(12)	89.25(8)	C(10)-N(2)-Ni(2)	122.01(17)
O(7)-Ni(1)-O(12)	90.75(8)	C(11)-N(2)-Ni(2)	119.93(18)
O(11)-Ni(1)-O(12)	93.36(9)	O(6)-C(21)-Ni(2)#3	57.97(12)
O(11)#1-Ni(1)-O(12)	86.64(9)	O(5)-C(21)-Ni(2)#3	61.76(12)
O(12)#1-Ni(1)-O(12)	180.000(1)	C(19)-C(21)-Ni(2)#3	171.66(17)
O(3)-Ni(2)-O(10)	96.20(8)		

Symmetry transformations used to generate equivalent atoms: for Polymer **1:** #1 x+1,y,z #2 x-1,y,z; for Polymer **2:** #1 -x+1/2,-y+1/2,z-1/2 #2 -x+1,y,-z+1/2 #3 -x+1/2,y-1/2,z #4 -x+1/2,-y+1/2,z+1/2 #5 -x+1/2,y+1/2,z; for Polymer **3:** #1 -x+1,-y,-z+1 #2 x+1,y,z #3 x-1,y,z #4 x,y-1,z #5 -x+2,-y+1,-z+2 #6 -x+1,-y+1,-z+1 #7 -x,-y+1,-z+1 #8 x-1,y,z-1; for Polymer **4:** #1 -x+1,-y+2,-z+1 #2 x+1,y,z #3 x-1,y,z #4 x,y+1,z #5 -x+1,-y+1,-z

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#6 x+1,y-1,z   #7 -x+1,-y+1,-z+1 #8 -x+2,-y+1,-z+1.

Table 3 Hydrogen-bond geometry (Å) for polymers **1-4**

D-H···A	D-H	H···A	D···A	D-H···A	Symmetry codes
Polymer **1**					
O(8)-H(8B)...O(7)#3	0.89(8)	1.84(8)	2.649(4)	152(7)	#1 x+1,y,z; #2 x-1,y,z
O(8)-H(8A)...O(2)#4	0.80(5)	1.96(5)	2.739(4)	165(5)	#3 -x+1,-y+2,-z
N(1)-H(1)...O(3)#3	0.86	1.99	2.851(4)	177.8	#4 x,y+1,z
O(5)-H(5)...O(4)#5	0.82	1.99	2.800(4)	170.7	#5 -x+1,-y+2,-z+1
Polymer **2**					
N(1)-H(1A)...O(7)	0.86	2.08	2.551(3)	113.8	#1 -x+1/2,-y+1/2,z-1/2
					#2 -x+1,y,-z+1/2
O(1)-H(1)...O(2)#6	0.82	2.40	3.171(3)	157.7	#3 -x+1/2,y-1/2,z
O(1)-H(1)...O(4)#7	0.82	2.47	2.903(3)	114.1	#4 -x+1/2,-y+1/2,z+1/2
					#5 -x+1/2,y+1/2,z
O(8)-H(8A)...O(2)#6	1.03(7)	2.03(7)	3.049(4)	172(7)	#6 -x+1,y,-z+3/2
					#7 x+1/2,-y+1/2,-z+1
Polymer **3**					
O(1W)-H(1W1)...O(2)#4	0.848(10)	1.883(12)	2.720(3)	169(3)	#1 -x+1,-y,-z+1
O(1W)-H(1W2)...O(6)	0.846(10)	1.939(13)	2.742(3)	158(3)	#2 x+1,y,z
O(2W)-H(2W1)...O(8)#5	0.853(10)	2.161(11)	3.011(3)	175(3)	#3 x-1,y,z
O(2W)-H(2W2)...O(3)#4	0.842(10)	2.019(10)	2.860(2)	177(3)	#4 x,y-1,z
O(3W)-H(3W1)...O(6)#6	0.842(10)	1.918(10)	2.758(3)	177(3)	#5 -x+2,-y+1,-z+2
O(3W)-H(3W2)...O(5W)#2	0.843(10)	1.921(11)	2.761(3)	174(3)	#6 -x+1,-y+1,-z+1
O(4W)-H(4W1)...O(5W)#7	0.861(10)	2.052(15)	2.896(4)	166(4)	#7 -x,-y+1,-z+1
O(4W)-H(4W2)...O(6)	0.859(10)	2.348(14)	3.198(3)	170(4)	
O(5W)-H(5W1)...O(4)#6	0.849(10)	1.930(10)	2.779(3)	178(4)	
O(5W)-H(5W2)...O(8)#8	0.843(10)	2.418(19)	3.218(4)	159(4)	
O(7)-H(7)...O(2)#2	0.848(10)	1.821(13)	2.644(2)	163(3)	
N(1)-H(1)...O(4W)#2	0.850(10)	2.247(16)	3.052(3)	158(3)	
Polymer **4**					
O(13)-H(13D)...O(15)#3	0.86(2)	2.15(10)	2.903(5)	147(16)	#1 -x+1,-y+2,-z+1
O(12)-H(12E)...O(5)#4	0.85	2.07	2.919(3)	177.5	#2 x+1,y,z
O(12)-H(12D)...O(2)#5	0.85	2.12	2.970(3)	177.6	#3 x-1,y,z
O(11)-H(11C)...O(4)#4	0.85	1.86	2.705(3)	174.3	#4 x,y+1,z
O(10)-H(10D)...O(14)#6	0.85	1.89	2.738(4)	176.0	#5 -x+1,-y+1,-z
O(9)-H(9D)...O(13)#7	0.85	1.93	2.779(3)	178.9	#6 x+1,y-1,z
O(9)-H(9C)...O(8)#8	0.85	1.98	2.826(3)	178.8	#7 -x+1,-y+1,-z+1
O(13)-H(13C)...O(6)	0.84(2)	1.89(3)	2.713(3)	167(7)	#8 -x+2,-y+1,-z+1
N(1)-H(1)...O(15)	0.86	2.34	3.138(4)	154.6	
N(1)-H(1)...O(1)	0.86	2.09	2.535(3)	111.7	
O(11)-H(11D)...O(7)	0.85	2.44	2.911(3)	115.5	
O(11)-H(11D)...O(8)	0.85	1.83	2.676(3)	174.5	
O(10)-H(10E)...O(3)	0.85	2.53	3.028(3)	118.9	
O(10)-H(10E)...O(4)	0.85	1.81	2.657(3)	176.2	

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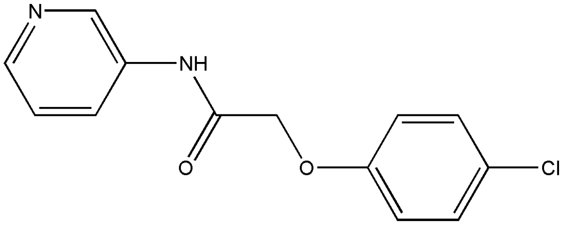
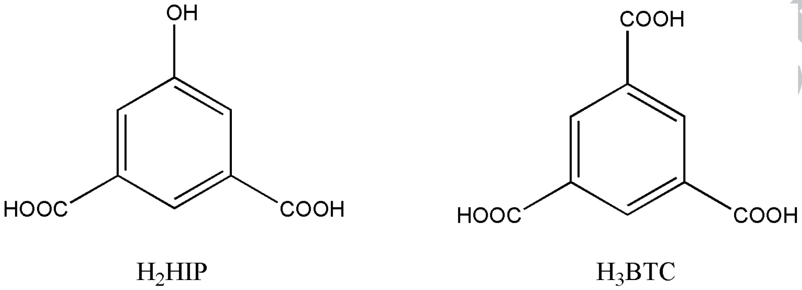


Scheme **1**. Structure of the L = 2-(4-Chloro-phenoxy)-N-pyridin-3-yl-acetamide (L) ligand.



H_2HIP H_3BTC

Scheme **2**. Structrue of the carboxylic acids in this work.

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Figure captions:

Fig. 1. (a) Coordination environment around the Zn(II) centers in **1**. All the hydrogen atoms are omitted for clarity; (b) two-dimensional polymeric frameworks of polymer **1**; (c) The 3D structure of polymer **1**.

Fig. 2. (a) Coordination environment around the Cd(II) centers in **2**. All the hydrogen atoms are omitted for clarity; (b) The layer constructed by Cd(II) atoms and HIP ligands; (c) The 3D structure of polymer **2**; (d) The$\{4^{24};6^{4}\}$-**bcu** topology of polymer **2**.

Fig. 3. (a) Coordination environment around the Co(II) centers in **3**. All the hydrogen atoms are omitted for clarity; (b) two-dimensional supramolecular framework of polymer **3**; (c) The 3D supramolecular framework of polymer **3**.

Fig. 4. The solid-state fluorescence spectra of complexes **1-2** and the free ligand **L** at room temperature.

Fig. 5. The TG curves for polymers **1-4**.

Fig. 6. XRD patterns of the complexes **1-4**.

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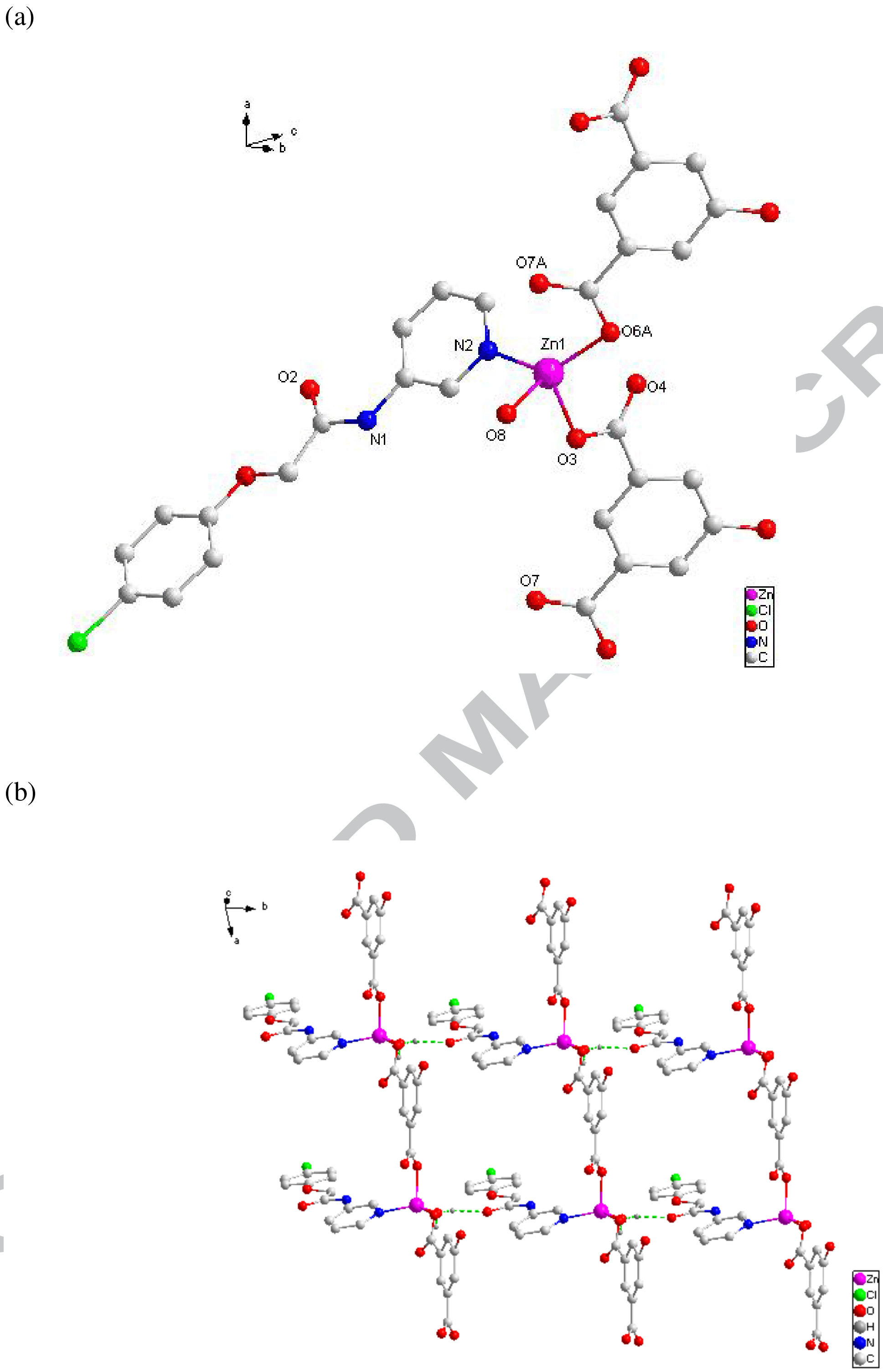

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(c)

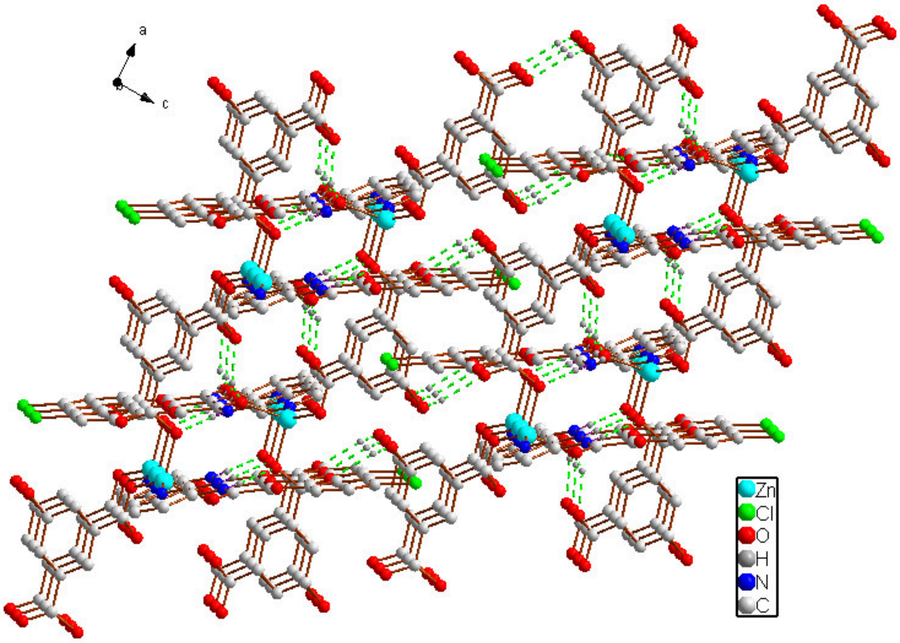
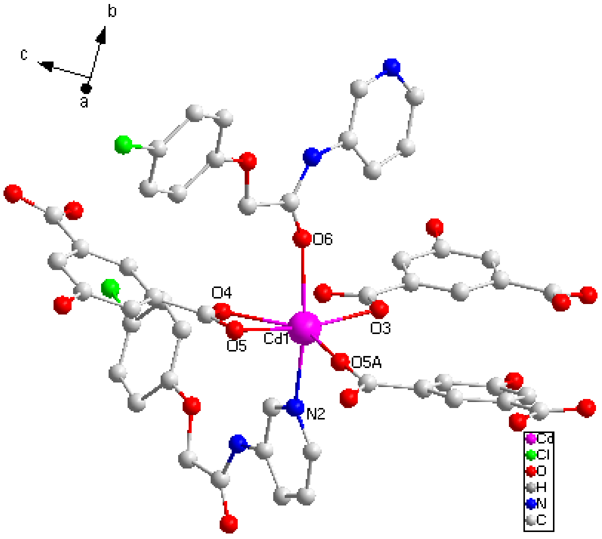


Fig. 1. (a) Coordination environment around the Zn(II) centers in **1**. All the hydrogen atoms are omitted for clarity; (b) two-dimensional supramolecular framework of polymer **1**; (c) The 3D supramolecular framework of polymer **1**.

(a)



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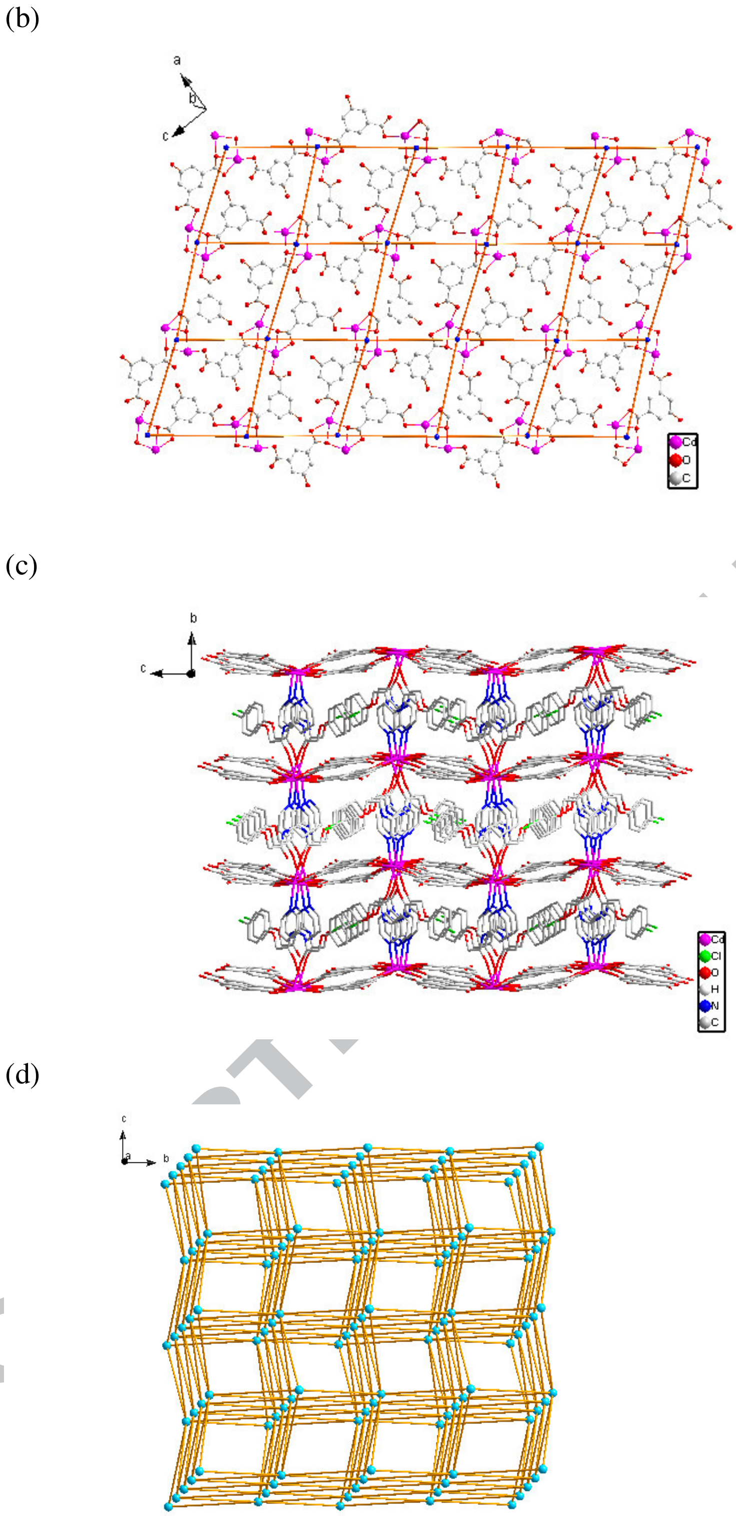

Fig. 2. (a) Coordination environment around the Cd(II) centers in **2**. All the hydrogen atoms are omitted for clarity; (b) The layer constructed by Cd(II) atoms and HIP ligands; (c) The 3D structure of polymer **2**; (d) The$\{4^{24};6^{4}\}$-**bcu** topology of polymer **2**.

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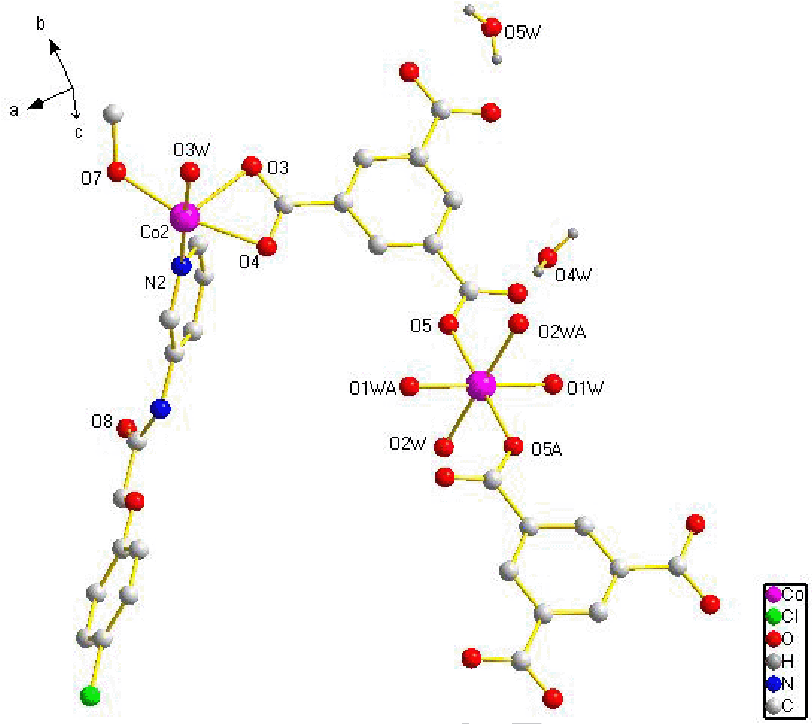
(a)



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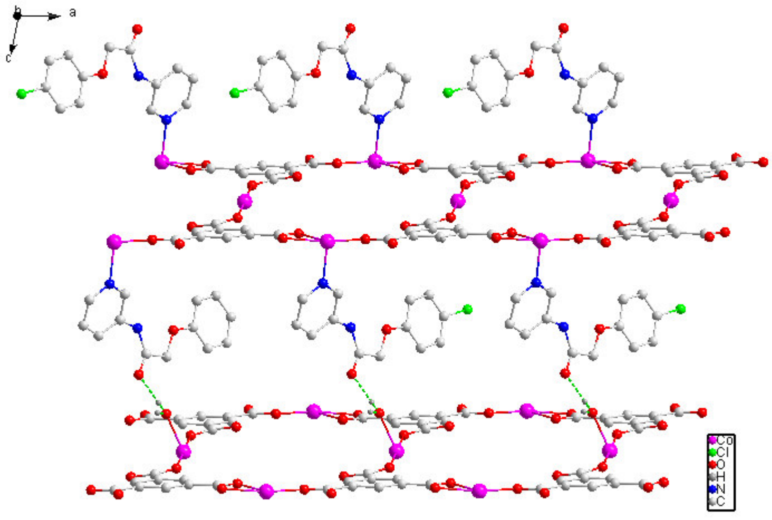

(c)

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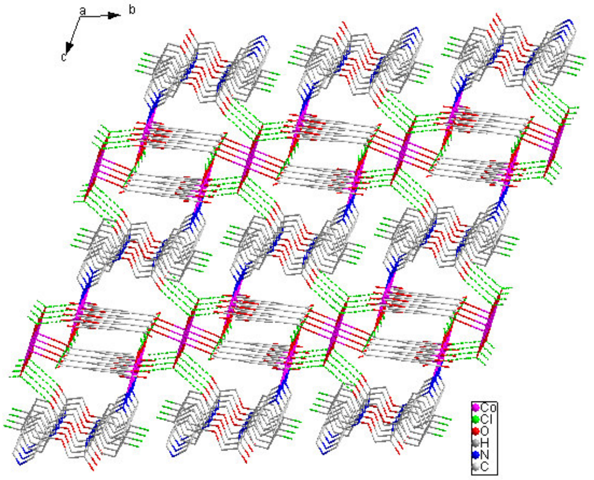

Fig. 3. (a) Coordination environment around the Co(II) centers in **3**. All the hydrogen atoms are omitted for clarity; (b) two-dimensional supramolecular framework of polymer **3**; (c) The 3D supramolecular framework of polymer **3**.

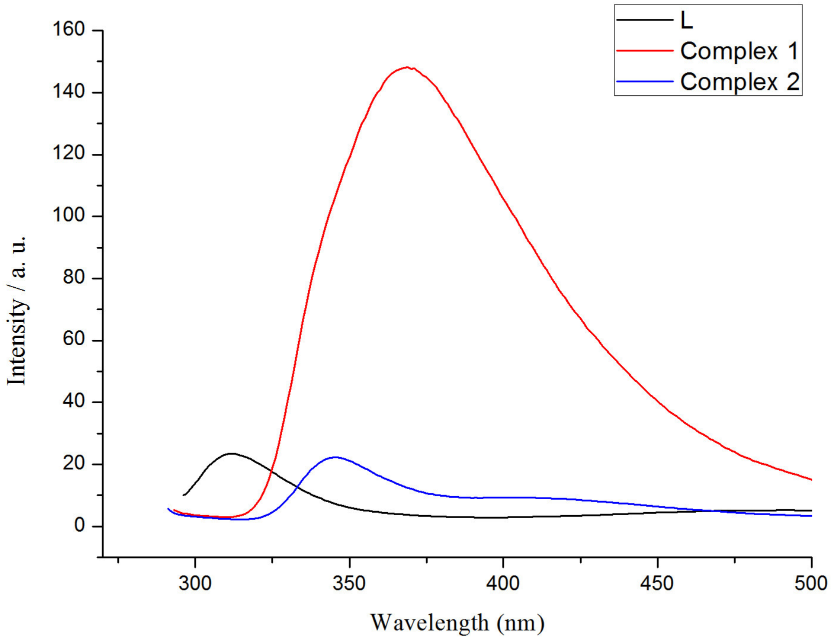

Fig. 4. The solid-state fluorescence spectra of complexes **1-2** and the free ligand **L** at room temperature.

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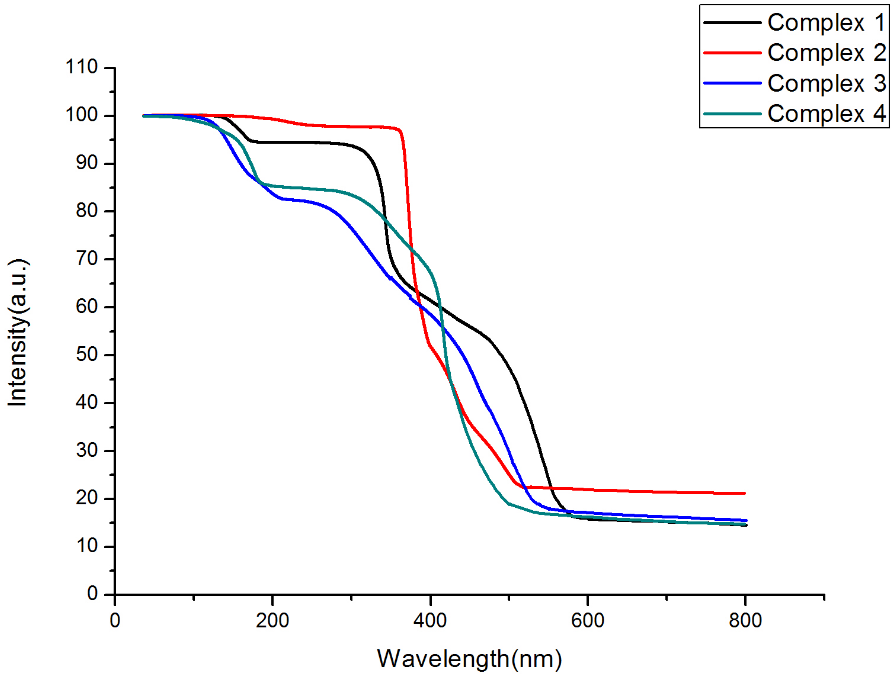

Fig. 5. The TG curves for polymers **1-4**.

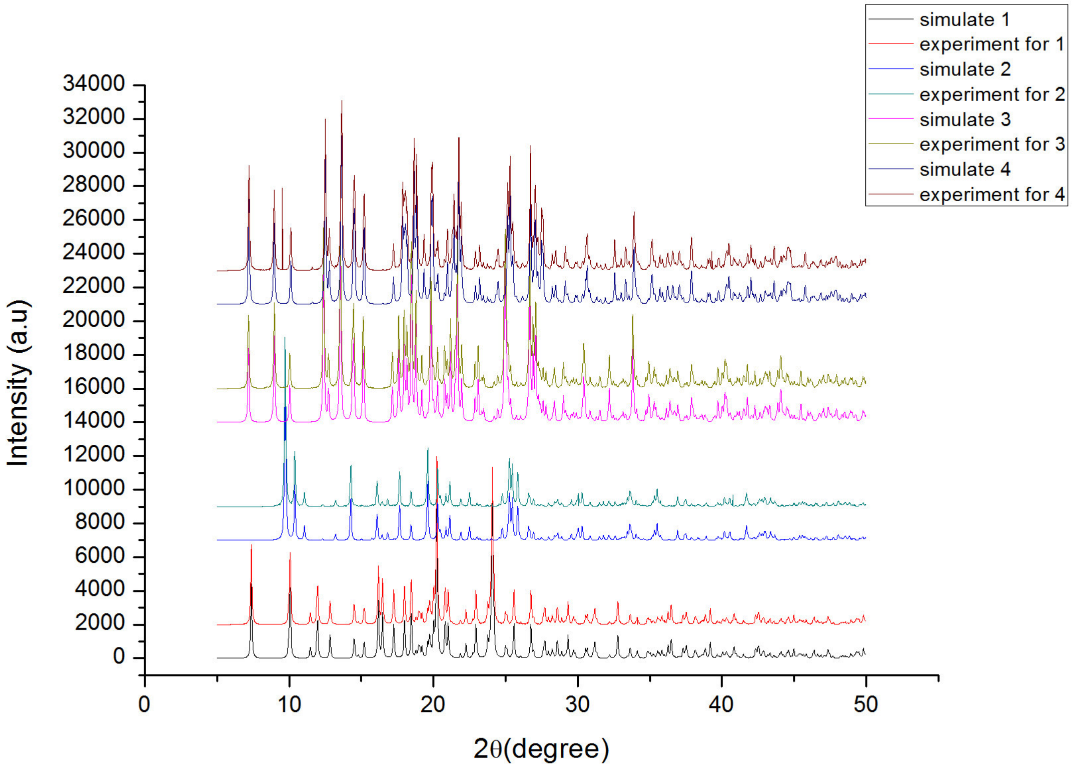

Fig. 6. XRD patterns of the complexes **1-4**.

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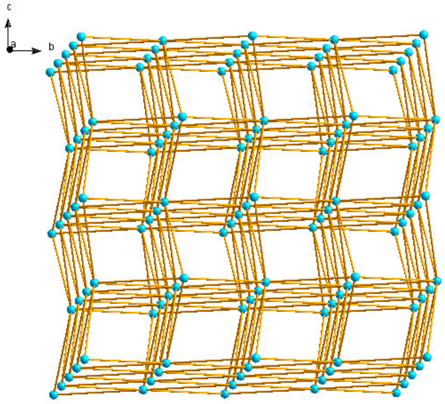

A series of coordination polymers, namely, [Zn(L)(HIP)(H_2O)] (**1**), [Cd(L)(HIP)]·1/2H_2O (**2**), [$Co_{1.5}$(L)(BTC)(CH_3OH)(H_2O)$_3$]·2H_2O (**3**), [$Ni_{1.5}$(L)(BTC)(H_2O)$_4$]·3H_2O (**4**), where L = 2-4-(Chloro-phenoxy)-N-pyridin-3-yl-acetamide, H_2HIP = 5-hydroxyisophthalic acid, H_3BTC = 1,3,5-benzenetricarboxylic acid, have been synthesized under hydrothermal conditions. Single crystal X-ray diffraction analysis reveals that the complexes **1**, **3** and **4** display 1D chain structure. In complex **2**, each Cd_2 unit is linked into BTC form two dimensional layer structures, which are further connected by μ_2-L ligand to construct 3D architecture with $\{4^{24};6^4\}$-**bcu** topology.

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