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吡嗪-2,3,5,6-四甲酸及 4,4′-联吡啶与 *ds*-金属配合物合成、结构及发光性质

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摘要：合成了 3 种金属配合物{[Cd(pztc)(H$_2$O)$_2$]·(H$_2$bpy)·3(H$_2$O)}$_n$ (**1**), {[Zn$_3$(pztc)$_2$(Hbpy)$_2$(bpy)(H$_2$O)$_4$]·7H$_2$O}$_n$ (**2**), [Cu$_6$(pztc)$_4$(Hbpy)$_4$(bpy)$_2$(H$_2$O)$_8$]·17H$_2$O (**3**)(bpy=4,4′-联吡啶, H$_4$pztc=吡嗪-2,3,5,6-四甲酸),并对其进行了红外光谱分析、元素分析、热重分析、荧光光谱分析和 X-射线单晶衍射分析。其中 **1** 和 **3** 中存在丰富的氢键,**2** 为 3D 网状结构,存在金鱼状(H$_2$O)$_{10}$ 水簇。**3** 为由化学键连接成的 1D 链状结构, 由氢键连接成 3D 超分子结构。当激发光波长为 320 nm 时,**1** 和 **2** 的最大荧光发射峰分别在 496 nm 和 494 nm。

关键词：吡嗪-2,3,5,6-四甲酸；4,4′-联吡啶；水簇；荧光光谱

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Syntheses, Structures, and Fluorescent Properties of Three *ds*-Block Metal Complexes with Pyrazine-2,3,5,6-tetracarboxylic Acid and 4,4′-Bipyridine

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Abstract: Three coordination polymers {[Cd(pztc)(H$_2$O)$_2$]·(H$_2$bpy)·3(H$_2$O)}$_n$ (**1**), {[Zn$_3$(pztc)$_2$(Hbpy)$_2$(bpy)(H$_2$O)$_4$]·7H$_2$O}$_n$ (**2**), and [Cu$_6$(pztc)$_4$(Hbpy)$_4$(bpy)$_2$(H$_2$O)$_8$]·17H$_2$O (**3**) (bpy =4,4′-bipyridine, H$_4$pztc =pyrazine-2,3,5,6-tetracarboxylic acid) have been synthesized and characterized by infrared spectra, elemental analysis, thermal analysis, fluorescence and single-crystal X-ray diffraction. The hydrogen bonding interactions in complexes **1** and **3** are abundant. Complex **2** displays a 3D network and exists an interesting goldfish-like (H$_2$O)$_{10}$ cluster. Complex **3** shows 1D herringbone chain structures, which can be linked by hydrogen bonds to form a 3D supramolecular structure. In addition, on excitation of 320 nm, complexes **1** and **2** emit fluorescence with emission maximum at 496 nm and 494 nm respectively. CCDC: 888444, **1**; 888445, **2**; 888446, **3**.

Key words: pyrazine-2,3,5,6-tetracarboxylic acid; 4,4′-bipyridine; water cluster; fluorescent properties

The rational design and syntheses of coordination polymers have achieved considerable progress in the fields of supramolecular chemistry and crystal engineering, owing to their potential applications in luminescence, magnetism, catalysis, gas storage, ion exchange, and nonlinear optics as well as due to their

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intriguing variety of architectures[1-2]. A lot of such coordination polymers have been prepared through making use of not only strong covalent bonding but also weak bonding such as hydrogen bonds and $\pi\cdots\pi$ stacking interactions between polyfunctional organic ligands and metal ions[3-4].

The synthesis of coordination polymers containing two different types of ligands allows for greater flexibility in the design of coordination networks than those based on only one[5-6]. Up to now, there are a few complexes containing *ds*-block metal ions and pyrazine-2,3,5,6-tetracarboxylic acid (H_4pztc) or other bridging multifunctional organic ligands with N- and O- donors[7-9]. To the best of our knowledge, there is no study about Cd(Ⅱ), Zn(Ⅱ), and Cu(Ⅱ) complexes with pyrazine-2,3,5,6-tetracarboxylic acid (H_4pztc) and 4,4′-bipyridine (bpy). H_4pztc containing O- and N- atoms have proven to be elegant candidate for the rich coordination modes and function as hydrogen-bond accepters as well as hydrogen-bond donors[3-4]. 4,4′-bipyridine has been relatively well-known an excellent building block in the design and construction of supramolecular polymers. It can not only act as an exobidentate bridging ligand, but can also serve as a terminal ligand or an uncoordinated guest molecule, which may be further involved in hydrogen bonding and/or $\pi\cdots\pi$ stacking interactions[10]. In this work, complexes $\{[Cd(pztc)(H_2O)_2]\cdot(H_2bpy)\cdot 3(H_2O)\}_n$ (**1**), $\{[Zn_3(pztc)_2(Hbpy)_2(bpy)(H_2O)_4]\cdot 7H_2O\}_n$ (**2**), and $[Cu_6(pztc)_4(Hbpy)_4(bpy)_2(H_2O)_8]\cdot 17H_2O$ (**3**) have been characterized by elemental analysis, IR spectra, thermal gravimetric analysis, and single crystal X-ray diffraction. The luminescent properties of complexes **1** and **2** have also been investigated.

1 Experimental

1.1 General

Pyrazine-2,3,5,6-tetracarboxylic acid was synthesized by reported method[11], other chemicals purchased were of reagent grade and used without purification. Elemental analyses for C, H, and N were performed on a Perkin-Elmer 240 CHN elemental analyzer. IR spectra were recorded from 400 to 4 000 cm^{-1} with a Bruker TENSOR 27 spectrophotometer using a KBr pellet. TGA experiments were performed on a NETZSCH TG 209 instrument with a heating rate of 10 ℃·min^{-1}. Fluorescence spectra were measured on a Cary Eclipse EL06063917 fluorescence spectrophotometer with a xenon arc lamp as the light source.

1.2 Syntheses of the complexes

Complex **1**: 6 mL ethanolic solution of bpy (0.046 9 g, 0.3 mmol) and 15 mL aqueous solution of H_4pztc (0.038 4 g, 0.15 mmol) were added to 15 mL aqueous solution of $Cd(NO_3)_2\cdot 4H_2O$ (0.092 5 g, 0.3 mmol). The mixture was adjusted to pH =2.5 and stirred for 2 h. Colorless parallelogram crystals of **1** were obtained after the filtrate was allowed to stand at room temperature for one week. Yield: 66% based on Cd. Elemental analysis, Found(%): C, 35.33; H, 3.26; N, 9.15. Calcd. for $C_{18}H_{20}CdN_4O_{13}$ (%): C, 35.28; H, 3.29; N, 9.14. IR (KBr, cm^{-1}): 3 474vs, 1 607vs, 1 447 m, 1 417m, 1 308m, 629w, 491w.

Complex **2**: 6 mL ethanolic solution of bpy (0.046 9 g, 0.3 mmol) and 15 mL aqueous solution of H_4pztc (0.0384 g, 0.15 mmol) were added to 15 mL aqueous solution of $ZnSO_4\cdot 7H_2O$ (0.086 3 g, 0.3 mmol). The mixture was stirred for 1 h. Colorless rectangular crystals of **2** were obtained after the filtrate was allowed to stand at room temperature for one week. Yield: 62% based on Zn. Elemental analysis, Found(%): C, 40.32; H, 3.51; N, 10.24. Calcd. for $C_{46}H_{48}Zn_3N_{10}O_{27}$ (%): C, 40.35; H, 3.53; N, 10.23. IR (KBr, cm^{-1}): 3 473vs, 3 372vs, 1 648vs, 1 441m, 1 323 s, 603w, 476w.

Complex **3** was prepared with the same method as **2** using $Cu(ClO_4)_2\cdot 6H_2O$ (0.111 1 g, 0.3 mmol) instead of $ZnSO_4\cdot 7H_2O$. Blue square crystals of 3 were obtained after the filtrate was allowed to stand at room temperature for about 10 days. Yield: 58% based on Cu. Elemental analysis, Found(%): C, 39.61; H, 3.69; N, 10.07. Calcd. for $C_{92}H_{102}Cu_6N_{20}O_{57}$(%): C, 39.73; H, 3.67; N, 10.07. IR (KBr, cm^{-1}): 3 415vs, 1 633vs, 1 421m, 1 302s, 642w, 424w.

1.3 X-ray crystallography

Crystal diffraction data were collected on a

computer-controlled Rigaku Saturn 724 CCD detector for **1** and rigaku saturn CCD area detector for **2** and **3**. The detector was equipped with graphite-monochromatized Mo $K\alpha$ radiation (λ=0.071 073 nm) by using the ω- and φ-scan technique. The structures were solved by direct methods and refined with full-matrix least-squares on F^2 using SHELXS-97 and SHELXL-97[12]. All non-hydrogen atoms were refined anisotropically. All hydrogen atoms were generated geometrically and refined with isotropic temperature factors. The crystallographic data are listed in Table 1.

CCDC: 888444, **1**; 888445, **2**; 888446, **3**.

2 Results and discussion

2.1 Structure description

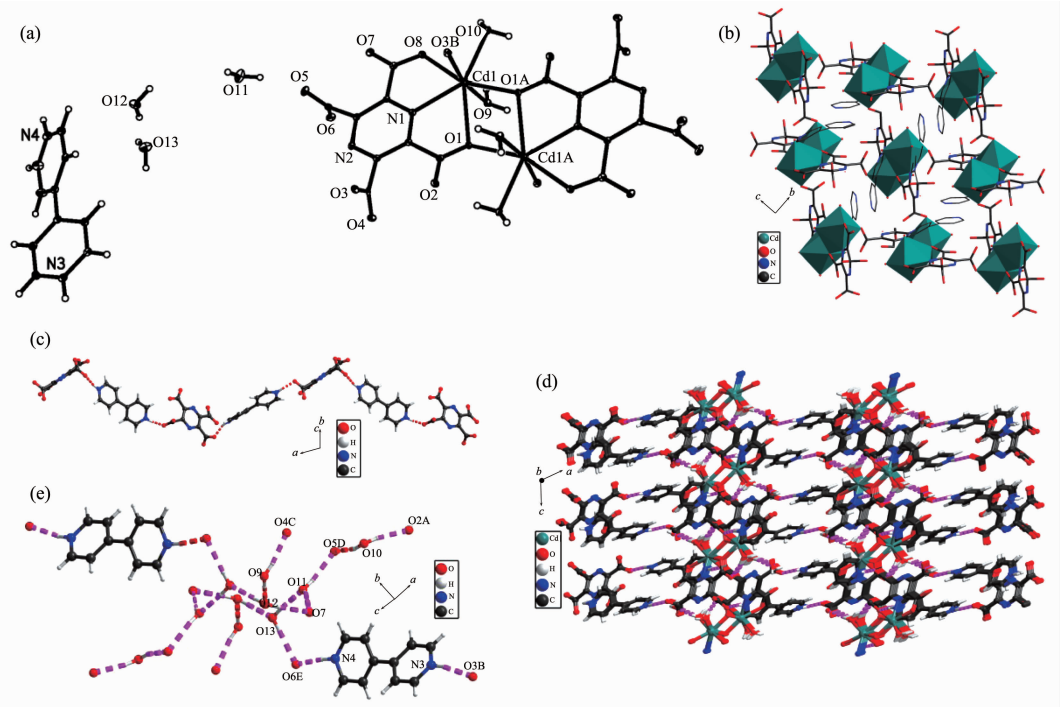
The asymmetric unit of **1** consists of one Cd(Ⅱ) ion, one $pztc^{4-}$, one lattice H_2bpy^{2+}, two coordinated water molecules, and three uncoordinated water molecules. As shown in Fig.1a, the Cd(Ⅱ) ion is 7-coordinated and exhibits a distorted pentagonal bipyramidal geometrical coordination environment. The equatorial sites are occupied by ONO (O1, N1, and O8) from one $pztc^{4-}$, O1A from a different $pztc^{4-}$, and O10 of a water molecule, while the axial sites are occupied by O9 of a coordinated water molecule and O3B from one $pztc^{4-}$. The adjacent Cd(Ⅱ) ions are connected by O1 and O1A into a $[Cd_2(pztc)_2(H_2O)_4]$ unit, which can continue to grow into a 2D grid layer and the lattice H_2bpy^{2+} fill in the 2D grid layer (Fig. 1b).

The $pztc^{4-}$ and H_2bpy^{2+} are linked by N–H⋯O hydrogen bonding interactions to construct an infinite 1D zigzag chain (Fig.1c), and the 1D chain expands the 2D grid layer to a 3D supramolecular structure (Fig.1d). The hydrogen bond interactions in **1** are abundant, three water molecules (O11, O12, and O13), one $pztc^{4-}$ O atom O7, and their symmetric equivalents form an interesting centrosymmetric eight-membered crown hydrogen bonding ring (Fig.1e) with two additional acyclic tripolymers (O5D, O10, O2A;

Table 1 Crystal data and structure refinement information for 1~3

Complex	**1**	**2**	**3**
Empirical formula	$C_{18}H_{20}CdN_4O_{13}$	$C_{46}H_{48}Zn_3N_{10}O_{27}$	$C_{92}H_{102}Cu_6N_{20}O_{57}$
Formula weight	612.78	1 369.05	2 781.18
Crystal system	Monoclinic	Orthorhombic	Orthorhombic
Space group	$P2_1/c$	$Pcca$	$Pnna$
a / nm	1.688 5(10)	1.986 2(4)	2.215 4(4)
b / nm	0.963 9(5)	1.164 5(2)	2.047 9(4)
c / nm	1.491 0(9)	2.281 3(5)	2.350 8(5)
β / (°)	113.828(10)	90.00	90.00
V / nm^3	2.220(2)	5.276 5(18)	10.66 6(4)
Z	4	4	4
D_c / (Mg·m^{-3})	1.833	1.723	1.732
μ / mm^{-1}	1.063	1.456	1.292
F(000)	1 232	2 800	5 696
θ range / (°)	1.32 to 27.85	2.22 to 27.88	1.84 to 26.73
Reflections collected / unique	22 538 / 5 275 (R_{int}=0.059 5)	41 694 / 6 289 (R_{int}=0.037 9)	80 227 / 11 326 (R_{int}=0.060 6)
Goodness of fit on F^2	1.007	1.010	1.006
Final R^a indices [$I>2\sigma(I)$]	R_1=0.032 6, wR_2=0.062 5	R_1=0.031 0, wR_2=0.076 8	R_1=0.045 2, wR_2=0.110 1
R^a indices (all data)	R_1=0.044 3, wR_2=0.066 7	R_1=0.035 1, wR_2=0.079 7	R_1=0.054 1, wR_2=0.115 6
Parameters	373	429	1 011
Observed reflections [$I>2\sigma(I)$]	4 349	5 736	9 912
$(\Delta\rho)_{max}$, $(\Delta\rho)_{min}$ / (e·nm^{-3})	584, −635	509, −410	615, −588

$^aR_1=\sum||F_o|-|F_c||/|F_c|$; $wR_2=[\sum w(F_o^2-F_c^2)^2/\sum w(F_o^2)^2]^{1/2}$



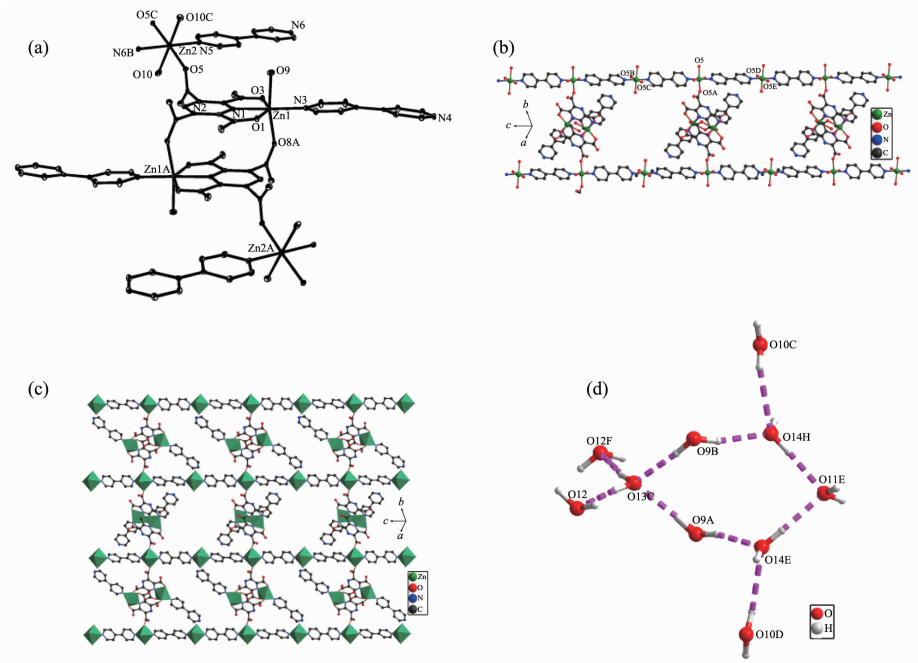
Uncoordinated water molecules in (b, c, and d), and hydrogen atoms in (b) are omitted; Symmetry code: A: $-x+1, -y+1, -z$; B:$-x+1, y+1/2, -z+1/2$ in (a); A: $-x+1, -y+1, -z$; B: $-x+1, y-1/2, -z+1/2$; C: $x, y+1, z$; D: $x, -y+3/2, z-1/2$; ^{v} $-x, -y+1, -z$ in (e)

Fig.1　Structure unit showing with ellipsoids at the 50% probability level (a), the 2D grid layer (b), the infinite 1D zigzag hydrogen bonding chain (c), the 3D supramolecular structure (d), and the crown of hydrogen bonding ring (e) of complex **1**

their symmetric equivalents), two additional acyclic tetramers (O6E, N4, N3, O3B; their symmetric equivalents), and two additional acyclic dimmer (O9, O4C; their symmetric equivalents) dangling from opposite ends of the hydrogen bonding ring respectively and pointing away from the hydrogen bonding ring. The end of every acyclic polymers are O2A, O3B, O4C, O7, and their symmetric equivalents. These atoms come from different $pztc^{4-}$ ligands, so they can expand the crown hydrogen bonding structure to a 3D stair-like supramolecular structure.

The crystal structure of **2** consists of a centrosymmetry polynuclear $[Zn_4(pztc)_2(Hbpy)_2(bpy)_2(H_2O)_6]$ structural unit (Fig.2a). There are two independent Zn(Ⅱ) ions (Zn1 and Zn2) in different coordination environments. Zn1 is 6-coordinated and exhibits a distorted octahedral geometry. The equatorial sites are occupied by ONO (O1, N1, and O3) donors from a $pztc^{4-}$ ligand and one N atom (N3) from an $Hbpy^{+}$ ligand, while the axial sites are occupied by O8A from another $pztc^{4-}$ ligand and O9 of a water molecule. Zn2 has a slightly distorted octahedral geometry. The equatorial sites are occupied by O atoms (O5, O5C) from different $pztc^{4-}$ ligands and O atoms (O10, O10C) of two water molecules, and the axial sites are occupied by two N atoms (N5, N6B) from different bpy molecules. The crystal structure can be further linked by N5-Zn2-N6 to construct a 1D double chain (Fig.2b).

In complex **2**, bpy molecules act as exobidentate bridging ligands and link Zn2 ions, while $Hbpy^{+}$ serve as terminal ligands coordinating to Zn1 ions. From Fig. 2b, we can see that O5 atoms coordinating to Zn2 are alternately stretch along two directions, and the Zn2-O5 along different directions can further construct the 1D double chains to 2D layers (Fig.2c) and 3D networks. Interestingly, the coordinated and lattice water molecules are connected by hydrogen bonding to construct a goldfish-like $(H_2O)_{10}$ cluster (Fig.2d). The average O⋯O distance of 0.277 5 nm within the $(H_2O)_{10}$ cluster is close to the distance found in the regular ice (0.274 nm) and other water clusters[13]. The bpy ligands in **2** exert significant



Lattice water molecules and hydrogen atoms in (a), (b) and (c) are omitted; Symmetry code: A: $-x$, $-y+1$, $-z$; B: $-x+1/2$, y, $z-1/2$; C: $-x+1/2$, $-y$, z in (a); A: $-x+1/2$, $-y$, z; B: $-x+1/2$, y, $z+1/2$; C: x, $-y$, $z+1/2$; D: $-x+1/2$, y, $z-1/2$; E: x, $-y$, $z-1/2$ in (b); A: $-x+1/2$, y, $z+1/2$; B: x $-y+1$, $z+1/2$; C: x, $-y$, $z+1/2$; D: $-x+1/2$, $y+1$, $z+1/2$; E: $-x+1/2$, y, $z-1/2$; F: $-x+1/2$, $-y+1$, z; G: $-x+1/2$, y, $z+1/2$; H: x, $-y+1$, $z-1/2$ in (d)

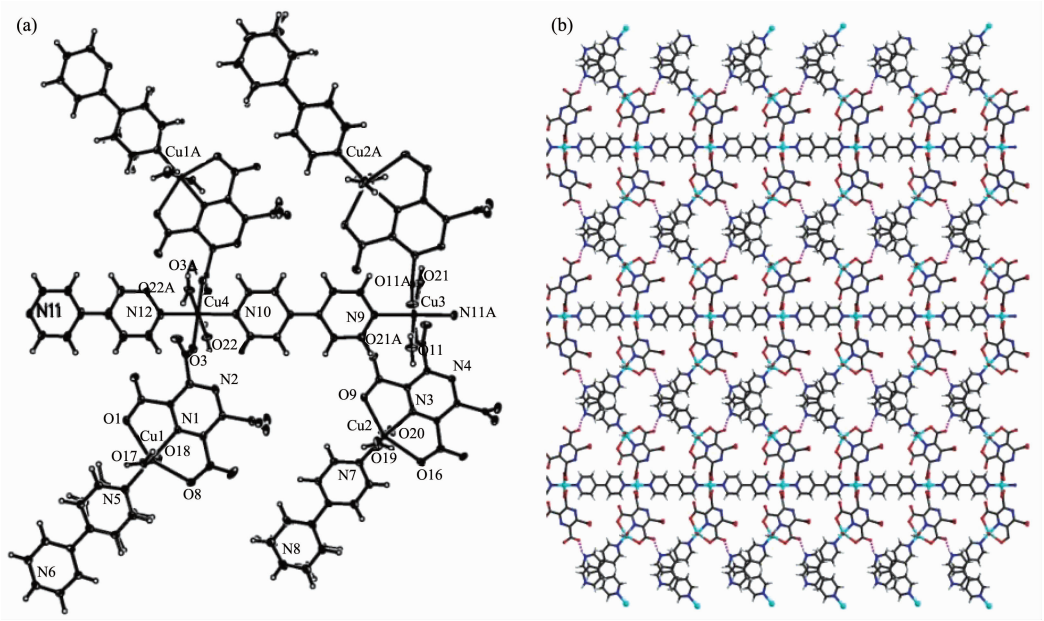
Fig.2 (a) Structure unit showing with ellipsoids at the 50% probability level(a), the infinite 1D double chain (b), the 2D layer (c), and the goldfish-like water cluster (d) in **2**

influence on the self-assembly 3D structure compared with the reported zinc coordination polymers constructed from H_4pztc[7], and the synthesis method is simpler than the hydro (solvo)thermal syntheses used by the reported zinc coordination polymers constructed from other multifunctional organic ligands with N- and O- donors and bpy[5-6,14].

The crystal structure of **3** consists of an axisymmetric polynuclear $[Cu_6(pztc)_4(Hbpy)_4(bpy)_2(H_2O)_8]$ structure unit. The coordination modes of bpy and $Hbpy^+$ molecules in **3** are similar with that in **2**. As shown in Fig.3a, there are four independent Cu(Ⅱ) ions (Cu1, Cu2, Cu3, and Cu4) in different coordination environments. Cu1 is 6-coordinated and exhibits a distorted octahedral geometry. The equatorial sites are occupied by ONO (O1, N1, and O8) from a $pztc^{4-}$ ligand and one N atom (N5) from one $Hbpy^+$ ligand, while the axial sites are occupied by two O atoms (O17, O18) of two water molecules. The coordination environment of Cu2 is similar with Cu1. Cu3 adopts a slightly distorted square planar geometry, and coordinates with two O atoms (O11, O11A) from different $pztc^{4-}$ ligands and two N atoms (N9, N11B) from two bpy molecules. The coordination environment of Cu4 is similar with Cu3. The axisymmetric polynuclear units are connected by bpy molecules to generate an infinite 1D herringbone chain (Fig.3a). The 1D herringbone chains are connected by N–H⋯O hydrogen bonding interactions to construct a 2D layer structure (Fig.3b). The 2D layers are further assembled via O–H⋯O hydrogen bonding interactions to build a 3D supramolecular structure.

2.2 Thermal analysis (TGA)

TGA were carried out for **1**~**3** in the temperature range of 30~800 ℃ under the atmosphere of air with heating rate of 10 ℃·min^{-1} (Fig.4). For **1**, the weight loss of 13.9% from 30~177 ℃ corresponds to the loss of 2 coordinated and 3 uncoordinated water molecules (Calcd. 14.7%), the degradation of the bpy, H_2bpy^+, and $pztc^{4-}$ occur in the temperature of 208~600 ℃. The remaining weight of 21.5% is presumably CdO, which is in agreement with the calculated value of



Lattice water molecules are omitted; Symmetry code: A: x, $-y-1/2$, $-z+1/2$; B: $x+1$, y, z in (a)

Fig.3　Structure unit showing with ellipsoids at the 50% probability level of **3** (a) and the 2D hydrogen bonding layer structure (b) of **3**

21%. The TG curve of **2** shows weight loss of 14.89% within 117 ℃ corresponding to 4 coordinated and 7 lattice water molecules (Calcd. 14.47%). Between 117~266 ℃, there is almost no weight change. The complex continues to decompose above 266 ℃. The weight loss of **3** from 30 ℃ to 227 ℃ is 15.84%, which was resulted from the loss of 8 coordinated and 17 lattice water molecules (Calcd. 16.18%). When the temperature was further increased, decomposition is not achieved until 392 ℃. The remaining weight of 15.22% is presumably CuO, which is in agreement with the calculated value of 17.13%.

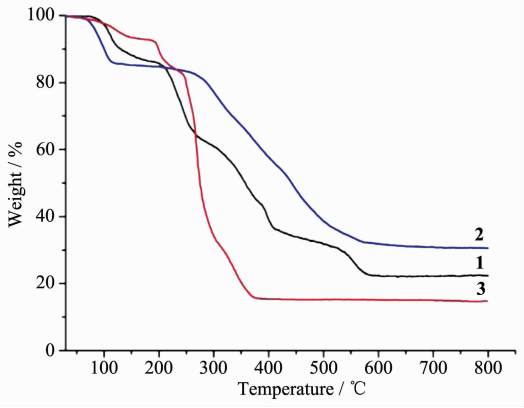


Fig.4　TG curves of **1**~**3**

2.3　Fluorescence

Previous studies have shown that polymeric compounds containing cadmium and zinc exhibit photoluminescent properties and they have potential applications in optical amplifiers, optical waveguides and OLEDs[15]. The photoluminescent properties of complexes **1** and **2** were also investigated (Fig.5). The fluorescent emission spectra of compounds **1** and **2** were detected in the solid state at room temperature. These two complexes display blue/green emissions at 496 nm for **1** (λ_{ex}=320 nm) and 494 nm for **2** (λ_{ex}=320 nm), which are similar to those of the corresponding ligands. Since Cd^{2+} and Zn^{2+} are difficult to oxidize or reduce due to the d^{10} configuration, emissions of **1** and **2** are neither attributable to ligand-to-metal charge

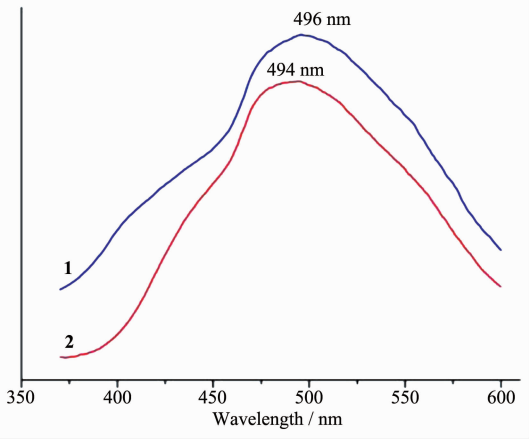


Fig.5　Emission spectra of **1** and **2**

transfer bands (LMCT) nor metal-to-ligand charge transfer bands (MLCT). They may be attributable to fluorescent emissions from an intraligand excited state[16]. The enhancement of luminescence may be attributed to ligands coordinated to the metal center, which effectively increases the rigidity of the ligands reducing the loss of energy by radiationless decay. Complexes **1** and **2** may be the potential fluorescent materials for blue/green-light emitting diode devices because of the blue/green fluorescence emission.

3 Conclusion

Three *ds*-block metal coordination polymers constructed from H_4pztc and bpy at room temperature were synthesized. In complex **1**, bpy molecules exist as H_2bpy^{2+} and act as uncoordinated ions. While in complexes **2** and **3**, bpy molecules exist as bpy and $Hbpy^+$, participating in the coordination. The hydrogen bond interactions in **1** are abundant. Three water molecules, one $pztc^{4-}$ O atom, and their symmetric equivalents form an interesting centrosymmetric eight-membered crown hydrogen bonding ring, which can be expanded by $pztc^{4-}$ to construct a 3D stair like supramolecular structure. The coordinated and lattice water molecules in complex **2** are connected by hydrogen bonds to construct an interesting goldfish-like $(H_2O)_{10}$ cluster. Complex **3** displays an interesting 1D herringbone chain. In addition, complexes **1** and **2** show intense emission bands, which render them to be potentially used as fluorescent materials.

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