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Syntheses and Luminescence of Four Supramolecular Coordination Complexes with Flexible Ligand[1]

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Abstract—Four d^{10}-based complexes with chemical formulae {$[Zn(L^1)_2(H_2O)_2(4,4'\text{-Bipy})_2]$ (**I**), {$[Zn_2(L^1)_4(Mi)] \cdot 4H_2O$} (**II**), {$[Zn(L^1)_2(Phen)] \cdot H_2O$} (**III**) {$[Cd(L^1)_2(Phen)] \cdot 2H_2O$} (**IV**) ($HL^1$ = *p*-hydroxy phenylacetic acid, 4,4'-Bipy = 4,4'-bipyridine, Phen = 1,10-phenanthroline, Mi = 1,4-bis(imidazol-1-yl)butane) have been synthesized and structurally characterized by single crystal X-ray diffraction (CIF files CCDC nos. 1047119 (**I**), 1047120 (**II**), 1047121 (**III**), 1047122 (**IV**)). The significant effect of assistant ligands and metal ions on assembly of **I**–**IV** has been demonstrated, which leads to the formation of distinct crystalline products. Complexes **I**–**IV** show various coordination motifs with different existing forms and coordination modes of the organic ligands. Furthermore, extend supramolecular networks are connected by secondary interactions such as hydrogen-bonding and aromatic stacking. The thermal stability and luminescent properties of the compounds were discussed in detail.

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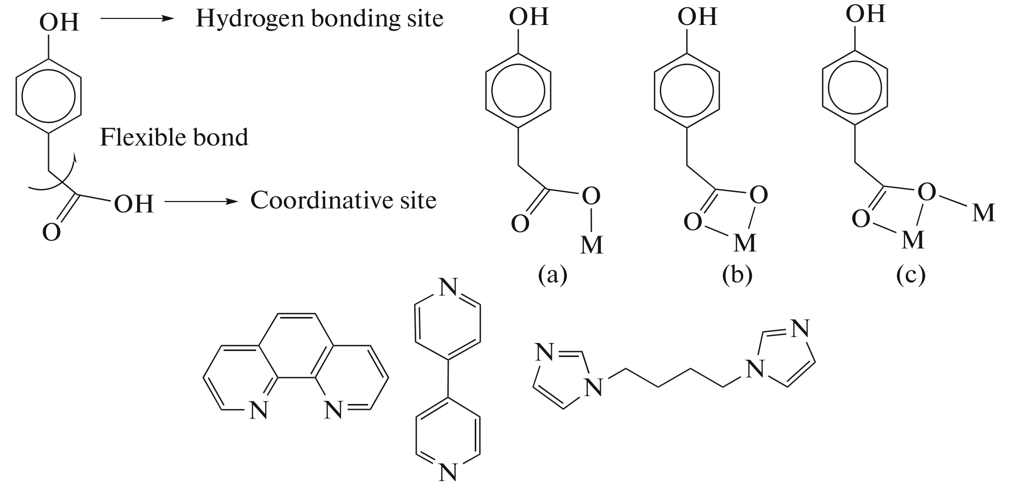
INTRODUCTION

Coordination polymers have attracted intense attention in recent years because of their intriguing structures and potential applications as functional materials [1–3]. Many efforts have been paid to the rational design of MOFs for the specific needs of applications. A conventional strategy of using long *exo*-multidentate ligands has been successful to construct frameworks with porosity [4]. Moreover, the flexible carboxylic acids are good candidates for the construction of new coordination polymers as the carboxyl groups can form C–O–M–O cyclic mode with central metal ions, thus, improving the stability of transition metal complexes [4]. With the above in mind, we chose *p*-hydroxy phenylacetic acid (HL^1) as ligand to construct CPs based on the following considerations: (1) HL^1, as a derivative of the phenylacetic acid ligand, is a good spacer and has been rarely used in the assembly of coordination polymers; (2) the hydroxyl group may take as potential coordinative site, which may induce higher metal cluster [5]; (3) since the hydroxyl group are always actively involved in H-bonding interaction, which may stabilize the full networks and enrich the supramolecular chemistry. Taking account of the above, we would like to synthesize and explore new complexes with HL^1, N-donors and d^{10} metal ions. The organic ligands in this work are in the Scheme:



Scheme.

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On the other hand, the combination of different ligands can result in greater modulation of structural frameworks than single ligand [5–8]. Thus, mixed-ligands are undoubtedly a good choice for the construction of new polymeric structures. Furthermore, the coordination compounds with flexible ligands exhibit more complex structural feature due to the characteristics of flexible ligands [9–13]. We anticipated that increase architecture complexity may be introduced by using chelating or/and bridging aromatic N-donor linkers and there is an opportunity to drive the new modes of network assembly required to satisfy the unique constraints imposed by linker geometries. The reactions of HL^1 with Zn(II)/Cd(II) and three well-known organic linkers Mi, Phen and 4,4'-Bipy under mild conditions resulted the formation of four new complexes with different motifs. These compounds are characterized by single crystal X-ray diffraction. Furthermore, the luminescent properties of compounds **I–IV** were discussed in detail.

EXPERIMENTAL

Materials and method. All reagents were purchased from commercial sources and used as received. IR spectra were recorded with a Perkin-Elmer Spectrum One spectrometer in the region 4000–400 cm^{-1} using KBr pellets. TGA was carried out with a Metter-Toledo TA 50 in dry dinitrogen (60 mL min^{-1}) at a heating rate of 5°C min^{-1}. X-ray powder diffraction (XRPD) data were recorded on a Rigaku RU200 diffractometer at 60 kV, 300 mA for CuK_α radiation (λ = 1.5406 Å) with a scan speed of 2°C/min and a step size of 0.013° in 2θ. Luminescence spectra for crystal solid samples were recorded at room temperature on an Edinburgh FLS920 phosphorimeter.

Syntheses of complex I. A mixture of $Zn(OAc)_2 \cdot 2H_2O$ (0.0212 g), HL^1 (0.0245 g), 4,4'-Bipy (0.0152 g), CH_3OH (5 mL) and deionised water (5 mL) was stirred for 30 min in air. The resulting solution was kept at room temperature for two weeks, the crystals formed were filtered off, washed with water and dried in air.

For $C_{36}H_{34}N_4O_8Zn$ (M = 716.04)

anal. calcd., %:	C, 60.38;	H, 4.79;	N, 7.82.
Found, %:	C, 60.02;	H, 4.71;	N, 7.68.

IR (KBr; ν, cm^{-1}): 3392 v.s, 3049 m, 1595 v.s, 1503 m, 1435 m, 1355 v.s, 1218 v.s, 1132 m, 1069 m, 807 m, 619 m.

Complex **II** was synthesized by the same method used for **I** except that 4,4’-Bipy was replaced by Mi (0.0216 g).

For $C_{52}H_{56}N_8O_{16}Zn_2$ (M = 1187.85)

anal. calcd., %:	C, 52.58;	H, 5.43;	N, 9.43.
Found, %:	C, 52.41;	H, 3.32;	N, 9.52.

IR (KBr; ν, cm^{-1}): 3135 v.s, 3060 m, 1600 v.s, 1429 m, 1400 v.s, 1230 v.s, 1098 m, 944 v.s, 813 v.s, 653 m.

Complex **III** was synthesized by the same method used for **I** except that 4,4'-Bipy was replaced by Phen (0.0115 g).

For $C_{28}H_{24}N_2O_7Zn$ (M = 565.86)

anal. calcd., %:	C, 59.43;	H, 4.27;	N, 4.95.
Found, %:	C, 59.30;	H, 4.30;	N, 4.92.

IR (KBr; ν, cm^{-1}): 3306 v.s, 3009 m, 1588 v.s, 1503 v, 1427 v, 1372 v.s, 1223 v.s, 1012 v, 847 v.s, 716 v, 522 m.

Complex **IV** was synthesized by the same method used for **III** except that $Zn(OAc)_2 \cdot 2H_2O$ was replaced by $Cd(OAc)_2 \cdot 2H_2O$ (0.0156 g).

For $C_{28}H_{26}N_2O_8Cd$ (M = 630.91)

anal. calcd., %:	C, 53.30;	H, 4.15;	N, 4.44.
Found, %:	C, 53.19;	H, 5.06;	N, 4.32.

IR (KBr; ν, cm^{-1}): 3438 v.s, 3032 m, 1549 v.s, 1509 v.s, 1412 v.s, 1235 v.s, 1092 v, 847 m, 727 v.s, 630 m, 511 m.

X-ray crystallography. Single crystal X-ray diffraction analysis of the title compounds were carried out on a Bruker SMART APEX II CCD diffractometer equipped with a graphite monochromated MoK_α radiation (λ = 0.71073 Å) by using ϕ/ω scan technique at room temperature. Data were processed using the Bruker SAINT package and the structures solution and the refinement procedure was performed using SHELX-97 [14]. The structure was solved by direct methods and refined by full-matrix least-squares fitting on F^2. The hydrogen atoms of organic ligands were placed in calculated positions and refined using a riding on attached atoms with isotropic thermal parameters 1.2 times those of their carrier atoms. The hydrogen atoms of lattice water molecule in compound **II** were not located using the different Fourier method. Table 1 shows crystallographic data of **I–IV**. Selected bond distances and bond angles are listed in Table 2. Some H-bonded parameters are listed in Table 3. Supplementary material has been deposited with the Cambridge Crystallographic Data Centre (CCDC nos. 1047119 (**I**), 1047120 (**II**), 1047121 (**III**), 1047122 (**IV**); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

Table 1. Crystal data and structure refinement information for compounds **I** and **II**

Parameter	Value	
	I	**II**
Formula weight	716.06	1179.83
Crystal system	Monoclinic	Triclinic
Space group	$P2_1/c$	$P\bar{1}$
Crystal color	Colorless	Colorless
a, Å	5.7118(4)	9.223(16)
b, Å	7.7411(6)	16.487(3)
c, Å	36.162(3)	18.545(3)
α, deg	90	78.847(3)
β, deg	94.003(7)	81.400(3)
γ, deg	90	80.984(3)
V, Å^3	1595.0(2)	2711.7(8)
Z	2	2
ρ_{calcd}, g/cm^3	1.491	1.445
μ, mm^{-1}	0.832	0.961
F(000)	744	1224
θ Range, deg	2.86–25.19	1.27–25.30
Reflection collected	9493	14101
Independent reflections (R_{int})	9493 (0.0728)	14101 (0.0272)
Reflections with $I > 2\sigma(I)$	2263	6515
Number of parameters	229	707
R_1, wR_2 ($I > 2\sigma(I)$)*	0.0685, 0.1548	0.0515, 0.1517
R_1, wR_2 (all data)*	0.0946, 0.1682	0.0843, 0.1746

* $R = \Sigma(F_o - F_c)/\Sigma(F_o)$; $wR_2 = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma(F_o^2)^2\}^{1/2}$.

RESULTS AND DISCUSSION

The results of crystallographic analysis revealed that the asymmetric unit of complex **I** contains one crystallographically unique Zn atom, two L^1 ligands, two 4,4'-Bipy ligands and two coordinative water molecules. As shown in Fig. 1a, the Zn atom is coordinated by four oxygen atoms from two symmetric monodentate carboxylic group of L^1 ligands and two coordinative water molecules, and two N atoms from two symmetric 4,4'-Bipy ligands, completing a octahedral geometry. The Zn–O/N distance are in the normal ranges and are agreed with the reported Zn polymers [15].

In addition, careful analysis reveals that the hydrogen bonded interaction association of water molecules, phenolic groups, benzene rings and carboxylate groups in **I** leads to the formation of 2D supramolecular network (Fig. 1b and Table 3). The oxygen atom (O(4)) of phenolic group could take as donor to bind with N(1) from pyridyl ring of 4,4'-Bipy. Also, the O(2) taking as donor involves in O(1) of carboxylate group, which stabilize the full network. It should be noted that there is not any packing interaction between rings from adjacent 4,4'-Bipy molecules.

To investigate the effect of the assistant ligand on the complex networks, the 4,4'-Bipy was deliberately replaced by the more flexible mi ligand. A new compound **II** was obtained. Complex **II** displays a 1D structure. The structure of **II** contains one Zn^{2+} ion, two L^1 ligands, half Mi ligand and two lattice water molecules. The Zn atom in the is coordinated by two oxygen atoms from two different carboxylic group of L^1 ligands and two N atoms from one bridging Mi ligand, completing a tetrahedral geometry (Fig. 2a and Table 2). The two carboxylic groups show monodentate mode. The adjacent Zn(II) centers are linked by the Mi spacers to form a 1D zigzag array along the *xy* plane and the L^1 ligands are decorated at the sides of the chain, as shown in Fig. 2b.

To investigate the influence of the terminal ligand on the complex networks, the bridging spacers was deliberately replaced by the aromatic and chelating

Table 2. Selected bond distances (Å) and angles (deg) for **I** and **II**

Bond	*d*, Å	Bond	*d*, Å
I			
Zn(1)–O(1)	2.088(3)	Zn(1)–O(2)	2.154(3)
Zn(1)–N(5)	2.169(4)		
II			
Zn(1)–O(7)	2.088(3)	Zn(1)–O(10)	1.977(3)
Zn(1)–N(4)	2.169(4)	Zn(1)–N(7)	2.019(3)
Zn(2)–O(2)	1.979(3)	Zn(2)–O(5)	2.009(4)
Zn(2)–N(1)	1.999(4)	Zn(2)–N(5)	1.998(3)
III			
Zn(1)–O(2)	1.962(2)	Zn(1)–O(4)	2.011(2)
Zn(1)–O(5)	2.314(3)	Zn(2)–N(1)	2.064(3)
Zn(1)–N(2)	2.089(3)		
IV			
Cd(1)–O(1)	2.637(8)	Cd(1)–O(2)	2.279(6)
Cd(1)–O(4)	2.463(8)	Cd(1)–O(5)	2.337(7)
Cd(1)–N(1)	2.364(7)	Cd(1)–N(2)	2.293(7)
Angle	ω, deg	Angle	ω, deg
I			
O(1)Zn(1)O(2)	87.66(12)	O(1)Zn(1)N(5)	89.40(12)
O(2)Zn(1)N(5)	87.64(13)	O(1)Zn(1)O(2*A*)	92.34(12)
O(1)Zn(1)N(5*A*)	90.60(12)	O(1*A*)Zn(1)O(2)	92.34(12)
O(2)Zn(1)N(5*A*)	92.36(13)	O(1*A*)Zn(1)N(5)	90.60(12)
II			
O(7)Zn(1)O(10)	114.14(14)	O(7)Zn(1)N(4)	99.66(16)
O(7)Zn(1)N(7)	113.22(14)	O(10)Zn(1)N(4)	107.62(14)
O(10)Zn(1)N(7)	96.94(14)	N(4)Zn(1)N(7)	105.73(14)
O(2)Zn(2)O(5)	94.13(15)	O(2)Zn(2)N(1)	97.85(14)
O(2)Zn(2)N(5)	126.61(14)	O(5)Zn(2)N(1)	132.83(14)
O(5)Zn(2)N(5)	98.87(15)	N(1)Zn(2)N(5)	109.51(14)
III			
O(2)Zn(1)O(4)	130.16(8)	O(2)Zn(1)O(5)	97.94(9)
O(2)Zn(1)N(1)	109.89(9)	O(2)Zn(1)N(2)	106.19(9)
O(4)Zn(1)O(5)	59.95(8)	O(4)Zn(1)N(1)	112.88(8)
O(4)Zn(1)N(2)	104.96(9)	O(5)Zn(1)N(1)	88.03(8)
O(5)Zn(1)N(2)	155.60(8)	N(1)Zn(1)N(2)	80.49(9)
IV			
O(1)Cd(1)O(2)	51.5(2)	O(1)Cd(1)O(4)	151.1(2)
O(1)Cd(1)O(5)	107.3(2)	O(1)Cd(1)N(1)	76.0(2)
O(1)Cd(1)N(2)	91.8(2)	O(2)Cd(1)O(4)	136.9(2)
O(2)Cd(1)O(5)	90.0(2)	O(2)Cd(1)N(1)	126.2(2)
O(2)Cd(1)N(2)	115.0(2)	O(4)Cd(1)O(5)	53.5(2)
O(4)Cd(1)N(1)	84.3(2)	O(4)Cd(1)N(2)	102.3(3)
O(5)Cd(1)N(1)	95.9(3)	O(5)Cd(1)N(2)	154.8(3)

Table 3. Geometric parameters of hydrogen bonds for complexes **I–IV**

Contact D–H···A	Distance, Å			Angle D–H···A, deg
	D–H	H···A	D···A	
		I		
O(2)–H(1)···O(1)	0.79(5)	2.10(5)	2.865(5)	162(5)
O(4)–H(4)···N(1)	0.82	1.93	2.715(6)	161(2)
O(2)–H(2)···O(3)	0.82	1.85	2.586(5)	149(8)
		II		
O(3)–H(3)···O(4*w*)	0.82	1.86	2.671(5)	171
O(6)–H(6)···O(1*w*)	0.82	1.84	2.657(5)	176
O(8)–H(8*A*)···O(4)	0.82	1.87	2.688(6)	173
O(12)–H(12)···O(1)	0.82	1.82	2.631(6)	173
		III		
O(1*w*)–H(1*wA*)···O(3)	0.86(3)	2.30(6)	2.886(5)	126(7)
O(1*w*)–H(1*wB*)···O(4)	0.85(4)	2.51(5)	2.906(4)	109(5)
O(6)–H(61)···O(2)	0.93	1.93	2.741(3)	146
O(1)–H(100)···O(1*w*)	0.97	1.75	2.695(5)	166
		IV		
O(2*w*)–H(2*wB*)···O(6)	0.83(8)	2.08(9)	2.846(12)	155(12)
O(1*w*)–H(1*wB*)···O(4)	0.84(7)	1.99(9)	2.817(11)	170(19)
O(3)–H(3)···O(5)	0.82	1.83	2.646(10)	175
O(1*w*)–H(1*wA*)···O(2*w*)	0.83(16)	1.93(17)	2.758(15)	173(16)
O(2*w*)–H(2*wA*)···O(3)	0.83(6)	2.03(9)	2.834(12)	163(12)
O(6)–H(6)···O(1*w*)	0.82	1.82	2.627(13)	166

Phen ligand. A new compound **III** was obtained. The asymmetric unit of **II** consists of one Zn^{2+} ion, two L^1 anions, one chelating Phen ligand and one free water molecule. In this compound, the L^1 shows monodentate and bisdentate coordinative modes. As shown in Fig. 3a, the independent Zn(1) atom is surrounded by three coordinating O atoms and two N atoms to form a trigonal bipyramidal coordination sphere with *S* parameter of 0.61 (0 value is expected for an ideal tetragonal pyramidal and 1.0 value for an ideal trigonal bipyramidal geometry, respectively) [16]. The Zn(1)–O bond lengths are at 1.962(2)–2.314(2) Å range (Table 2), conforming to the literature values for the related compounds [17]. The free water molecule O(1*w*) taking as donor connects the O(3) from monodentate carboxylate group, which extends the each subunit into 1D chain. Notably, the 1D chain are further connected by the H-bonded interactions between phenolic group and carboxylate group (O(6)···O(2) 2.741(3) Å) into a 2D sheet, as shown in Fig. 3b.

To investigate the effect of the metal ion on the complex networks, the Cd(II) resource was deliberately introduced into the HL^1–Phen system. A new compound **IV** was obtained. Complex **IV** displays a dinuclear structure. The structure of **IV** contains two Cd^{2+} ions, four L^1 ligands, two chelating Phen ligands, and four lattice water molecules. Each Cd atom in the dinuclear motif is coordinated by five oxygen atoms from two different carboxylic group of L^1 ligands and two N atoms from one chelating Phen ligand, completing a pentagonal bipyramid geometry (Fig. 4a and Table 2). The two carboxylic groups show η^1-η^1-μ_2 and η^1-η^2-μ_2 modes.

In addition, careful analysis reveals that the hydrogen bonded interaction association of solvent water molecules, phenolic groups and carboxylate groups in **IV** leads to the formation of 2D supramolecular network. The oxygen atoms (O(4) and O(5)) of carboxylate could take as acceptors to bind with O(1*w*) and O(3). The oxygen atoms (O(3) and O(6)) from hydroxyl groups acting as acceptors bind to O(2*w*). Also, the O(3) from hydroxyl group taking as donor involves in O(4) of carboxylate into a 1D chain, which are cross-lined by other H-bonded interactions between phenolic groups and free water molecules. There is weaker packing interaction between rings from adjacent Phen molecules (Fig. 4b).

In complexes **I–IV**, the L^1 displays different bridging fashions (Scheme). A comparison of the crystal structures of **I–III** as well as **III** and **IV** clearly indi-

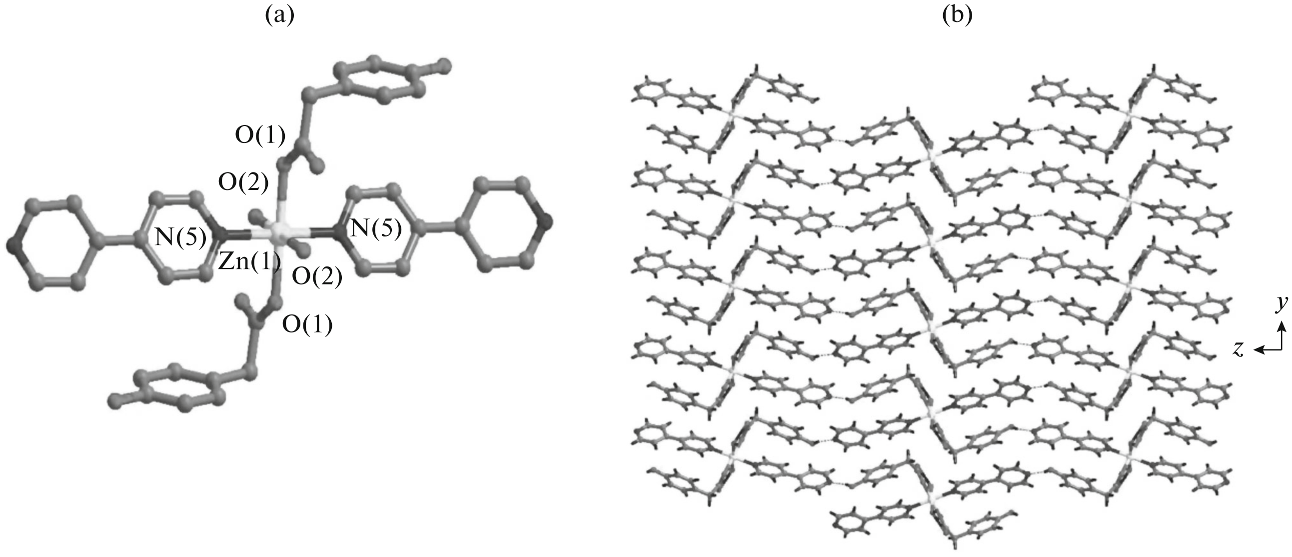

Fig. 1. The coordination geometries of the metal centers and the ligands geometries in **I** (displacement ellipsoids are drawn at the 30% probability level and H atoms are omitted for clarity) (a); view of the 2D packing framework directing by weak interactions (b).

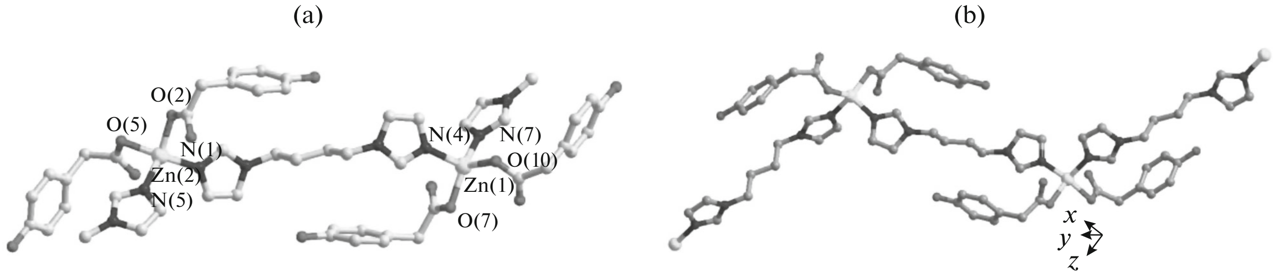

Fig. 2. The coordination geometries of the metal centers and the ligands geometries in **II** (displacement ellipsoids are drawn at the 30% probability level and H atoms are omitted for clarity) (a); view of the 1D covalent chain constructed by mi ligands (b).

cates that the auxiliary N-donor ligands have a significant influence on structural assembly of coordination architectures. When the auxiliary bridging ligands Mi and 4,4'-Bipy are introduced into the Zn–HL^1 systems, the extended 1D covalent and monodentate complexes are obtained. The resulting motifs can be ascribed to the flexible conformation on the bridging ligands. However, when the terminal phen is introduced, the expected low dimensional motif **III** is prepared because the chelating ligand will inhibit the extension of the networks. On the other hand, the same terminal N-donor ligand and coordinative numbers of Zn(II) and Cd(II) centers, the **III** and **IV** have the completely coordinative architecture due to the different atom radius [18].

In the FT-IR spectra, all the compounds show a broad band centered around 3300 cm^{-1} attributable to the O–H stretching frequency of the water. The asymmetric stretching vibrations $\nu(COO^-)$ appear around 1580 cm^{-1} for **I**–**IV**, and the symmetric stretching vibrations $\nu(COO^-)$ are observed 1420 cm^{-1}. For the complexes, the difference between the asymmetric and symmetric stretches, $\Delta\nu_{as}(COO^-) - \nu_s(COO^-)$, are on the order of 150 and or 200 cm^{-1} indicating that carboxyl groups are coordinated to the metal in a bidentate and monodentate modes [19], consistent with the observed X-ray crystal structures of **I**–**IV**.

The stability of complexes **I**–**IV** was studied by thermogravimetric analyses (TGA). The TGA diagram of I shows two weight loss steps. The first weight loss began at 35°C and completed at 135°C. The observed weight loss of 3.2% is corresponding to the loss of the coordinative water molecule (calcd. 3.0%). Soon after, the deposition of all the ligands starts on. The second weight loss occurs in the range 175–750°C, which can be attributed to the elimination of L^1 and 4,4'-Bipy ligands. The complex **II** has also two weight loss steps. The first weight loss began at 35°C and completed at 70°C. The observed weight loss of 6.3% is corresponding to the loss of all the water mol-

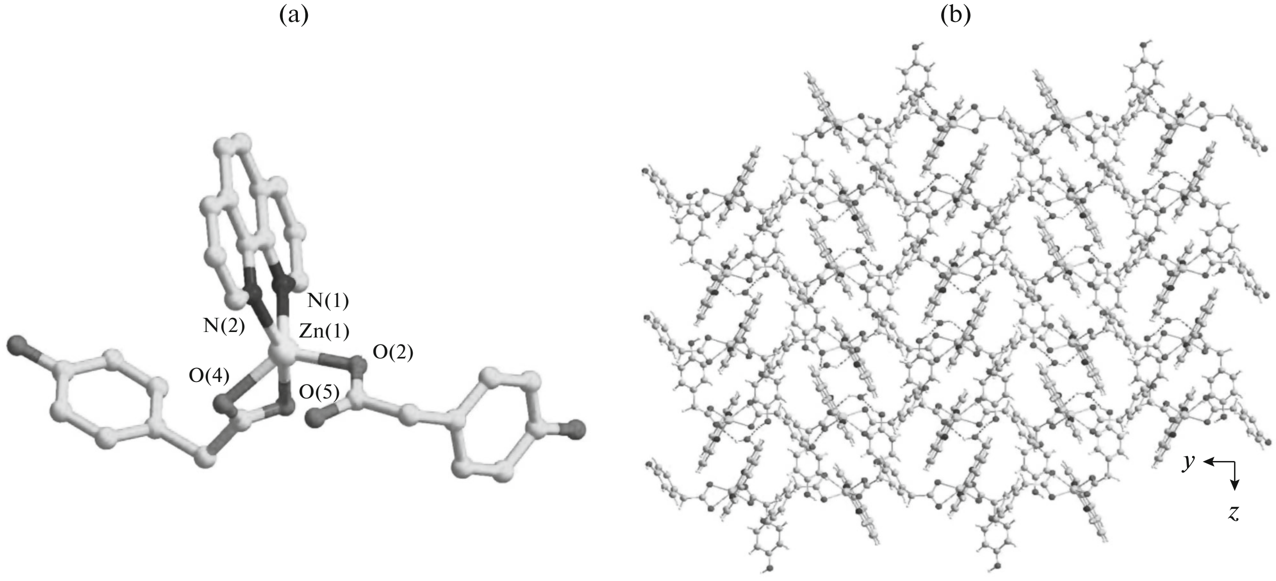

Fig. 3. The coordination geometries of the metal centers and the ligands geometries in **III** (displacement ellipsoids are drawn at the 30% probability level and H atoms are omitted for clarity) (a); view of the 2D packing framework directing by weak interactions (b).

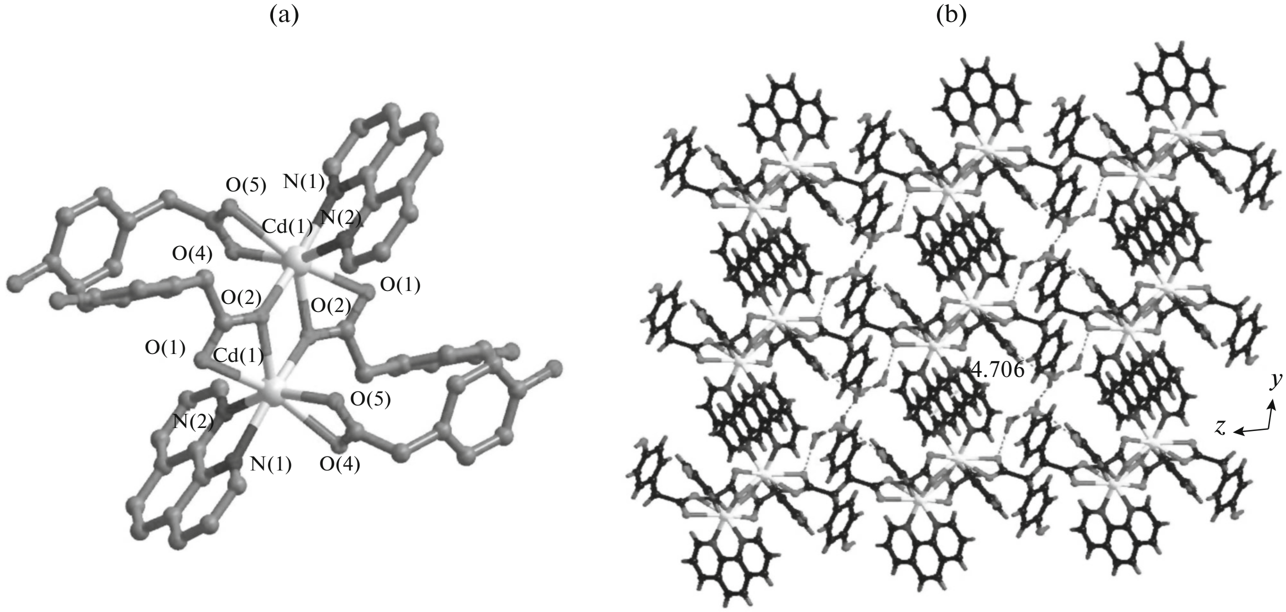

Fig. 4. The coordination geometries of the metal centers and the ligands geometries in **IV** (displacement ellipsoids are drawn at the 30% probability level and H atoms are omitted for clarity) (a); 2D packing chain directing by weak interactions (b).

ecules (calcd. 6.0%). The framework could be kept at 250°C and then start the decomposition. Complex **III** has also two weight loss steps. The free water molecule is removed and completed at 130°C (calcd. 2.81%, exp. 3.13%). The free water molecules are removed and completed at 120°C in **IV**, which corresponds to the loss of the coordinative water molecule (calcd. 11.2%, exp. 10.9%). The framework could be kept at 280°C and then start the decomposition.

Additionally, to confirm the phase purity and stability of compounds **I**–**IV**, all the original samples were characterized by X-ray powder diffraction (XRPD). Although the experimental patterns have a few unindexed diffractions lines and some are slightly broadened in comparison to those simulated from single-crystal models, it can still to be considered that the bulk synthesized materials and as-grown crystal are homogeneous for compounds **I**–**IV**.

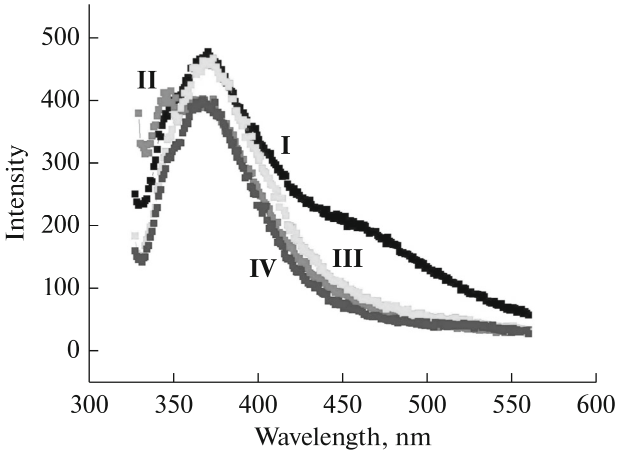

Fig. 5. View of the fluorescent emission spectra of complexes **I**, **II**, **III**, **IV**.

Coordination complexes constructed from d^{10} metal centers and conjugated organic ligands are promising candidates for photoactive materials [17, 20]. Thus, the fluorescent spectra of complexes **I**–**IV** were recorded at room temperature, which show the maximum emission bands at 371 nm (λ = 320 nm), 369 (λ = 320), 365 (λ = 320), 362 (λ = 320), respectively (Fig. 5). Moreover, the maximal emission of HL^1 ligand is observed at 361 nm (λ = 320 nm). Accordingly, the emission peaks of these complexes should be ascribed to interligand $\pi \rightarrow \pi^*$ and or $n \rightarrow \pi^*$ transitions [20].

In conclusion, we have presented synthetic strategy that successfully achieved four complexes of **I**–**IV**, which show the effect of N-donor ligand and metal ion on the modulation of resulting motifs. The photoluminescence properties of **I**–**IV** were also explored.

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