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Two transition metal supramolecular compounds based on 2-(4-isopropylbenzoyl)benzoic acid: syntheses, crystal structures, and luminescent properties

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Two transition metal supramolecular compounds based on 2-(4-isopropylbenzoyl)benzoic acid: syntheses, crystal structures, and luminescent properties

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Two new supramolecular compounds, $[Zn_2(L)_3(4,4'\text{-bpy})(OH)]_n$ (**1**) and $[Cd(L)_2(2,2'\text{-bpy})(H_2O)]\cdot 2H_2O$ (**2**) (HL = 2-(4-isopropylbenzoyl)benzoic acid, 4,4′-bpy = 4,4′-bipyridine, 2,2′-bpy = 2,2′-bipyridine), have been hydrothermally synthesized and characterized by elemental analysis, infra-red spectroscopy, thermal gravimetric analyzes, and single-crystal X-ray diffraction. **1** exhibits 1-D chain and **2** is 0-D mononuclear. They are both linked into 2-D supramolecular layers by non-covalent interactions. Luminescence properties were also investigated.

Keywords: Supramolecular; 2-(4-Isopropylbenzoyl)benzoic acid; Non-covalent interactions; Water cluster; Luminescent property

1. Introduction

Supramolecular chemistry, which covers entities formed by associations of two or more chemical species, is a remarkable area in crystal engineering [1–5]. Design and synthesis of supramolecular materials has provoked interest for diverse structural flexibilities and promising applications in catalysis, magnetism, electronics, and optics [6–9]. Generally, the crystal structure is stabilized and directed into supramolecular architecture through non-covalent interactions (such as hydrogen-bonding and $\pi\cdots\pi$ stacking interactions) [10–13]. Hydrogen bonding is the most important organizing force in supramolecular assemblies by virtue of unique strength and directionality that may control short-range packing; many researchers have focused on investigation of hydrogen-bonding interactions [14–18]. Although hydrogen-bonding interaction is well-recognized, rational use of it to design and synthesize supramolecular architectures is still quite challenging because of the multiformity of hydrogen bonding modes [19,20].

Though quite a few compounds constructed with substituted benzoate ligands have been obtained [21–26], only two examples have been reported based on 2-(4-isopropylbenzoyl)

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benzoic acid (HL) [23]. HL can adopt monodentate, bidentate bridging or chelate with carboxylic and the isopropylbenzoyl group may direct the structures of coordination polymers by steric hindrance. Zn(II), Cd(II), and Hg(II) compounds have intriguing structures [27], a wide variety of geometries, different coordination numbers and optical applications [28,29]. We synthesized two d^{10} metal compounds based on 2-(4-isopropylbenzoyl)benzoic acid (HL), $[Zn_2(L)_3(4,4'\text{-bpy})(OH)]_n$ (**1**), and $[Cd(L)_2(2,2'\text{-bpy})(H_2O)]\cdot 2H_2O$ (**2**), exhibiting 1-D chain and 0-D mononuclear structures, respectively, and they are both linked into 2-D supramolecular layers by non-covalent interactions. Herein, we report the syntheses, crystal structures, infra-red (IR) spectra, and thermal gravimetric analyzes (TGA) of the two supramolecular compounds. Luminescent properties are also investigated.

2. Experimental

2.1. *Materials and Instrumentation*

All chemicals were obtained from commercial sources and used without purification. IR (KBr pellets) spectra were recorded from 4000 to 400 cm^{-1} using a Perkin-Elmer Spectrum One FT-IR spectrometer. Elemental analyzes (C, H, and N) were performed on a Perkin-Elmer 2400 elemental analyzer. Thermal behaviors were studied by TGA on a Perkin-Elmer Thermal Analyzer under N_2 with a heating rate of 10 °C min^{-1}. Solid-state luminescence spectra were measured on a Cary Eclipse spectrofluorometer (Varian) equipped with a xenon lamp and quartz carrier at room temperature.

2.2. *Syntheses*

2.2.1. Synthesis of $[Zn_2(L)_3(4,4'\text{-bpy})(OH)]_n$. (1). A mixture of $ZnCl_2\cdot 4H_2O$ (0.0260 g, 0.10 mmol), HL (0.0060 g, 0.02 mmol), 4,4′-bpy (0.0156 g, 0.10 mmol), and H_2O (8 mL) was adjusted to pH 5.5 with 0.10 mol L^{-1} NaOH solution. It was then sealed in a 25-mL Teflon reactor and heated at 160 °C for 72 h. After the sample cooled to room temperature at 5 °C h^{-1}, the product was recovered by filtration and air-dried. Colorless crystals were found mixed with a small quantity of white powder. The diamond-shaped colorless crystals of **1** were isolated from the white powder. Yield: 75% based on HL. Anal. Calcd for $Zn_2C_{61}H_{55}O_{10}N_2$ (Mr = 1106.84): C, 66.19; H, 5.00; N, 2.53%. Found: C, 66.13; H, 4.92; N, 2.47%. IR (KBr, cm^{-1}): 3440 (s), 3064 (m), 2959 (s), 1672 (s), 1605 (s), 1550 (s), 1391 (s), 1281 (m), 934 (s), 816 (m), 714 (s), 497 (s), 446 (m).

2.2.2. Synthesis of $[Cd(L)_2(2,2'\text{-bpy})H_2O]\cdot 2H_2O$. (2). A mixture of $CdCl_2\cdot 4H_2O$ (0.0255 g, 0.10 mmol), HL (0.0060 g, 0.02 mmol), 2,2′-bpy (0.0156 g, 0.10 mmol) and H_2O (8 mL) was adjusted to pH 6.5 with 0.10 mol L^{-1} NaOH solution. It was then sealed in a 25-mL Teflon reactor and heated at 160 °C for 72 h. After the sample cooled to room temperature at 5 °C h^{-1}, the product was recovered by filtration and air-dried. Colorless crystals were found mixed with a small quantity of white powder. The cubic shaped colorless crystals of **2** were isolated from the white powder. Yield: 80% based on HL. Anal. Calcd for $CdC_{44}H_{44}O_9N_2$ (Mr = 857.22): C, 62.02; H, 5.08; N, 3.24%. Found: C, 61.89; H, 5.01; N, 3.20%. IR (KBr, cm^{-1}): 3445 (s), 3067 (s), 2987 (w), 1667 (s), 1643 (w), 1591 (w), 1439 (s), 1218 (w), 1014 (s), 977 (w), 935 (w), 907 (s), 858 (w), 813 (w), 734 (s), 625 (w).

2.3. *X-ray crystallography*

X-ray intensity data for **1** and **2** were collected on a Bruker SMART APEX CCD diffractometer with Mo-*K*α radiation ($\lambda = 0.71073$ Å) at 273 K. The crystal structures were resolved by direct methods and refined by employing full-matrix least squares on F^2 (SHELXTL-97). Hydrogens of organic ligands and coordinated water are located geometrically. Due to small crystallographic disorder for **1**, C15, C16, C17, C50, C51, and O1 in L^- are disordered over two sites, with total occupancy of 1. Details of the final refinement are given in table 1. Selected bond lengths and angles are listed in table 2. Hydrogen-bonding parameters are listed in table S1.

3. Results and discussion

3.1. *Crystal structure of 1*

The asymmetric unit of **1** consists of two Zn(II) centers, three L^-, two halves of 4,4′-bpy and one μ_2-OH; 4,4′-bpy ligands link the structure into a 1-D chain.

As shown in figure 1, both Zn1 and Zn2 show distorted square pyramidal geometries. Three carboxylic oxygens from two L^- (O1, O2, O7 for Zn1 and O4, O5, O8 for Zn2) and one nitrogen from 4,4′-bpy (N1 for Zn1 and N2 for Zn2) occupy corners of the basal plane, and one μ_2-OH bridges Zn1 and Zn2 at axial sites. The Zn–O/N distances [1.896(3) Å–2.281(4) Å] and the O–Zn–O/N angles [56.1(6)°–153.16(16)°] are normal for reported Zn(II) compounds [30]. In **1**, μ_2-OH and one μ_2-bridging carboxylate of L^- link two Zn into a binuclear cluster [$Zn_2(\mu_2\text{-}OH)(CO_2)$] with Zn–Zn distance of 3.2352(8) Å. The other two L^- chelate to each Zn at sides of the binuclear cluster; 4,4′-bpy ligands bridge the

Table 1. Details of crystal data and structure refinement parameters for **1** and **2**.

Compound	1	2
Empirical formula	$Zn_2C_{61}H_{55}O_{10}N_2$	$CdC_{44}H_{44}O_9N_2$
Formula weight	1106.81	857.22
Temperature (K)	296(2)	296(2)
Wavelength λ (Å)	0.71073	0.71073
Crystal system	Monoclinic	Monoclinic
Space group	*P*2(1)/*n*	*P*21/*c*
a (Å)	13.0634(10)	15.230(2)
b (Å)	13.9934(11)	24.255(3)
c (Å)	30.559(2)	10.8662(14)
α (°)	90.00	90.00
β (°)	100.2890(10)	100.540(2)
γ (°)	90.00	90.00
V (Å^3)	5496.5(7)	3946.3(9)
Z	4	4
Dc (g/cm^3)	1.338	1.443
Absorption coefficient (mm^{-1})	0.933	0.613
F(000)	2300.0	1768.0
R_{int}	0.0536	0.1107
Goodness-of fit on F^2	0.944	0.929
R_1[a] $[I > 2\sigma(I)]$	0.0557	0.0564
wR_2[b] $[I > 2\sigma(I)]$	0.1436	0.1140

[a] $R_1 = \sum||F_O| - |F_c||/\sum|F_O|$.

[b] $wR_2 = \sum[w(F_O^2 - F_c^2)^2]/\sum[w(F_O^2)^2]^{1/2}$.

Table 2. Selected bond distances (Å) and angles (°) for **1** and **2**.

$[Zn_2(L)_3(4,4'\text{-bpy})(OH)]_n$ (**1**)			
Zn(1)–O(10)	1.896(3)	Zn(1)-O(7)	2.005(3)
Zn(1)–N(1)	2.048(4)	Zn(1)–O(1)	2.09(2)
Zn(1)–O(2)	2.116(4)	Zn(2)–O(8)	1.958(3)
Zn(2)–O(10)	1.908(3)	Zn(2)–N(2)	2.063(3)
Zn(2)–O(4)	2.097(4)	Zn(2)–O(5)	2.281(4)
O(10)–Zn(1)–O(7)	100.70(13)	O(4)–Zn(2)–O(5)	59.48(14)
O(10)–Zn(1)–N(1)	122.87(15)	O(7)–Zn(1)–N(1)	92.86(14)
O(10)–Zn(1)–O(1)	109.8(13)	O(7)–Zn(1)–O(1)	94.6(4)
N(1)–Zn(1)–O(1)	124.1(13)	O(10)–Zn(1)–O(2)	105.80(16)
O(7)–Zn(1)–O(2)	145.66(19)	N(1)–Zn(1)–O(2)	90.80(15)
O(1)–Zn(1)–O(2)	56.1(6)	O(10)–Zn(2)–O(8)	106.99(14)
O(10)–Zn(2)–N(2)	105.33(14)	O(8)–Zn(2)–N(2)	96.34(14)
O(10)–Zn(2)–O(4)	117.60(15)	O(8)–Zn(2)–O(4)	129.10(14)
N(2)–Zn(2)–O(4)	94.49(14)	O(10)–Zn(2)–O(5)	93.70(15)
O(8)–Zn(2)–O(5)	95.95(14)	N(2)–Zn(2)–O(5)	153.16(16)
$[Cd(L)_2(2,2'\text{-bpy})H_2O]\cdot 2H_2O$ (**2**)			
Cd(1)–O(1W)	2.280(5)	Cd(1)–O(5)	2.281(5)
Cd(1)–O(2)	2.310(6)	Cd(1)–N(2)	2.341(6)
Cd(1)–N(1)	2.310(6)	Cd(1)–O(1)	2.599(5)
Cd(1)–O(4)	2.688(5)	O(1W)–Cd(1)–O(5)	89.24(19)
O(1W)–Cd(1)–O(2)	107.3(2)	O(5)–Cd(1)–O(2)	85.7(2)
O(1W)–Cd(1)–N(2)	122.5(2)	O(5)–Cd(1)–N(2)	137.3(2)
O(2)–Cd(1)–N(2)	107.6(2)	O(1W)–Cd(1)–N(1)	83.6(2)
O(5)–Cd(1)–N(1)	88.3(2)	O(2)–Cd(1)–N(1)	167.5(2)
N(2)–Cd(1)–N(1)	70.0(2)	O(1W)–Cd(1)–O(1)	82.88(18)
O(5)–Cd(1)–O(1)	131.15(19)	O(2)–Cd(1)–O(1)	51.92(19)
N(2)–Cd(1)–O(1)	84.4(2)	N(1)–Cd(1)–O(1)	137.8(2)
O(1W)–Cd(1)–O(4)	138.41(17)	O(5)–Cd(1)–O(4)	51.81(16)
O(2)–Cd(1)–O(4)	86.1(2)	N(2)–Cd(1)–O(4)	87.90(18)
N(1)–Cd(1)–O(4)	81.6(2)	O(1)–Cd(1)–O(4)	131.75(18)

binuclear units into a 1-D chain along the *a* axis. The angle between the two pyridine rings of 4,4′-bpy is 29.274(4)°.

Hydrogen bonding and π···π interactions play important roles in **1** (figure 2). On one side of the chain, O10 of μ_2-OH are hydrogen-bonding donors to O4#2 of the neighboring chain (O10…O4#2 = 2.930(5) Å, O10–H10…O4#2 = 158(3)°; symmetry code: #2, $-x$, $2-y$, $2-z$) to form a Zn_2O_4 hexatomic ring (figure 2(B)) with Zn–Zn distance of 4.239 Å. On the other side of the chain, the C36 ring is stacked by the C36#3 ring (symmetry code: #3, $-x$, $1-y$, $2-z$) of the neighboring chain *via* offset face-to-face π···π stacking interaction (figure 2(C)) with centroid–centroid and interplanar separation distances between the two neighboring aromatic rings of 4.458 and 3.673 Å, respectively. The hydrogen bonding and π···π interactions link the 1-D chains into 2-D supramolecular layers.

3.2. *Crystal structure of 2*

X-ray single crystal diffraction reveals that **2** is a discrete structure. The asymmetric unit of **2** consists one Cd(II), two L^-, one 2,2′-bpy, one coordinated water, and two lattice waters. Cd(II) is seven-coordinate by four carboxylic oxygens (O1, O2, O4 and O5) from two L^-, two nitrogens (N1 and N2) from one 2,2′-bpy and one coordinated water (O1W), forming a slightly distorted octahedron (figure 3). The Cd-O/N bond distances from 2.280

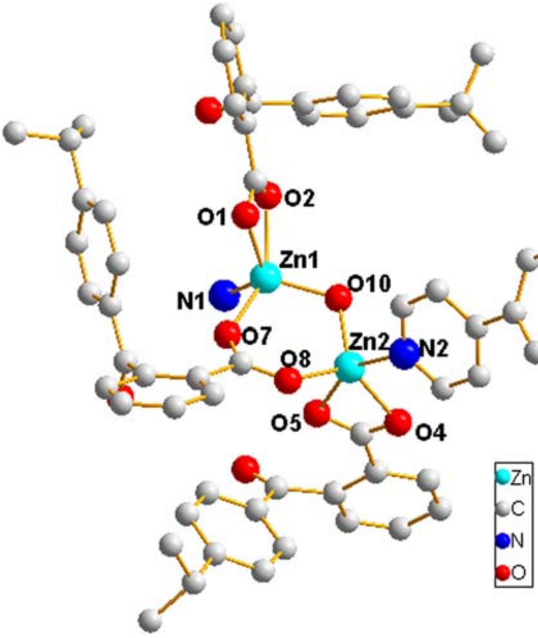

Figure 1. Coordination environments of Zinc(II) atoms in **1** (symmetry code: #1, $-1+x$, y, z; #4, $1+x$, y, z. All hydrogen atoms are omitted for clarity).

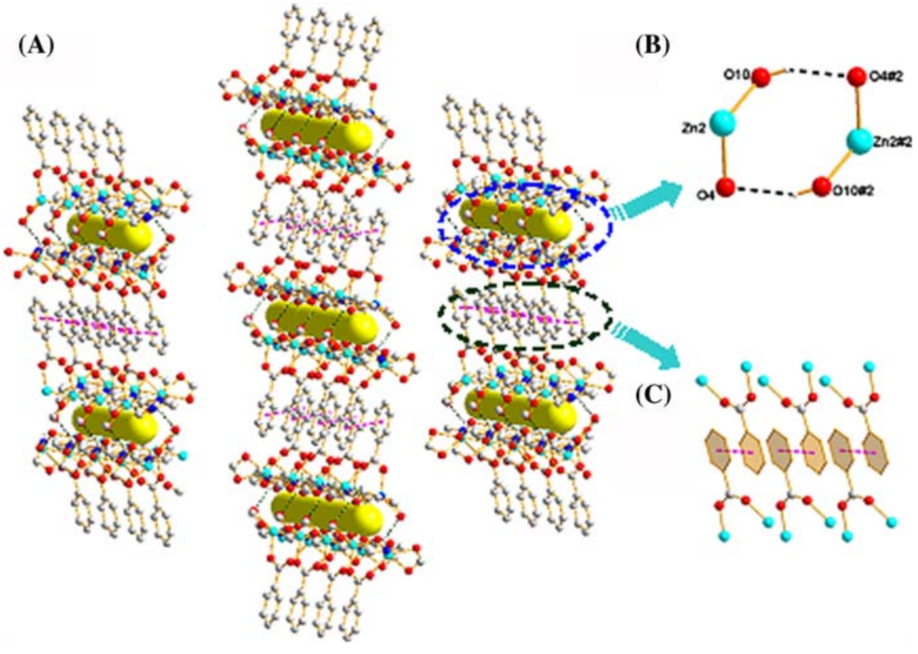

Figure 2. (A) 2-D supramolecular layers constructed *via* hydrogen bonding and $\pi\cdots\pi$ interactions in **1** (all bulky cymene skeletons omitted for clarity). (B) The Zn_2O_4 hexatomic ring linked through hydrogen bondings between neighboring chains in **1** (symmetry code: #2, $-x$, $2-y$, $2-z$). (C) The $\pi\cdots\pi$ interactions between the neighboring chains in **1**.

(5) to 2.688(5) Å are comparable with those in related Cd(II) compounds [31]. The two L^- and the 2,2′-bpy chelate Cd(II).

The most striking feature of **2** is that there exist strong hydrogen bonds among lattice and coordinated water with hexamer water clusters formed. As shown in figure 4, the hexamer water cluster can be seen as a tetramer of four lattice waters (O2W, O2W#1, O3W, and O3W#1) in a core which links two coordinated waters (O1W and O1W#1) dangling at two diagonal vertices of a quadrilateral. The O…O distances of the water cluster are 2.7525–3.0312 Å (figure 4(C)), with an average value of 2.855(2) Å, which approaches the reported 2.85 Å in liquid water [32]. The small water clusters in the crystal packing help understand the structures and characteristics of liquid water.

Discrete molecules are linked into regular 1-D chains along the *c*-axis by hydrogen-bonding interactions with water clusters. In the chain, water clusters also have strong hydrogen bonds with oxygens of L^- (O1W···O5#2 = 2.7343 Å, O3W···O6#2 = 2.8572 Å, O3W···O2#2 = 2.7709 Å, symmetry code: #2, $-x$, $1-y$, $1-z$) (figure 4(A)). As shown in figure 5, weak C–H···O hydrogen bonds between 2,2′-bpy and L^- link the chains into 2-D supramolecular layers. The hydrogen bonding-lengths and bond angles are given in table S1.

3.3. *Photoluminescence*

Solid-state luminescence spectra of HL, **1** and **2** at room temperature are shown in figure 6. Luminescent emission bands with peak maxima were observed at 478 nm (shoulder)

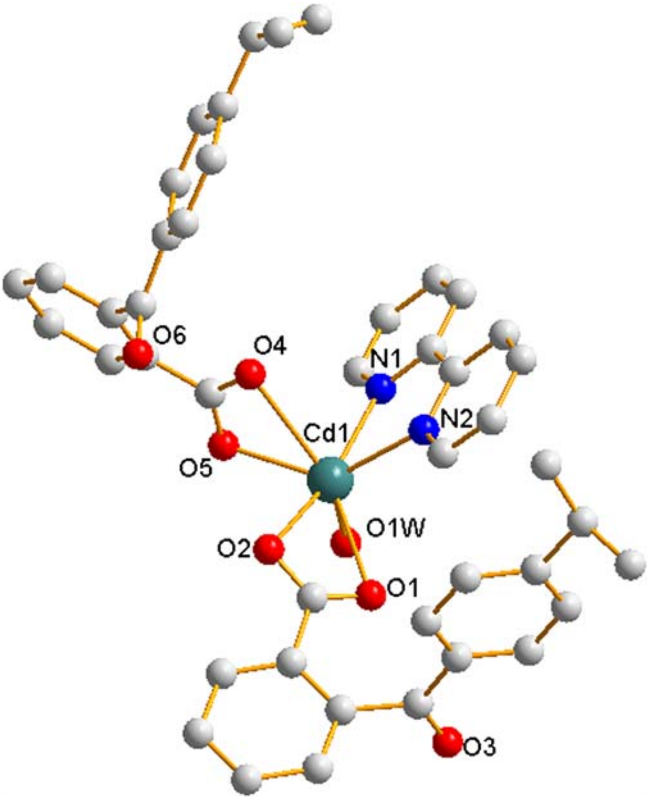

Figure 3. Coordination environment of Cd1 in **2** (all hydrogens and lattice waters are omitted for clarity).

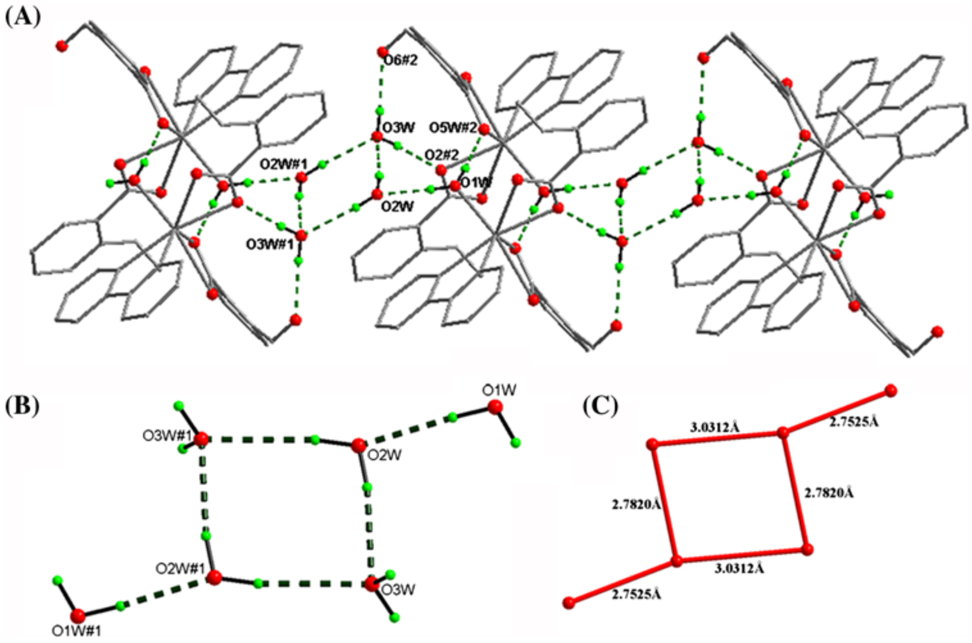

Figure 4. (A) A regular 1-D chain formed by the strong O–H···O hydrogen bonding interactions (symmetry code: #1, –*x*, 1 − *y*, −*z*; #2, –*x*, 1 − *y*, 1 − *z*). (B) View of the hexamer water clusters (symmetry code: #1, –*x*, 1 − *y*, −*z*). (C) The O…O distances of the hydrogen bonding of the hexamer water cluster.

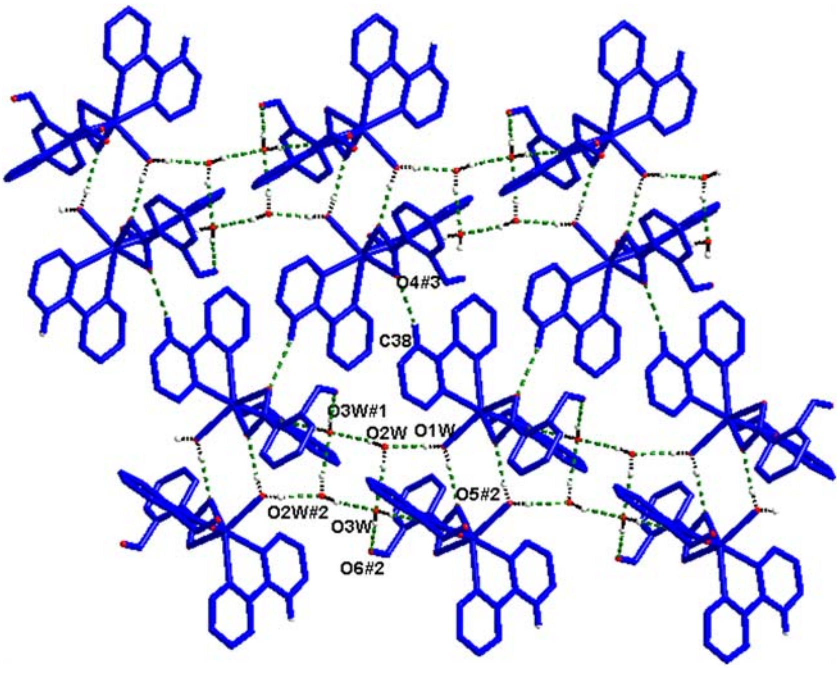

Figure 5. Perspective view of the 2-D supramolecular layer in **2** (symmetry code: #1, –*x*, 1 − *y*, −*z*; #2, −*x*, 1 − *y*, 1 − *z*; #3, *x*, 1.5 − *y*, −0.5 + *z*).

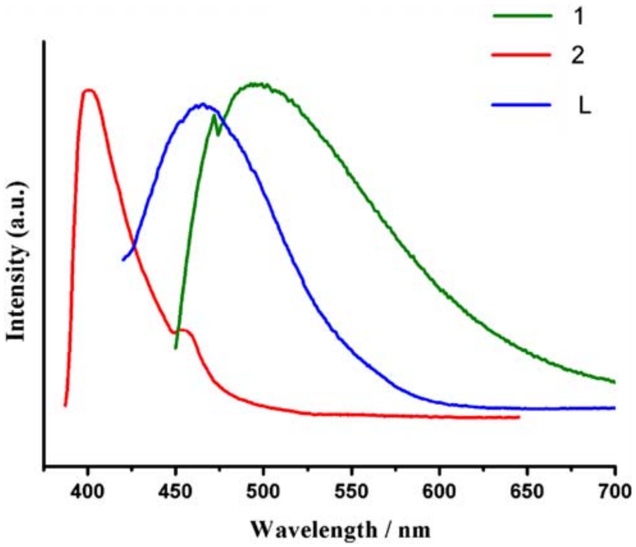

Figure 6. The solid state luminescent spectra of **1**, **2,** and HL ligand measured at room temperature.

and 510 nm ($\lambda_{ex} = 414$ nm) for **1**, 405 nm and 462 (shoulder) ($\lambda_{ex} = 395$ nm) for **2** and 465 nm ($\lambda_{ex} = 380$ nm) for HL, respectively. By comparing with the solid state luminescent spectra of free 4,4′-bpy (figure S4), 2,2′-bpy (figure S5) and HL, the emissions at 478 nm in **1**, 405 nm and 462 in **2** are ascribed to intraligand charge transfer of coordinated 4,4′-bpy, 2,2′-bpy, and HL because of the similarity to the emissions of free 4,4′-bpy, 2,2′-bpy, and HL. The emission at 510 nm in **1** is neither metal-to-ligand charge transfer (MLCT) nor ligand-to-metal charge transfer, but is assigned to ligand-to-ligand charge transitions since Zn(II) ions are difficult to be oxidized or reduced [33,34]. The green emission of **1** and blue emission of **2** indicate that the two compounds may be candidates as photoactive materials.

3.4. *Thermal analysis*

The thermal stabilities of **1** and **2** were studied by TGA and are recorded in figure S3. For **1**, the first weight loss of 1.72% occurred at 100 °C, corresponding to release of hydroxyl (Calcd 1.54%); the second step from 320 to 685 °C is due to release of organic species (Obsd. 86.88%, Calcd. 87.09%). For **2**, a gradual weight loss from 243 to 297 °C corresponds to loss of water in the cluster (Obsd. 6.58%, Calcd. 6.37%). The temperature required for water molecule removal is well above 100 °C, suggesting that the hydrogen bonding that builds the supramolecular structure must be strong and that waters are strongly incorporated as part of the cluster.

4. Conclusion

Two new supramolecular coordination compounds, $[Zn_2(L)_3(4,4'\text{-bpy})(OH)]_n$ (**1**) and $[Cd(L)_2(2,2'\text{-bpy})(H_2O)]\cdot 2H_2O$ (**2**), were synthesized and characterized. **1** and **2** exhibit a 1-D chain and 0-D discrete structure, respectively. Hydrogen-bonding interactions play an important role in formation of 2-D supramolecular layers in **1** and **2**. Luminescent properties show that **1** and **2** have green and blue fluorescence, which indicate that they may be candidates as luminescent materials.

Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center. CCDC numbers are 827488 and 861297 for **1** and **2**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB21EZ, UK (Fax: +44 1223 336033; Email: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

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