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A series of 2D coordination complexes constructed from a flexible bis(methylbenzimidazole) ligand and different dicarboxylates

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ABSTRACT

Five coordination complexes $[Cd(hbmb)(tbi)]_n$ (**1**), $[Cd(hbmb)(adi)]_n$ (**2**), $[Zn(hbmb)_{0.5}(atda)]_n$ (**3**), $\{[Zn_2(hbmb)(suc)_{1.5}(OH)]\cdot 2H_2O\}_n$ (**4**), and $\{[Zn_2(hbmb)(fum)_{1.5}(OH)]\cdot 2H_2O\}_n$ (**5**) (H_2tbi = 5-tert-butylisophthalic acid, H_2adi = adipic acid, H_2atda = 1,3-adamantanediacetic acid, H_2suc = succinic acid, H_2fum = fumaric acid, hbmb = 1,1′-(1,6-hexane)bis-(2-methylbenzimidazole)) have been hydrothermally synthesized and structurally characterized. Complex **1** is an undulated 2D structure with $(6^3)_2$ topology, containing left- and right-handed helical chains. Complex **2** possesses a puckered two-dimensional (2D) network with $4^4{\cdot}6^2$ topologies symbol. Complex **3** exhibits a 2D (4, 4) parallelogram network and also owns left- and right-handed helical chains. Complexes **4** and **5** display 2D rectangular grids and can be simplified as a four-connected $4^4{\cdot}6^2$ topological network. Moreover, the photoluminescence properties and thermal stabilities of the five complexes were investigated.

1. Introduction

Great attention in coordination complexes has been attracted owing to their intriguing structural motifs and their potentials as functional materials in catalysis, optics, magnetism, molecular adsorption, molecular recognition, etc. [1–9]. The design of effective ligands and the proper choice of metal centers are crucial to the construction and structural tuning of the resulting complexes [10,11]. Although significant progress has been achieved in the design and preparation of coordination complexes [12], it is still difficult to predict the exact structures of the final frameworks [13], because of the fact that the crystallization process of coordination complexes depends on various factors, such as the coordination nature of ligand, the solvent systems, counter ions, etc. [14,15]. Hence, further studies on this topic are required for understanding the roles of these factors in the formation of coordination complexes [13].

The employment of the “mix-ligand” synthetic strategies has gradually become an effective method for the construction of coordination complexes [16–22]. Up to now, polycarboxylate ligands have been proven to be excellent structural constructors owing to their diverse coordination modes to metal ions and the strong coordinating ability of carboxylates [23–25]. On the other hand, flexible ligands have been extensively used to construct metal complexes [26–32]. Such ligands bearing alkyl spacers are a good choice of N-donor ligands, and hold the energetic minimum, the flexible nature of spacers allows the ligands to bend and rotate when it coordinates to metal centers, and this often causes the structural diversities [33–35]. In previous contributions, we have reported lots of interesting structures based on flexible N-donor ligands 1,4-bis-(2-methylbenzimidazol-1-ylmethyl) benzene (bmb) [36,37] and 1,4-bis(1,2,4-triazol-1-ylmethyl)-2,3,5,6-tetramethylbenzene (btmx) [38].

To further investigate the influence of flexibility and conformational freedom from a $–CH_2–$ spacer on the resultant structure of complexes [39,40], we choose the longer flexible bis(methylbenzimidazole) ligand hbmb (Scheme S1) as organic linker in the presence of different dicarboxylates as coligands (Scheme S2) in this paper. Five different structural coordination complexes, $[Cd(hbmb)(tbi)]_n$ (**1**), $[Cd(hbmb)(adi)]_n$ (**2**), $[Zn(hbmb)_{0.5}(atda)]_n$ (**3**), $\{[Zn_2(hbmb)(suc)_{1.5}(OH)]\cdot 2H_2O\}_n$ (**4**), and $\{[Zn_2(hbmb)(fum)_{1.5}(OH)]\cdot 2H_2O\}_n$ (**5**) (H_2tbi = 5-tert-butylisophthalic acid, H_2adi = adipic acid, H_2atda = 1,3-adamantanediacetic acid, H_2suc = succinic acid, H_2fum = fumaric acid, hbmb = 1,1′-(1,6-hexane)bis-(2-methylbenzimidazole)) have been obtained. Fluorescence and thermal stability have been carried out on these complexes.

2. Experimental section

2.1. General information and materials

All reagents and solvents were commercially available except for hbmb, which was synthesized according to the literature [41]. FT-IR spectra were recorded on a Bruker-ALPHA spectrophotometer with KBr pellets in 400–4000 cm^{-1} region. Elemental analyses (C, H, and N) were carried out on a FLASH EA 1112 elemental analyzer. Powder

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X-ray diffraction (PXRD) patterns were recorded using Cu Kα_1 radiation on a PANalytical X'Pert PRO diffractometer. Thermal analyses were performed on a Netzsch STA 449C thermal analyzer from room temperature at a heating rate of 10 °C min^{-1} in air. The luminescence spectra for the powdered solid samples were measured at room temperature on a Hitachi 850 fluorescence spectrophotometer. The excitation slit and emission slit were both 2.0 nm.

2.2. Synthesis

2.2.1. [Cd(hbmb)(tbi)]$_n$ (**1**)

A mixture of $Cd(NO_3)_2 \cdot 4H_2O$ (0.2 mmol), hbmb (0.1 mmol), H_2tbi (0.2 mmol), NaOH (0.4 mmol) and H_2O (8 mL) was placed in a 25 mL Teflon-lined stainless steel container. The mixture was sealed and heated at 160 °C for three days. After the mixture was cooled to ambient temperature at a rate of 5 °C/h, colorless crystals of **1** were obtained with a yield of 75% (based on Cd). *Anal.* Calc. for $C_{34}H_{38}CdN_4O_4$: C, 60.13, H, 5.64, N, 8.25. Found: C, 60.14, H, 5.63, N, 8.27%. IR (KBr, cm^{-1}): 3451 (m), 3058 (w), 2955 (m), 1707 (w), 1608 (s), 1556 (s), 1415 (s), 1367 (s), 1272 (m), 1162 (w), 932 (w), 747 (s).

2.2.2. [Cd(hbmb)(adi)]$_n$ (**2**)

A mixture of $Cd(NO_3)_2 \cdot 4H_2O$ (0.2 mmol), hbmb (0.1 mmol), H_2adi (0.2 mmol), NaOH (0.4 mmol) and H_2O (8 mL) was placed in a 25 mL Teflon-lined stainless steel container. The mixture was sealed and heated at 150 °C for three days. After the mixture was cooled to ambient temperature at a rate of 5 °C/h, colorless crystals of **2** were obtained with a yield of 70% (based on Cd). *Anal.* Calc. for $C_{28}H_{34}CdN_4O_4$: C, 55.77; H, 5.68; N, 9.29. Found: C, 55.76; H, 5.69; N, 9.30%. IR (KBr, cm^{-1}): 3422 (w), 3060 (m), 2951 (m), 2860 (m), 1555 (s), 1412 (s), 1304 (m), 1237 (m), 1121 (w), 1013 (w), 928 (w), 852 (w), 759 (s).

2.2.3. [Zn(hbmb)$_{0.5}$(atda)]$_n$ (**3**)

A mixture of $Zn(Ac)_2 \cdot 2H_2O$ (0.2 mmol), hbmb (0.1 mmol), H_2atda (0.2 mmol), and NaOH (0.2 mmol) in H_2O (10 mL) was kept in a 25 mL Teflon-lined stainless steel vessel at 160 °C for three days. After the mixture was cooled to room temperature at a rate of 5 °C/h, colorless crystals suitable for X-ray diffraction were obtained with a yield of 65% (based on Zn). *Anal.* Calc. for $C_{25}H_{31}N_2O_4Zn$: C, 61.41; H, 6.39; N, 5.72. Found: C, 61.42; H, 6.39; N, 5.71%. IR (KBr, cm^{-1}): 3426 (w), 3060 (w), 2925 (s), 2894 (s), 2846 (m), 1591 (s), 1408 (s), 1298 (m), 1239 (w), 1158 (w), 1016 (w), 942 (w), 755 (s).

2.2.4. {[Zn$_2$(hbmb)(suc)$_{1.5}$(OH)]·2H$_2$O}$_n$ (**4**)

A mixture of $Zn(Ac)_2 \cdot 2H_2O$ (0.2 mmol), hbmb (0.1 mmol), H_2suc (0.2 mmol), and NaOH (0.4 mmol) in H_2O (8 mL) was kept in a 25 mL Teflon-lined stainless steel vessel at 160 °C for three days. After the mixture was cooled to room temperature at a rate of 5 °C/h, colorless crystals of **4** were obtained with a yield of 80% (based on Zn). *Anal.* Calc. for $C_{14}H_{18}N_2O_{4.5}$ Zn: C, 47.74; H, 5.29; N, 7.95. Found: C, 47.75; H, 5.29; N, 7.94%. IR (KBr, cm^{-1}): 3438 (m), 3050 (w), 2929 (m), 2860 (w), 1578 (s), 1409 (s), 1014 (m), 936 (w), 806 (w), 754 (s).

2.2.5. {[Zn$_2$(hbmb) (fum)$_{1.5}$(OH)]·2H$_2$O}$_n$ (**5**)

The procedure is similar to that of **2**, except that $Zn(Ac)_2 \cdot 2H_2O$ was used instead of $Cd(NO_3)_2 \cdot 4H_2O$ and H_2fum was used instead of H_2adi. Yield, 50% (based on Zn). *Anal.* Calc. for $C_{14}H_{16.5}N_2O_{4.5}Zn$: C, 47.94; H, 4.88; N, 7.98. Found: C, 47.93; H, 4.87; N, 7.97%. IR (KBr, cm^{-1}): 3429 (m), 3052 (w), 2933 (w), 2860 (w), 1591 (s), 1460 (m), 1370 (s), 990 (w), 802 (w), 754 (m).

2.3. Single-crystal X-ray diffraction measurements

All data were collected on a Rigaku Saturn 724 detector with graphite monochromatic Mo Ka radiation (λ = 0.71073 Å) at temperature of 20 ± 1 °C. Absorption corrections were applied by using multi-scan program. The data were corrected for Lorentz and polarization effects. The structures were solved by direct methods and refined with a full-matrix least-squares technique based on F^2 with the SHELXL-97 crystallographic software package [42]. All of the non-hydrogen atoms were refined anisotropically. The hydrogen atoms were assigned with common isotropic displacement factors and included in the final refinement by using geometrical restrains. Crystallographic crystal data and structure processing parameters for **1**–**5** are summarized in Table 1. Selected bond lengths and bond angles of **1**–**5** are listed in Table 2.

3. Results and discussion

3.1. Structure description

3.1.1. Crystal structure of complex **1**

Single crystal X-ray diffraction analysis shows that **1** crystallizes in the acentric orthorhombic space group *Pna*2$_1$ which belongs to the crystal class *mm*2, where optical activity can occur as specific physical effects [46]. As shown in Fig. 1a, Cd1(II) ion adopts a distorted octahedral coordination environment [47,48], defined by four oxygen atoms from two tbi^{2-} anions and two nitrogen atoms from two hbmb. It can be clearly seen that the Cd2 atom is five coordinated by three oxygen atoms from two tbi^{2-} anions and two nitrogen atoms from two hbmb, taking a distorted square-pyramidal geometry (τ = 0.13) [49,50]. The distances of Cd–O/N bond range from 2.177(7) to 2.572(8) Å, which are similar to other Cd-based complexes [51]. Interestingly, there are two kinds of independent tbi^{2-}. Two carboxylate groups of one kind of tbi^{2-} anions both adopt bisdentate chelated coordination mode. The other kind of tbi^{2-} anions act as a μ_2-bridge, in which one carboxylate group adopts a bidentate chelate mode and the other adopts a monodentate coordination mode. Two kinds of tbi^{2-} anions are connected by Cd(II) atoms alternately giving rise to a 1D zigzag chain (Fig. 1b). The hbmb ligand adopts asymmetric *trans*-conformation with two different $N_{donor} \cdots N\text{–}C_{sp3} \cdots C_{sp3}$ torsion angles of 71.406° and 97.303°, bridging two Cd(II) atoms forming a 21-membered rings (Fig. 1c). The 1D chains containing 21-membered rings are further extended by hbmb ligands into a 2D layer structure (Fig. 1d). The hbmb ligand linking the 1D chains also adopts asymmetric *trans*-conformation with two different $N_{donor} \cdots N\text{–}C_{sp3\text{-}} \cdots C_{sp3}$ torsion angles of 77.056° and 88.484°, and the bridged Cd···Cd distances along μ-hbmb are 13.1275 Å between two adjacent 1D chains. It's worth noting that **1** crystallizes in the non-centrosymmetric polar space group *Pna*2$_1$. Accordingly, all bis-methylbenzimidazole ligands are oriented in the same direction [52–55], as shown in Fig. 1d. Interestingly, every two hbmb and two tbi^{2-} anions are alternately linked by adjacent Cd(II) ions to form left- and right-handed helical chains. While the helical pitch of left-handed helices is 17.9650(4) Å corresponding to the length of *b*-axis, and the Cd···Cd separation across tbi^{2-} and hbmb bridge is 9.2131 Å and 9.0227 Å, separately (Fig. 1e-left). The helical pitch of right-handed helices is 19.2040(4) Å corresponding to the length of *c*-axis, and the bridged Cd···Cd distances along μ-tbi^{2-} and μ-hbmb are 9.2131 and 13.1275 Å, respectively (Fig. 1e-right).

As depicted in Fig. 1f, topological analysis is performed on **1**. If the Cd(II) ion were treated as a 3-connector. hbmb, tbi^{2-} and 21-membered rings are defined as linkers, respectively, the undulated 2D framework of **1** can be described as $(6^3)_2$ topology.

Table 1
Crystallographic data and structure refinement details for **1**–**5**.

Complexes	1	2	3	4	5
Formula	$C_{34}H_{38}CdN_4O_4$	$C_{28}H_{34}CdN_4O_4$	$C_{25}H_{31}N_2O_4Zn$	$C_{28}H_{37}N_4O_9Zn_2$	$C_{28}H_{34}N_4O_9Zn_2$
Formula weight	679.08	602.99	488.89	704.40	701.37
T (K)	293(2)	293(2)	293(2)	293(2)	293(2)
λ (Mo K), (Å)	0.71073	0.71073	0.71073	0.71073	0.71073
Crystal system	Orthorhombic	Orthorhombic	Monoclinic	Triclinic	Triclinic
Space group	$Pna2_1$	$Pbcn$	$P2_1/c$	$P\bar{1}$	$P\bar{1}$
a (Å)	19.072(4)	9.2186(18)	10.836(2)	11.143(2)	11.093(2)
b (Å)	17.965(4)	17.128(3)	18.097(4)	12.284(3)	12.359(3)
c (Å)	19.204(4)	17.041(3)	11.340(2)	12.748(3)	12.561(3)
α (°)	90	90	90	69.85(3)	70.98(3)
β (°)	90	90	92.98(3)	84.94(3)	84.67(3)
γ (°)	90	90	90	72.46(3)	71.61(3)
V (Å^3)	6580(2)	2690.7(9)	2220.9(8)	1561.8(5)	1544.8(6)
Z	4	4	4	2	2
$2\theta_{max}$ (°)	25.00	25.00	25.00	25.00	25.00
D_{calc} (g cm^{-3})	1.371	1.489	1.462	1.496	1.495
Absorption coefficient (mm^{-1})	0.706	0.852	1.141	1.592	1.609
F(000)	2800	1240	1028	728	712
Reflections collected	67437	15287	23075	13153	15177
Unique reflections	11591	2369	4127	5807	5400
R_{int}	0.0937	0.0474	0.0516	0.0367	0.0813
Goodness-of-fit (GOF)	1.121	1.024	0.964	0.904	0.892
R_1 [$I > 2\sigma(I)$][a]	0.0849	0.0789	0.0554	0.0589	0.0856
wR_2 (all data) [b]	0.1995	0.1877	0.1357	0.1619	0.2021
Flack parameter[c]	0.00(5)	–	–	–	–

[a] $R_1 = ||F_o| - |F_c||/|F_o|$.

[b] $wR_2 = [w(|F_o^2| - |F_c^2|)^2/w|F_o^2|^2]^{1/2}$. $w = 1/[\sigma^2(F_o)^2 + 0.0297P^2 + 27.5680P]$, where $P = (F_o^2 + 2F_c^2)/3$.

[c] Absolute structure parameter [43–45].

3.1.2. Crystal structure of complex **2**

When H_2tbi was replaced by H_2adi, a structurally different complex **2** was obtained. The asymmetric unit of **2** consists of half of Cd(II) ion, half of hbmb ligand, half of adi^{2-} anion. As shown in Fig. 2a, the Cd(II) ion adopts a distorted octahedral geometry via coordinating with four oxygen atoms (O1, O2, O1A, O2A) from two adi^{2-} anions and two nitrogen atoms (N1 and N1A) from two hbmb ligands. The Cd–O bond distances vary from 2.346(2) to 2.3950(18) Å, while the Cd–N bond length is 2.2876(18) Å. They are in conformity with those found in reported corresponding cadmium complexes [51]. hbmb adopts symmetric *trans*-conformation with the $N_{donor}\cdots N–C_{sp3}\cdots C_{sp3}$ torsion angle of 94.033°. The adi^{2-} anions adopt symmetric *cis*-conformation.

In **2**, each hbmb ligand bridges two Cd(II) ions to generate a 1D zigzag chain with a Cd···Cd distance of 16.7491(18) Å along the *a*-axis (Fig. 2b). The adi^{2-} anions connect two adjacent Cd(II) ions using its two carboxylate groups in a bisdentate chelated coordination mode, resulting in a 1D wave-like chain (Fig. 2c). The interlaced connection of Cd/hbmb and Cd/adi^{2-} chains constructs the 2D network by sharing Cd(II) ions (Fig. 2d). A better insight into such an elegant framework can be obtained by the topological approach (Fig. 2e). The Cd(II) ions can be simplified as 4-connected, and hbmb and adi^{2-} anions serve as linkers. Hence, the 2D network of **2** can be described as a 4-connected topology with a point symbol of $4^4{\cdot}6^2$.

3.1.3. Crystal structure of complex **3**

Single crystal X-ray diffraction analysis reveals that **3** crystallizes in monoclinic space group $P2_1/c$. As shown in Fig. 3a, each Zn(II) atom is five-coordinated by one nitrogen atom (N1) from one hbmb and four oxygen atoms (O1, O2, O3, O4) from three H_2atda. The structural index parameter (τ) [56] is 0.58, indicating that the geometry around Zn(II) is best described as a trigonal bipyramidal distorted square-based pyramid (TBPDSBP) [56,57]. The Zn–O distances are in the range of 1.968(3)–2.345(5) Å, while the Zn–N bond length is 2.081(3) Å, which are in the normal range [58]. hbmb adopts a symmetric *trans*-conformation with the $N_{donor}\cdots N–C_{sp3}\cdots C_{sp3}$ torsion angles of 91.916°. Ligand H_2atda adopts asymmetric *trans*-conformation.

In **3**, two carboxylate groups of H_2atda adopt different coordination modes: one carboxylate group adopts bidentate chelating mode and the other adopts μ_2-η^1: η^1-bridging mode. The atda^{2-} anions act as μ_3-bridge linking Zn(II) ions to produce a one-dimensional (1D) double-chain structure having 8- and 20-membered rings (Fig. 3b). The Zn···Zn distance is 3.8540 and 9.6667 Å in the two types of rings, respectively. Such 1-D chains are further interlinked by flexible hbmb ligands to form a 2D layer (Fig. 3c). The bridged Zn···Zn distance along μ-hbmb is 12.8278 Å. As exhibited in Fig. 3d, every one hbmb and one H_2atda are in turn connected by Zn(II) ions to form left- and right-handed helical chains. Both of the helical pitches are 11.340(2) Å corresponding to the length of *c*-axis. From a topological perspective, if the $[ZnC_2O_4]_2$ units are regarded as 4-connected nodes, and hbmb and atda^{2-} anions are defined as linkers, respectively, the 2D framework of **3** can be described as $4^4{\cdot}6^2$ topology (Fig. 3e).

3.1.4. Crystal structure of complex **4** and complex **5**

X-ray single crystal diffraction reveals that complexes **4** and **5** are isomorphous. Herein, only the structure of **4** is discussed in detail. **4** crystallizes in the triclinic space group $P\bar{1}$ and has two crystallographically independent Zn(II) center. As shown in Fig. 4a, Zn1 is five-coordinated, with a distorted square-pyramidal geometry ($\tau = 0.39$) [49,50], by one nitrogen atom (N3) of one hbmb ligand, three carboxylate oxygen atoms (O3, O4, O5) from two suc^{2-} anions, and one oxygen atom (O1) from one hydroxyl group. However, the Zn2 atom adopts a distorted tetrahedral coordination environment, defined by one nitrogen atom (N1) of one hbmb ligand, two carboxylate oxygen atoms (O2, O7) from two suc^{2-} anions, and one oxygen atom (O1) from one hydroxyl group. All the Zn–O and Zn–N bond lengths fall in the normal range except that the Zn(2)–O(6) distance is 2.585(4) Å (Fig. 4a), suggesting a non-negligible interaction with the uncoordinated carboxylate oxygen atom, which can be described as a semi-chelating coordination mode [59,60].

Table 2
Selected bond lengths (Å) and bond angles (°) for **1**–**5**.

Complex **1**					
Cd(1)–O(4)	2.265(6)	Cd(1)–O(1)	2.272(7)	Cd(1)–O(2)	2.459(7)
Cd(1)–O(3)	2.487(7)	Cd(2)–O(6)#1	2.177(7)	Cd(2)–O(7)	2.228(7)
Cd(2)–O(8)	2.572(8)	Cd(1)–N(1)	2.301(8)	Cd(1)–N(3)	2.313(9)
Cd(2)–N(7)	2.278(8)	Cd(2)–N(5)	2.300(9)	O(4)–Cd(1)–O(1)	146.5(3)
O(4)–Cd(1)–N(1)	93.3(3)	O(1)–Cd(1)–N(1)	111.5(3)	O(4)–Cd(1)–N(3)	96.2(3)
O(1)–Cd(1)–N(3)	102.2(3)	N(1)–Cd(1)–N(3)	97.5(3)	O(4)–Cd(1)–O(2)	101.0(2)
O(1)–Cd(1)–O(2)	55.3(2)	N(1)–Cd(1)–O(2)	97.1(3)	N(3)–Cd(1)–O(2)	156.7(3)
Complex **2**					
Cd(1)–N(1)	2.2876(18)	Cd(1)–N(1)#1	2.2876(18)	Cd(1)–O(2)	2.346(2)
Cd(1)–O(2)#1	2.346(2)	Cd(1)–O(1)#1	2.3950(18)	Cd(1)–O(1)	2.3950(17)
N(1)–Cd(1)–N(1)#1	96.20(9)	N(1)–Cd(1)–O(2)	95.25(7)	N(1)#1–Cd(1)–O(2)	148.48(7)
N(1)–Cd(1)–O(2)#1	148.48(7)	N(1)#1–Cd(1)–O(2)#1	95.25(7)	O(2)–Cd(1)–O(2)#1	90.06(10)
N(1)–Cd(1)–O(1)#1	116.41(6)	N(1)#1–Cd(1)–O(1)#1	94.27(6)	O(2)–Cd(1)–O(1)#1	54.44(6)
O(2)#1–Cd(1)–O(1)#1	91.84(7)	N(1)–Cd(1)–O(1)	94.27(6)	N(1)#1–Cd(1)–O(1)	116.41(6)
O(2)–Cd(1)–O(1)	91.84(7)	O(2)#1–Cd(1)–O(1)	54.44(6)	O(1)#1–Cd(1)–O(1)	134.24(8)
Complex **3**					
Zn(1)–O(3)	1.968(3)	Zn(1)–O(4)	1.976(3)	Zn(1)–O(2)	2.046(4)
Zn(1)–O(1)	2.345(5)	Zn(1)–N(1)	2.081(3)	O(3)–Zn(1)–O(4)	120.01(13)
O(3)–Zn(1)–O(2)	120.46(16)	O(4)–Zn(1)–O(2)	113.50(16)	O(3)–Zn(1)–N(1)	100.02(12)
O(4)–Zn(1)–N(1)	95.33(12)	O(2)–Zn(1)–N(1)	99.13(14)	O(3)–Zn(1)–O(1)	97.95(16)
O(4)–Zn(1)–O(1)	90.28(18)	O(2)–Zn(1)–O(1)	56.77(15)	N(1)–Zn(1)–O(1)	155.22(14)
Complex **4**					
Zn(1)–O(1)	1.914(4)	Zn(1)–O(3)	2.020(4)	Zn(2)–O(2)	2.017(4)
Zn(1)–O(5)	2.101(4)	Zn(1)–O(4)	2.304(5)	Zn(2)–O(1)	1.907(4)
Zn(2)–O(7)#1	1.971(4)	Zn(2)–N(1)	2.016(4)	Zn(1)–N(3)	2.024(4)
O(1)–Zn(1)–O(3)	101.62(17)	O(1)–Zn(1)–N(3)	119.04(18)	O(3)–Zn(1)–N(3)	96.06(18)
O(1)–Zn(1)–O(5)	124.69(19)	O(3)–Zn(1)–O(5)	90.2(2)	N(3)–Zn(1)–O(5)	112.90(19)
O(1)–Zn(1)–O(4)	97.99(17)	O(3)–Zn(1)–O(4)	148.44(18)	N(3)–Zn(1)–O(4)	95.72(18)
O(5)–Zn(1)–O(4)	58.22(18)	O(1)–Zn(2)–O(7)#1	122.13(19)	O(1)–Zn(2)–N(1)	120.90(17)
O(7)#1–Zn(2)–N(1)	111.46(18)	O(1)–Zn(2)–O(2)	100.39(18)		
Complex **5**					
Zn(1)–O(1)	1.9027(14)	Zn(2)–O(6)	2.0338(16)	Zn(1)–O(3)	2.0131(18)
Zn(1)–O(4)	2.0024(15)	Zn(2)–O(1)#1	1.9107(13)	Zn(2)–O(7)	2.4397(18)
Zn(2)–O(5)#1	2.0242(15)	Zn(1)–N(1)	1.9994(17)	Zn(2)–N(4)#2	2.0033(18)
O(1)–Zn(1)–N(1)	119.52(7)	O(1)–Zn(1)–O(3)	126.83(7)	N(1)–Zn(1)–O(3)	109.06(7)
O(1)–Zn(1)–O(4)	100.14(6)	N(1)–Zn(1)–O(4)	96.95(6)	O(3)–Zn(1)–O(4)	93.88(7)
O(1)#1–Zn(2)–N(4)#2	119.17(7)	O(1)#1–Zn(2)–O(5)#1	102.70(6)	N(4)#2–Zn(2)–O(5)#1	97.90(7)
O(1)#1–Zn(2)–O(6)	123.52(7)	N(4)#2–Zn(2)–O(6)	112.39(7)	O(5)#1–Zn(2)–O(6)	91.20(7)
O(1)#1–Zn(2) –O(7)	95.86(6)	N(4)#2–Zn(2)–O(7)	94.83(7)	O(5)#1–Zn(2)–O(7)	148.55(6)

Symmetry transformations used to generate equivalent atoms:
for **1**: #1 *x*,*y*–1,*z* #2 –*x* + 1,–*y* + 1, *z*–1/2 #3 –*x* + 1, –*y* + 1, *z* + 1/2 #4 *x*, *y* + 1, z.
for **2**: #1–*x* + 1, *y*, –*z* + 1/2 #2 –*x*, *y*, –*z* + 1/2 #3 –*x* + 2, –*y*, –*z*.
for **3**: #1 –*x* + 1, –*y* + 2,–*z* #2 –*x*, –*y* + 2, –*z* #3 –*x*, –*y* + 2, –*z* + 1.
for **4**: #1 *x* + 1, *y*, *z* #2 –*x* + 2, –*y* + 1, –*z* + 1 #3 –*x* + 2,–*y* + 2,–*z* #4 *x*–1, *y*, *z*.
for **5**: #1 *x*–1, *y*, *z* #2 –*x*,–*y*,–*z* + 1 #3 –*x* + 1,–*y*–1,–*z* + 2 #4 *x* + 1, *y*, *z*.

In **4**, *trans*-suc^{2-} anions adopt different coordination modes: two carboxylate groups of one kind of suc^{2-} anions adopts bidentate and monodentate chelating modes, respectively, as well as two carboxylate groups of the other kind of suc^{2-} anions all adopts μ_2-η^1: η^1-bridging mode. Each pair of Zn(II) ions is bridged by one carboxylate group of suc^{2-} anions and one oxygen atom from one coordinated water molecule to product a dinuclear unit with a Zn···Zn separation of 3.3035 Å. The dinuclear units $[Zn_2(CO_2)O]$ are further extended by suc^{2-} anions into a 1D ladder chain along the *c* direction (Fig. 4b). The 1D chains are further stacked by the linking of hbmb adopting asymmetric *cis*-conformation with two different N_{donor}···N–C_{sp3}···C_{sp3} torsion angles of 78.505° and 102.176°, thus leading to the formation of a 2D framework (Fig. 4c-left). In addition, two hbmb ligands and two $[Zn_2(CO_2)O]$ units construct a 30-membered rings (Fig. 4c-right). The Zn···Zn distance across hbmb is 6.1026 Å. Topological analysis was carried out to get insight into the structure of **4**. If the dinuclear units $[Zn_2(CO_2)O]$ is simplified as a 4-connected node and the hbmb as well as the suc^{2-} ligand are considered as linkers, the 2D rectangular grids can be described as a $4^4 \cdot 6^2$ topology (Fig. 4d). Furthermore, the adjacent 2D layers are extended into a 3D supramolecular framework (Fig. 4e) by the π···π stacking interactions with the centroid-to-centroid separation of 3.6168 Å between neighboring imidazole rings and phenyl rings from benzimidazoles (Fig. 4f) [61].

3.2. Effects of hbmb and dicarboxylates on the structures of complexes 1–5

Based on character of the flexible ligand, hbmb can exhibit *cis*/*trans*-conformation and symmetric (with the same N_{donor}···N–C_{sp3}···C_{sp3} torsion angles of two methylbenzimidazol arms)/asymmetric (with different N_{donor}···N–C_{sp3}···C_{sp3} torsion angles of two methylbenzimidazol arms) conformations. From the structural description above, hbmb presents asymmetric *trans*-conformation in **1**, symmetric *trans*-conformation in **2**–**3** and asymmetric *cis*-conformation in **4**–**5**. All of the N_{donor}···N-C_{sp3}···C_{sp3} torsion angles are different in **1**–**5**. The free conformations of -(CH_2)- spacer greatly enrich structures of complexes **1**–**5**. In **1**, hbmb bridges adjacent Cd(II) ions leading to left-handed helices. At the same time, hbmb and H_2tbi are linked by adjacent Cd(II) ions forming right-handed helices. In **2**, each hbmb ligand bridges two Cd(II) ions to generate a 1D zigzag chain. For **3**, hbmb and H_2atda are in turn connected by Zn(II) ions to form left- and right-handed helical chains. For **4**, two hbmb ligands associate two $[Zn_2(CO_2)O]$

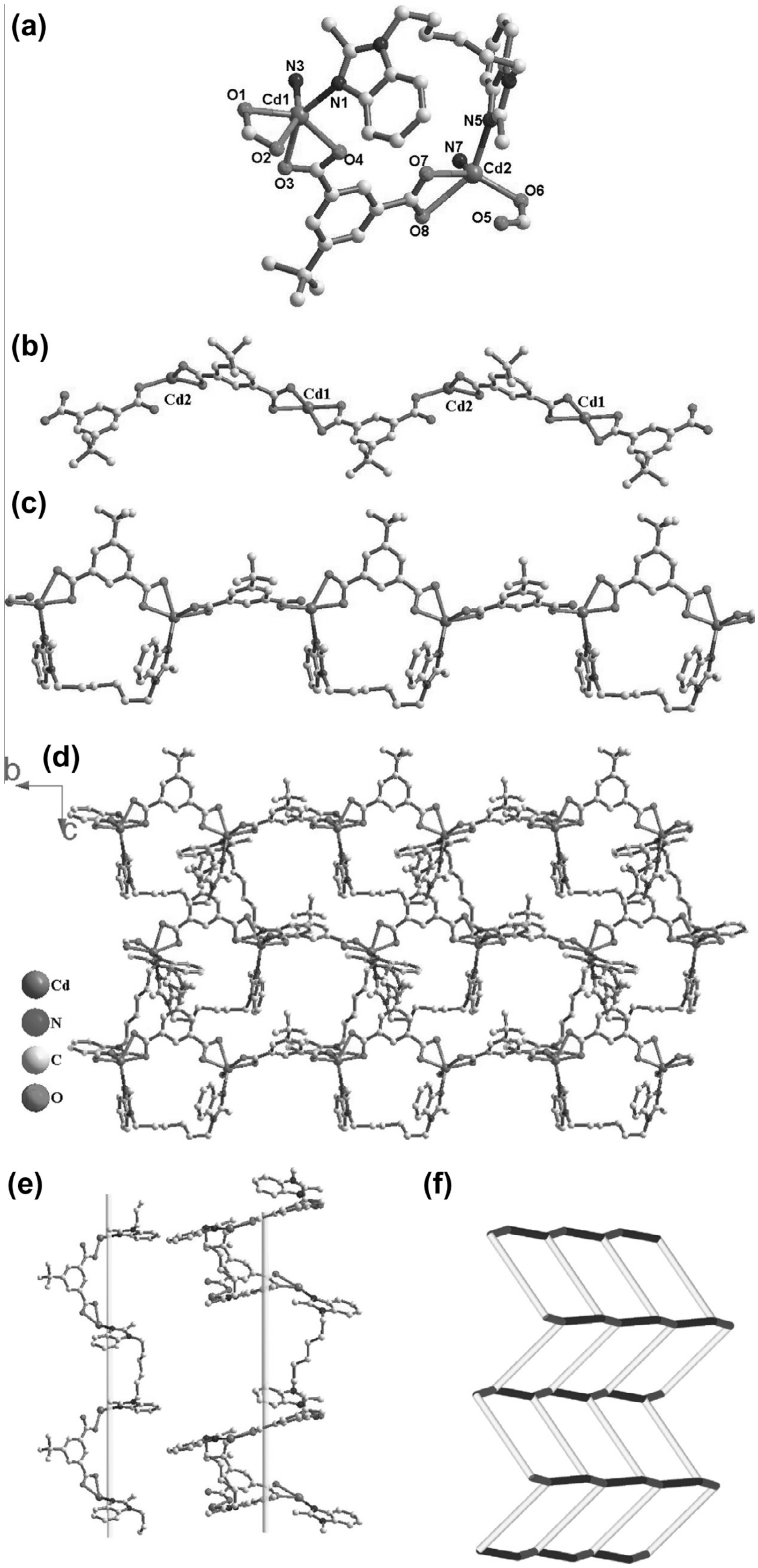

Fig. 1. (a) Coordination environment of Cd (II) ion in **1** with hydrogen atoms omitted for clarity. (b) An infinite 1D zigzag chain constructed from Cd(II) centers and tbi^{2-} anions in **1**. (c) The 1D chains containing 21-membered rings. (d) The 2D layer structure of **1** viewed along the a direction. (e) Views of the Cd(II)/hbmb/tbi^{2-} left-handed helical chain (left) and right-handed helical chain (right). (f) View of the undulating 2D network with $(6^3)_2$ topology in **1**.

units together to construct a 30-membered rings. Two bis(methylbenzimidazole) groups of hbmb can rotate freely when coordinating with central metal ions due to the flexible nature of the –(CH$_2$)– spacers, which leads to the different structure of complexes.

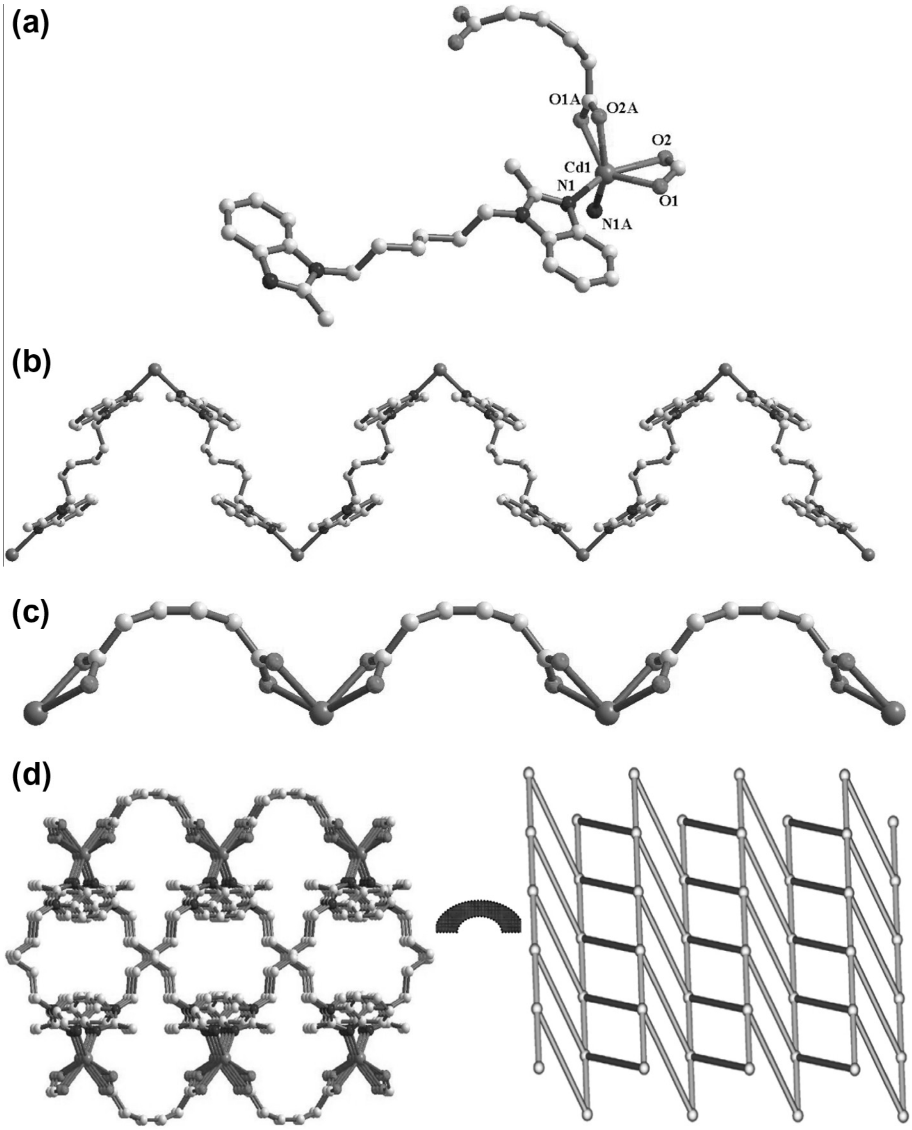

Fig. 2. (a) Perspective view of the coordination environment of Cd(II) ion in **2** (hydrogen atoms are omitted for clarity). (b) The 1D zigzag chain constructed by Zn (II) ions and hbmb. (c) The adi^{2-} anions with *cis*-conformation link the adjoining Cd(II) ions into a 1D wave-like chain. (d) 2D layered structure of complex **2** (left) and schematic representation of a 4-connected topology network with a point symbol of $4^4 \cdot 6^2$ (right).

In this work, we selected five dicarboxylates as auxiliary ligands. The carboxylate anions have great influence on building the final structures of the complexes. In **1**, each carboxylate group of the rigid ligand H_2tbi connects two Cd(II) atoms in bisdentate chelated coordination mode giving rise to a 1D zigzag chain. H_2tbi together with hbmb links adjacent Cd(II) ions forming right-handed helices. In **2**, two carboxylate groups of adi^{2-} anions, which adopt symmetric *cis*-conformation in a bisdentate chelated coordination mode, connect two adjacent Cd(II) ions forming a beautiful 1D wave-like chain. When flexible H_2atda was introduced instead of the rigid ligand H_2tbi, **3** was obtained. In **3**, two carboxylate groups of H_2atda show two different coordination modes: one adopts bidentate chelating mode and the other adopts μ_2-η^1: η^1-bridging mode. The atda^{2-} anion acts as μ_3-bridge linking Zn(II) ions to produce a 1D double-chain structure. Moreover, H_2atda alternately connects Zn(II) ions together with hbmb to form left- and right-handed helical chains. For **4**, suc^{2-} anions in two independent coordination modes bridge the $[Zn_2(CO_2)O]$ units to give rise to a 1D ladder chain. It can be observed that the dicarboxylate coligands have an important effect on the features of resultant complexes.

3.3. XRD results and thermal analyses

To confirm the phase purity of these complexes, the PXRD patterns were recorded for complexes **1**–**5**, and they were comparable to the corresponding simulated ones calculated from the single-crystal diffraction data (Fig. S1 in the Supporting Information), indicating a pure phase of each bulky sample.

The thermal stabilities were investigated as shown in Fig. S2 (Supporting Information). The TGA curve of **1** shows a one step weight loss process from 307 to 579 °C, corresponding to the decomposition of organic components. A CdO residue of 20.65% (Calc. 18.91%) is observed. The framework of **2** remains intact until it is heated to 292 °C, and the CdO residue of 22.56% (Calc. 21.30%) is obtained at 634 °C. The TGA curve of complex **3** exhibits that it is stable up to 352 °C and then loses weight from 352 to 582 °C, corresponding to the decomposition of hbmb and atda^{2-}. The remaining weight

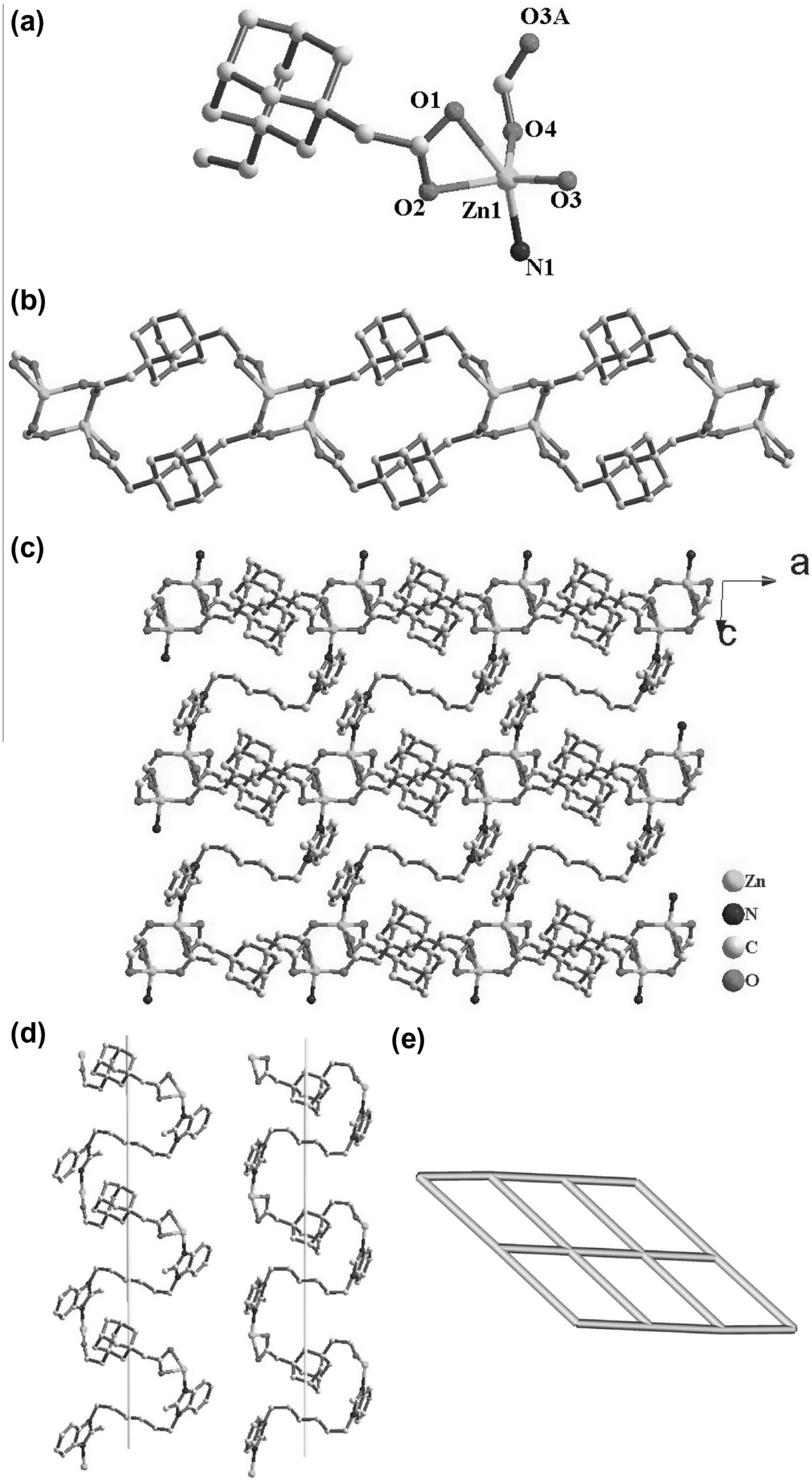

Fig. 3. (a) Coordination environment of Zn (II) ion in **3** with hydrogen atoms omitted for clarity. (b) The 1D double-chain structure containing 8- and 20-membered rings. (c) The 2D layer structure of **3** viewed along the b direction. (d) The Zn(II)/hbmb/atda^{2-} left- and right-handed helical chains. (e) Schematic description of 2D parallelogram network with $4^4{\cdot}6^2$ topology in **3**.

corresponds to the formation of ZnO (obsd, 15.22%; Calc. 13.50%). For **4**, the TGA shows that the first weight loss of 5.36% in the range of 65–112 °C is related to the loss of one lattice water molecule (Calc. 5.12%). The overall framework of **4** begins to decompose from 319 to

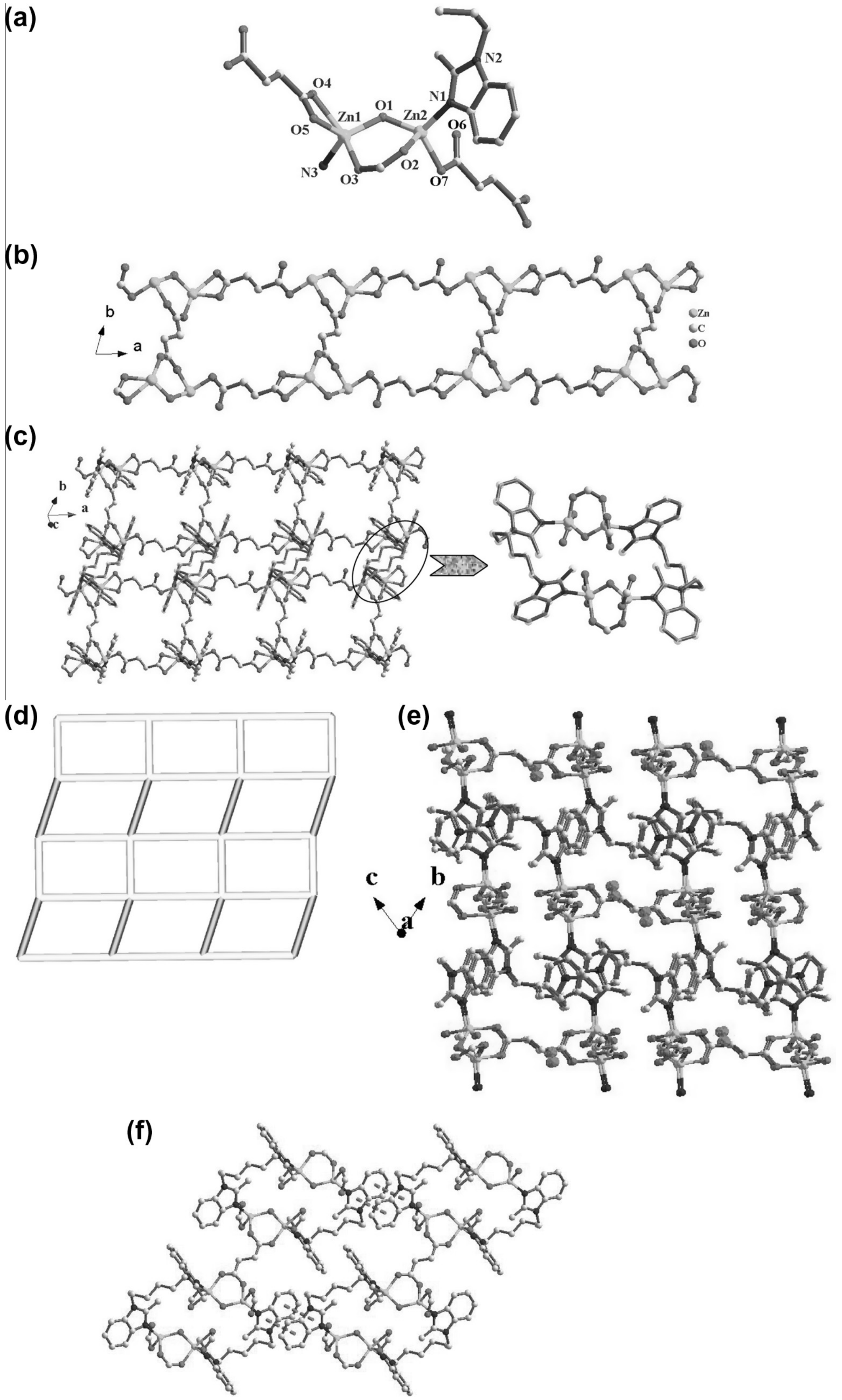

Fig. 4. (a) Coordination environments of Zn(II) in complex **4** (hydrogen atoms are omitted for clarity). (b) The suc^{2-} anions coordinate with Zn(II) ions to form a 1D ladder chain along the *c*-axis. (c) 2D layered structure of complex **4** (left) and a 30-membered rings constructed by two hbmb ligands and two [$Zn_2(CO_2)O$] units (right). (d) The 2D sheet consists of rectangular grids with $4^4 \cdot 6^2$ topology in **4**. (e) Schematic view of 3D structure with water molecules in **4**. (f) The illustration of π–π interactions between two neighboring layers (hydrogen atoms and the part of the structure are omitted for clarity).

599 °C, corresponding to the losses of hydroxyl group and hbmb as well as the decomposition of suc^{2-}, and the ZnO residue of 23.94% (Calc. 23.11%) is observed. Complex **5** shows a weight loss of 4.94% from 59 to 139 °C corresponding to the release of lattice water molecule (Calc. 5.14%), and then, a plateau region is observed. The further weight loss is observed at about 312 °C due to the decomposition of hydroxyl group and overall framework. A residue of ZnO (obsd, 24.54%, Calc. 23.21%) is obtained at 567 °C.

3.4. Luminescence properties

The coordination complexes with d^{10} metal centers have been investigated for fluorescence properties owning to their potential applications in photoactive materials [62–68]. Accordingly, the emission spectra of Zn(II)/Cd(II) complexes **1**–**5**, together with the free hbmb ligand and all of the different dicarboxylic acids were measured in the solid state at room temperature (Fig. S3 in the Supporting Information). Under the same experimental conditions, the emission intensities of H_2adi, H_2atda, H_2suc, and H_2fum (Fig. S3a) are much weaker than that of N-donor ligand hbmb, so it is considered that they have no significant contribution to the fluorescent emission of the complexes with the presence of hbmb. The free ligands hbmb and H_2tbi show intense emissions bands at 306 nm (λ_{ex} = 275 nm) and 319 nm (λ_{ex} = 273 nm), respectively. The solid state photoluminescence spectra of complexes **1**–**5** at ambient temperature are depicted in Fig. S3b. It is clear that there are emission bands at 445 nm (λ_{ex} = 365 nm) for **1**, 446 nm (λ_{ex} = 366 nm) for **2**, 301 nm (λ_{ex} = 276 nm) for **3**, 309 nm (λ_{ex} = 284 nm) for **4**, 442 nm (λ_{ex} = 368 nm) for **5**, respectively. For complex **1**, the emission band is highly red-shifted (139 and 126 nm) compared to the free hbmb and H_2tbi, respectively. This red shift may be tentatively assigned to ligand-to-metal charge transfer (LMCT) character [69,70]. **1** has interesting dual emissions, which may be ascribed to the different structural topology of **1** [71]. Obviously, the fluorescent emission bands of **3** and **4** can be attributed to the intraligand $\pi^* \rightarrow \pi$ charge transitions of hbmb because of their similar emission bands. Furthermore, the emission bands of **2** and **5** also present larger red shifts (140 and 136 nm) in contrast to that of the free hbmb ligand. Such broad emission band may be also caused by a ligand-to-metal charge-transfer (LMCT) transition [72,69,70].

From the above discussion, complexes **1**–**5**, which have the same N-donor ligand, show different photoluminescence behavior. The differences between the emission peaks for **1**–**5** are probably attributable to the differences in the dicarboxylate coligands and the coordination environments around the central metal ions [73]. The results indicate that the dicarboxylate coligands and the coordination environment around central metal ions have an important influence on the photoluminescence behavior.

4. Conclusion

Five new Zn(II)/Cd(II)-containing coordination complexes based on a flexible bis(methylbenzimidazole) ligand and different dicarboxylates have been synthesized and characterized by X-ray diffraction, elemental analyses, thermal analyses and photoluminescent measurements. These complexes show different 2D frameworks. The results of this work suggest that the metal centers and the dicarboxylate ligands can greatly affect the construction of various coordination complexes. Thermal studies show that they have good thermal stability. Moreover, compounds **1**–**5** display good luminescent properties at room temperature. Further study about the structures and properties of a series of coordination complexes constructed by the bis(methylbenzimidazole) ligands with more auxiliary polycarboxylate ligands and metal ions is currently in progress.

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Appendix A. Supplementary material

CCDC 881829, 881830, 881831, 881832, 881833 contains the supplementary crystallographic data for complex **1**, **2**, **3**, **4**, **5**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.ica.2012.07.033.

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