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Dimension Increase *via* Hierarchical Hydrogen Bonding from Simple Pincer-like Mononuclear complexes

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Abstract: A tetradentate symmetric ligand bearing both coordination and hydrogen bonding sites, N^1,N^3-bis(1-(1*H*-benzimidazol-2-yl)-ethylidene)propane-1,3-diamine (H$_2$bbepd) was utilized to synthesize a series of transition metal complexes, namely [Co(H$_2$bbepd)(H$_2$O)2]·2ClO$_4$ (**1**), [Cu(H$_2$bbepd)(OTs$^-$)]·OTs$^-$ (**2**),[Cu(bbepd)(CH$_3$OH)] (**3**), [Cd(H$_2$bbepd)(NO$_3$)$_2$]·CH$_3$OH (**4**), [Cd(H$_2$bbepd)(CH$_3$OH)Cl]·Cl (**5**), and [Cd(bbepd)(CH$_3$OH)$_2$] (**6**). These complexes show similar discrete pincer-like coordination units, possessing different arrangements of hydrogen bonding donor and acceptor sites. With or without the aid of uncoordinated anions and solvent molecules, such mononuclear units have been effectively involved in the construction of hierarchical hydrogen bonding assemblies (successively *via* level **I** and level **II**), leading to discrete binuclear ring (complex **2**), one-dimensional chain or ribbon (complexes **3**, **4** and **6**) and two-dimensional layer (complexes **1** and **5**) aggregates.

Keywords: Benzimidazolyl ring · Dimension increase · Hierarchical hydrogen bonding

Introduction

The self-assembly of metal–organic compounds to enable predictable and controllable synthesis of supramolecular solid materials has attracted much attention in recent decades due to their potential applications in catalysis, adsorption, separation, photoluminescence, magnetism.[1–3] In particular the frameworks based on the assembly of discrete coordination complexes as building blocks *via* different kinds of intermolecular weak interactions, such as hydrogen bonds, π···π interaction, anion-π interaction and so on, have attracted our interest.[4–6] This is due to the fact that: (i) the structures of discrete coordination complexes are easier to predict and controllably synthesize; (ii) the weak interaction sites of the organic ligands can be arranged and fixed in a desirable way *via* the direction of coordination bonds around metal ions; (iii) the building block is easy to tune by changing metal ions, labile small coordination molecules or anions. Based on this strategy, many supramolecular frameworks with different dimensions and topologies can be obtained from discrete mononuclear complexes with similar coordination structures but varied weak interaction sites, numbers and directions.[7–9]

Organic ligands based on 2-substituted benzimidazolyl rings have wide applications due to their potential biological activities, photo-/electrochemical and physical properties, and so on.[10,11] These ligands bear versatile coordination modes, and have the tendency to form various kinds of supramolecular aggregates by the formation of different weak interactions such as π···π stacking and hydrogen bonding.[12,13] Recently, we designed a tetradentate ligand containing double benzimidazolyl groups, N^1,N^3-*bis*(1-(1*H*-benzimidazol-2-yl)-ethylidene)propane-1,3-diamine (H$_2$bbepd).[14] This ligand tends to form discrete and stable mononuclear pincer-like complexes that contain tightly binding H$_2$bbepd ligands in the equatorial plane and labile coordination site(s) in the apical positions. These discrete mononuclear complexes are further used as precursors to assemble multinuclear complexes by the connection of weak interactions *via* a stepwise strategy. Considering that the H$_2$bbepd ligand possesses excellent coordination and hydrogen bonding sites (Fig. 1), herein, we use the discrete pincer-like complexes as building blocks in the assembly of hydrogen bonding aggregates.

Experimental

Materials and Method

N^1,N^3-bis(1-(1*H*-benzimidazol-2-yl)ethylidene)propane-1,3-diamine (H$_2$bbepd) was synthesized according to literature.[15] All the other chemicals were of reagent grade obtained from commercial sources and used without further purification. The C, H, N elemental analyses were performed on a Perkin-Elmer 240 elemental analyzer. IR spectra were recorded on a Nicolet/Nexus-670 FT-IR spectrometer with KBr pellets in the 4000–400 cm^{-1} region. X-ray powder diffraction data was recorded on a Bruker D8 Advance diffractometer at 40 kV, 40 mA with a Cu-target tube and a graphite monochromator.

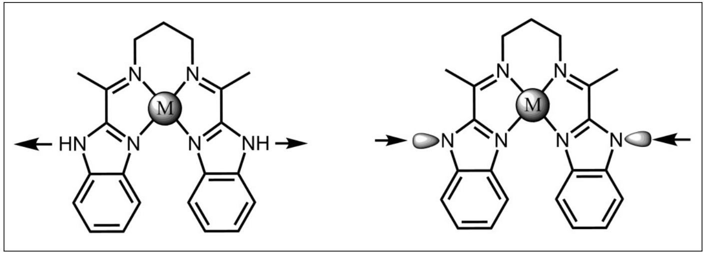

Fig. 1. The coordination mode and hydrogen bonding sites in H$_2$bbepd.

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Table 1. Crystallographic data and structure refinement summary for complexes **1–6**

Complex	**1**	**2**	**3**
Chemical formula	$C_{21}H_{26}Cl_2CoN_6O_{10}$	$C_{35}H_{36}CuN_6O_6S_2$	$C_{22}H_{24}CuO$
Formula weight	652.31	764.36	452.01
Space group	*P*-1	*P*-1	*P2(1)/n*
a [Å]	21.386(7)	9.6366(9	8.6416(3)
b [Å]	15.809(5)	13.0181(9)	17.1408(6)
c [Å]	8.142(3)	14.4328(10)	14.4710(5)
α [°]	90	87.793(6)	90
β [°]	97.455(6)	72.447(7)	105.253(4)
γ [°]	90	80.953(7)	90
V [Å^3]	2729.4(15)	1704.7(2)	2067.99(12)
Z	4	2	4
D [g cm^{-3}]	1.587	1.489	1.452
μ [mm^{-1}]	0.889	0.819	1.083
T [K]	293	293(2)	153
R_1/wR_2	0.1120/0.3273	0.0432/0.1090	0.0508/ 0.1488
Total/unique/R_{int}	7292/2666/0.0312	12997/6528/0.0344	12432/4459/0.0385
Complex	**4**	**5**	**6**
Chemical formula	C_{22} $H_{26}CdCl_2N_6O$	C_{22} $H_{26}CdN_8O_7$	$C_{23}H_{28}CdN_6O_2$
Formula weight	573.79	626.91	532.91
Space group	*P2(1)2(1)2(1)*	*P*-1	*C2/c*
a [Å]	8.3387(3)	8.750(2)	17.0813(9)
b [Å]	15.6267(6)	9.452(3)	11.1917(5)
c [Å]	17.9774(6)	15.2894(13)	14.0242(7)
α [°]	90	96.014(12)	90
β [°]	90	100.458(12)	120.569(5)
γ [°]	90	90.14(2)	90
V [Å^3]	2342.57(15)	1236.3(5)	3527.4(2)
Z	4	2	4
D [g cm^{-3}]	1.627	1.684	1.533
μ [mm^{-1}]	1.188	0.944	0.978
T [K]	153	153	293
R_1/wR_2	0.0390/ 0.1074	0.0328/ 0.0785	0.0314/ 0.0791
Total/unique/R_{int}	9713/4004/ 0.0303	11237/4791/0.0317	5241/2015/0.0289

Synthesis of $[Co(H_2bbepd)(H_2O)_2]·2ClO_4$ (1)

A solution of $Co(ClO_4)_2.6H_2O$ (36.6 mg, 0.1 mmol) and H_2bbepd (35.8 mg, 0.1 mmol) in MeOH (5 mL) was stirred for 10 min and filtered. The clear filtrate was left in a test tube for natural evaporation and red block crystals of **1** were obtained after several days. Yield: 70%. IR (KBr, cm^{-1}): 3374s, 2940, 1633m, 1438s, 1293s, 802w.

Synthesis of $[Cu(H_2bbepd)(OTs^-)]·OTs^-$ (2)

A solution of $Cu(OTs)_2·6H_2O$ (37.1 mg, 0.1 mmol, OTs^- = p-$CH_3C_6H_4SO_3$) and H_2bbepd (35.8 mg, 0.1 mmol) in MeOH (5 mL) was stirred for 30 min and filtered. The clear filtrate was left in a test tube, and slow diffusion of the Et_2O resulted in precipitation of blue crystals of **2**. Yield: 72%. IR (KBr, cm^{-1}): 3403m, 2923m, 1623s, 1432s, 744s.

Synthesis of $[Cu(bbepd)(CH_3OH)]$ (3)

Excess amount of NaOH (20 mg, 0.5 mmol) was added into a suspension of H_2bbepd (35.8 mg, 0.1 mmol) in MeOH (5 mL) and stirred for 10 min, and then $Cu(ClO_4)_2·6H_2O$ (29.1 mg, 0.1 mmol) was added, stirred for 10 min and filtered. The clear filtrate was left at room temperature for several days resulting in blue crystals of **3**. Yield: 65%. IR (KBr, cm^{-1}): 3402m, 2930m, 1613s, 1430s, 748s.

Synthesis of $[Cd(H_2bbepd)(NO_3)_2]·CH_3OH$ (4)

Complex **4** was obtained by a similar procedure as for **2** using $Cd(NO_3)_2·6H_2O$ (35.6 mg, 0.1 mmol) instead of $Cu(OTs)_2·6H_2O$. Yield: 65%. IR (KBr, cm^{-1}): 3491m, 3096m, 2957m, 1631m, 1449m, 1384s, 1346s, 752m.

Synthesis of $[Cd(H_2bbepd)(CH_3OH)Cl]·Cl$ (5)

Complex **5** was obtained by a similar procedure as for **2** using $CdCl_2·2H_2O$ (35.6 mg, 0.1 mmol) instead of $Cu(OTs)_2·6H_2O$. Yield: 75%. IR (KBr, cm^{-1}): 3487m, 3133s, 2924s, 1620s, 1445s, 750s.

Synthesis of $[Cd(bbepd)(CH_3OH)_2]$ (6)

Complex **6** was obtained by a similar procedure as for **3** using $Cd(ClO_4)_2·6H_2O$ (0.042 g, 0.1 mmol) instead of $Cu(ClO_4)_2·6H_2O$. Yield: 75%. IR (KBr, cm^{-1}): 3487m, 3133s, 2924s, 1620s, 1445s, 750s.

X-ray Crystallography

The diffraction data were collected on an Oxford Gemini S Ultra diffractometer equipped with Mo-Kα radiation (λ = 0.71073 Å) for complexes **1**–**6** at 150 K (complexes **3**–**5**) or 293 K (complexes **1**, **2** and **6**). Multi-scan adsorption corrections were applied for all complexes. The structures were solved by the direct methods (SHELXS) and refined by the full matrix least-squares method against F_o^2 using the SHELXTL software.[16] The coordinates of the non-hydrogen atoms were refined anisotropically. Most of hydrogen atoms were introduced in calculated positions and refined with fixed geometry with respect to their carrier atoms. The hydrogen atoms on disorder C atom in complexes **1** (C11) and **6** (C10) were not added but included in the formula. Experimental details of the X-ray structural analyses as well as the crystallographic data are provided in Table 1. Selected bond distances and angles are listed in Table 2. CCDC reference numbers 1402927-1402932. For crystallographic data in CIF see the Supplementary Data.

Results and Discussion

The Mononuclear Unit in Complexes 1–6

X-ray single crystal diffraction reveals that all the complexes are mononuclear complexes composed of one metal ion and one H_2bbepd or $bbepd^{2-}$ ligand, as shown in Fig. 2. The coordination sites on the H_2bbepd or $bbepd^{2-}$ ligand are arranged in a plane and occupy the equatorial plane of

Table 2. Selected details of the hydrogen bonds in complexes **1–6**

	Symmetry code	*d*(D–H) [Å]	*d*(H…A) [Å]	*d*(D…A) [Å]	∠D–H…A [°]
1					
N(2)-H(2B)···O(4)	-x+1/2,-y+3/2,-z-2	0.86	2.51	3.23(11)	141.3
O(1)-HWA)···O(2)	x,y,z-1	0.85	2.01	2.75(10)	145.4
O(1)-HWB)···O(5)	-x+1,y,-z-5/2	0.85	2.19	2.94(11)	146.5
2					
N(2)-H(2B)···O(5)	-x+1,-y,-z+1	0.86	1.94	2.796(4)	174.4
N(2)-H(2B)···O(4)	-x+1,-y,-z+1	0.86	2.66	3.188(4)	121.2
N(6)-H(6A)···O(2)	-x+1,-y,-z	0.86	1.92	2.780(4)	175.1
3					
O(1)-H(1)···N(6)#1	-x+1,-y,-z+1	0.91	1.76	2.674(4)	175.8
4					
N(2)-H(2B)···O(1)	-x+1,-y+1,-z+3	0.86	2.00	2.860(4)	177.8
N(6)-H(6A)···O(7)		0.86	1.90	2.749(4)	167.9
O(7)-H(7)···O(4)#2	x,y-1,z	0.82	2.00	2.821(4)	173.6
5					
N(2)-H(2A)···Cl(1)	-x+1,y+1/2,-z+1/2	0.86	2.29	3.121(5)	162.4
N(6)-H(6A)···Cl(2)	-x,y-1/2,-z+1/2	0.86	1.95	2.749(7)	153.3
O(1)-H(1W)···Cl(2)		0.83(2)	1.84(3)	2.649(6)	164(8)
6					
O(1)-H(2)···N(2)	-x+5/2,-y+1/2,-z+2	0.89(5)	1.87(5)	2.728(4)	163(4)

the metal ion, thus leaving labile coordination sites on the apical sites of the metal center. When using octahedral Co(II) ion or Cd(II) ions, the mononuclear unit contains two labile coordination sites that are occupied by anions or solvent molecules, whereas only one labile coordination site is left when Cu(II) ion possessing tetragonal pyramid coordination geometry is applied. The coordinated group(s) on the apical sites can be varied by using different anions. When ClO_4^- with weaker coordination ability was used in complex **1**, the ClO_4^- remained uncoordinated and the apical sites of Co(II) are occupied by water molecules (Fig. 2a). When anions with stronger coordination ability, such as OTs^- and NO_3^- were used, the apical sites of the metal ions are occupied by the anions, as observed in compounds **2** and **4** (Figs 2b and 2d). It is interesting to note that when Cl^- anion is used, the two apical sites of Cd(II) in compound **5** are coordinated by Cl^- and MeOH, respectively (Fig. 2e). When NaOH is introduced to the reaction system, the H_2bbepd is deprotonated to bbepd^{2-} anion, which can bind to one divalent metal ion to form a neutral unit, therefore the inorganic anions are not needed and the apical sites of the metal ions are only filled by solvent molecules (MeOH), as observed in complexes **3** and **6** (Figs 2c and 2f). The H_2bbepd or bbepd^{2-} ligand in the mononuclear complexes provides two hydrogen bonding sites by the benzimidazolyl rings, serving as hydrogen acceptors in complexes **3** and **6** (due to the deprotonation), but act as hydrogen donors in all other complexes. The coordinated solvent molecule(s) or anion(s) provide surplus hydrogen bonding sites. In detail, the coordinated water molecules (complex **1**) and MeOH (complexes **3**, **5** and **6**) can serve as hydrogen bonding donors, whereas the coordinated OTs^-, NO_3^- and Cl^- should be hydrogen-bonding acceptors. Therefore, the mononuclear coordination unit in complexes **1–6** can be seen as a building block containing four or three hydrogen bonding sites, respectively, as shown in Fig. 3.

Dimension Increase via Hydrogen Bonding in Complexes 1–6

As stated above, because the mononuclear units in complexes **1–6** possess strong hydrogen bonding sites, they tend to be involved in hydrogen bonding aggregation and result in dimension increase. In complex **1**, the mononuclear Co(II)-coor-

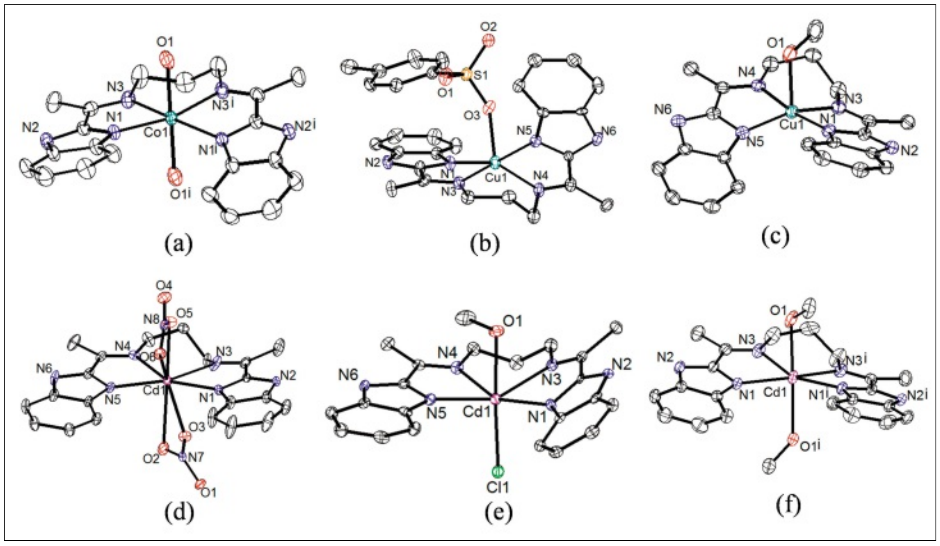


Fig. 2. The ORTEP drawings for mononuclear coordination unit in complexes **1–6** (a-f, respectively). Symmetry code for complex **1**: (i) 1 – x, y, –2.5 – z; Symmetry code for complex **6**: 2 – x, y, 1 – z.

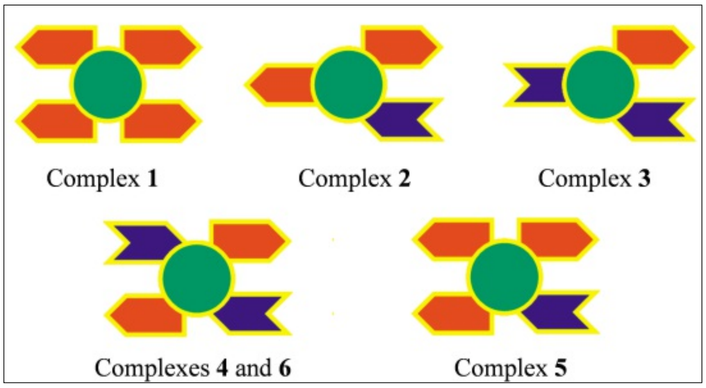


Fig. 3. Hydrogen bonding sites on the coordination units of complexes **1**–**6**. **1** has four H donors, **2** has two donors and one acceptor, **3** has one donor and two acceptors, **4** and **6** have two donors and two acceptors, and **5** has three donors and one acceptor.

dination unit contains only four hydrogen bonding donors (two NH groups on benzimidazolyl rings and two H_2O), thus they cannot be interconnected together solely by themselves. Instead, by the connection of uncoordinated ClO_4^- anions as hydrogen donors, the discrete coordination units in complex **1** are linked into infinite hydrogen bonding networks. As shown in Fig. 4 and Table 2, the O-H···O hydrogen bonds between coordinated water molecules and ClO_4^- anions, and the N-H···O hydrogen bonds between benzimidazolyl group and ClO_4^- anions connect the mononuclear units and ClO_4^- anions into a two-dimensional (2D) hydrogen-bonding network. Although the Co(II)-coordination unit in complex **1** has four hydrogen bonding sites, due to the linking of multiple bifurcated H-bonds (Fig. 4a), each mononuclear unit connects to six adjacent mononuclear units *via* bridging ClO_4^- anions, and can be therefore considered as a six-connecting node, and a 2D network with (3,6) network topological view is formed (Fig. 4b).

In complex **2**, the mononuclear Cu(II)-coordination unit contains two hydrogen bonding donors (two NH groups on benzimidazolyl rings) and one hydrogen bonding acceptor (O on OTs^- anion), which indicates that the mononuclear units may interconnect by themselves *via* the donors and acceptors. Indeed, two mononuclear units are joined by N-H···O hydrogen bonds between benzimidazolyl groups and OTs^- anions, giving rise to a binuclear ring. Uncoordinated OTs^- anions terminate the remaining H-donor sites on the binuclear rings *via* formation of additional N-H···O bonds. Therefore, the hydrogen bonding aggregate in complex **2** remains a 0D discrete structure, as shown in Fig. 5a. In comparison, the mononuclear Cu(II)-coordination unit in complex **3** contains two hydrogen bonding acceptors (deprotonated N on benzimidazolyl rings) and one hydrogen bonding donor (MeOH), which is in contrast to that in complex **2**. However, similar to the situation in complex **2**, two mononuclear units in complex **3** are also joined together by themselves, *via* O-H···N hydrogen bonds (red dash in Fig. 5b). The uncoordinated N atoms from deprotonated benzimidazolyl groups on the binuclear ring form additional C-H···N hydrogen bonds (purple dash in Fig. 5b) with the CH group on benzene from adjacent binuclear rings, linking binuclear units together and leading to a one-dimensional (1D) chain.

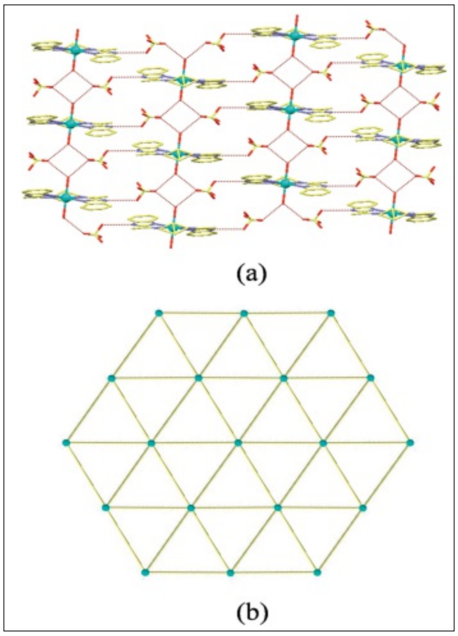


Fig. 4. (a) The 2D hydrogen bonding layer structure in complex **1**. (b) The schematic (3, 6) hydrogen bonding topology in complex **1**.

In complexes **4**–**6**, the mononuclear Cd(II)-coordination units all contain four hydrogen-bonding sites. In complexes **4** and **6**, there are two hydrogen bonding donors plus two acceptors, whereas three donors and one acceptor can be donated by each coordination unit in complex **5**. Similar to the situation in complexes **2** and **3**, the mononuclear unit in complex **4** also possesses hydrogen bond donor and acceptor sites on adjacent positions, therefore, a binuclear ring can be built by interconnection of two mononuclear units *via* N-H···O hydrogen bonds between the hydrogen bonding donors (coordinated NO_3^-) and hydrogen bonding acceptors (NH on the benzimidazolyl group) in complex **4**. These binuclear rings are further connected by uncoordinated MeOH molecules *via* O-H···O hydrogen bonds between the MeOH and NO_3^- and N-H···O hydrogen bonds between benzimidazolyl groups and MeOH, producing a 1D ribbon, as shown in Fig. 6a. In complex **5**, the angle between the adjacent hydrogen bond donor and acceptor is larger than that in complex **4**, thus the interconnection of donor and acceptor among mononuclear units leads to a 1D chain rather than a ring. The 1D chains are further connected by a O-H···Cl hydrogen bond between MeOH and uncoordinated Cl^-, and a N-H···Cl hydrogen bond between benzimidazolyl group and uncoordinated Cl^-, thereby forming a 2D network with (4,4) topology (Fig. 6b). Because there are no uncoordinated anion or solvent molecules in complex **6**, the mononuclear units in **6** can be interconnected only by themselves *via* O-H···N hydrogen bonds between deprotonated benzimidazolyl groups and coordinated MeOH molecules, yielding a 1D chain (Fig. 6c).

From the above discussions, we found that the final structures of the hydrogen bonding assemblies in complexes **1**–**6** are dependent on the attributes and arrangements of basic mononuclear units, coordinated and uncoordinated anions and solvent molecules. As shown in Scheme 1, the combination of six/five-coordinated metal ions and $H_2bbepd/bbepd^{2-}$ can result in seven types of mononuclear unit, and six of them are observed in complexes **1**–**6**. If we only consider the number and position of hydrogen bonding acceptors and donors, they can be classified into five

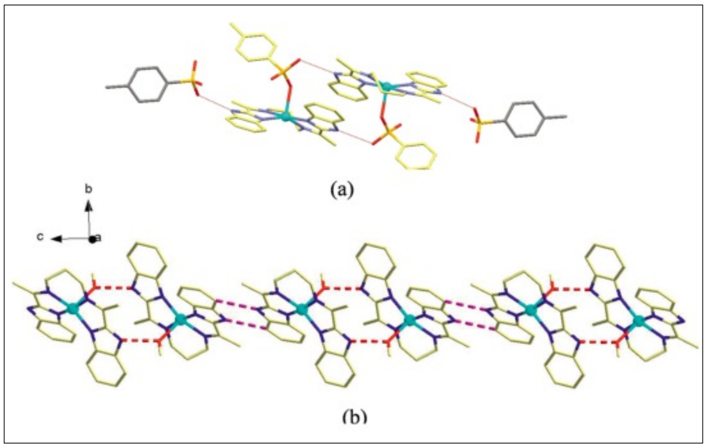


Fig. 5. (a) The binuclear ring constructed by mononuclear units and hydrogen bonds in complex **2**. (b) The 1D hydrogen bonding chain in complex **3**.

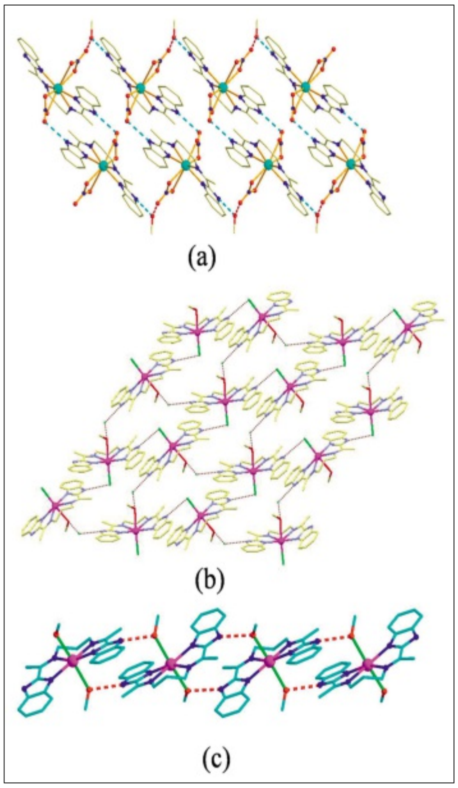
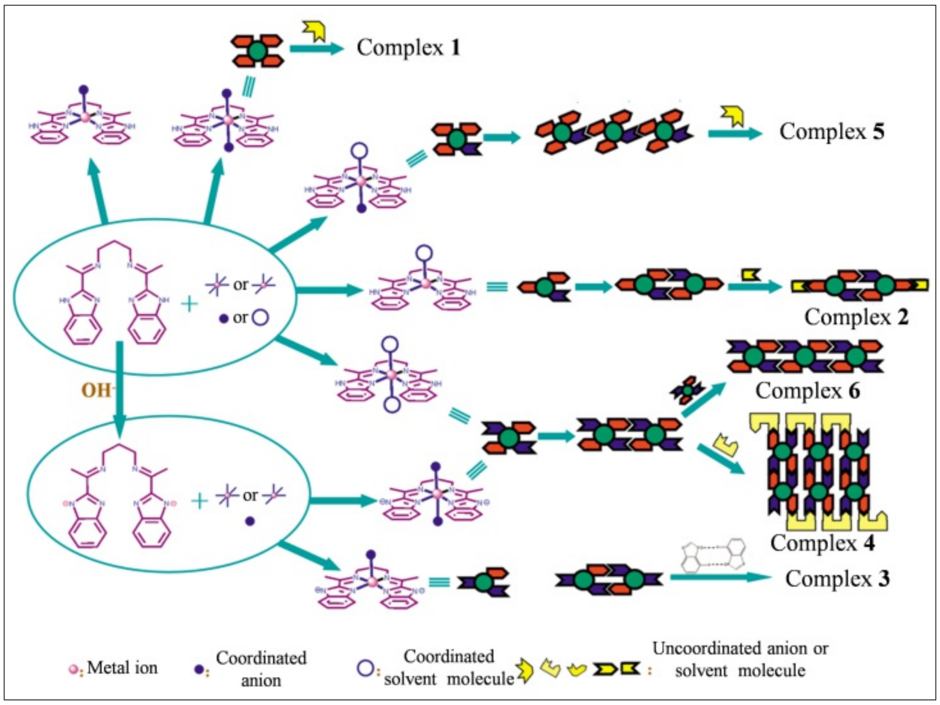


Fig. 6. (a) The 1D hydrogen bonding ribbon in complex **4**. (b) The 2D hydrogen bonding network and representative (4, 4)-topology in complex **5**. (c) The 1D hydrogen bonding chain in complex **6**.

types (as represented in Fig. 3). Obviously, if the mononuclear coordination unit only contains hydrogen bonding acceptors (or donors), they should connect into higher dimensional hydrogen bonding networks by the aid of uncoordinated anions or solvent molecules, as in complex **1**. All the other four types of mononuclear units contain both hydrogen bonding donors and acceptors on adjacent positions, so they can connect themselves *via* one donor plus one acceptor into 1D chain (complex **5**) or binuclear ring (complexes **2**, **3**, **4** and **6**), which is dependent on the angle between the donor and acceptor sites. The resulting 1D chain or binuclear ring can be seen as the **level I** hydrogen bonding assembly in these complexes. Furthermore, **level II** hydrogen bonding happens, leading the structures into higher dimensions. In complex **5**, the 1D chain formed by **level I** hydrogen bonding only leaves vacant H-donor sites, therefore acceptors from uncoordinated species (Cl^-) should take part in the formation of a higher dimensional hydrogen-bonding network. Such is also the case with complex **2**, although a binuclear ring structure is formed in **level I** assembly. In complex **3**, hydrogen bonding of **level I** leads to binuclear rings, which leaves vacant H-acceptor sites, but they can be further connected by the formation of another kind of hydrogen bonding (C-H···O), which is not accounted for in **level I** assembly. In comparison, in complex **6**, the binuclear rings resulting from **level I** assembly contain both vacant hydrogen donors and acceptors, which go on to connect themselves into 1D chains in **level II** hydrogen bonding assembly. And in complex **4**, although the **level I** hydrogen-bonding connected binuclear ring is similar to that in complex **6**, a different 1D ribbon structure is finally achieved because the uncoordinated MeOH, which acts as both H-donor and acceptor, is involved in the **level II** hydrogen bonding assembly.



Scheme 1. Hierarchical assembly of the hydrogen bonding aggregates from discrete coordination units in complexes **1–6**.

Conclusions

In summary, a double Schiff base ligand containing benzimidazolyl groups with tetradentate chelate coordination sites has been applied to the syntheses of mononuclear transition metal complexes. The coordination of Co(II), Cu(II) and Cd(II) with the ligand affords five types of mononuclear complexes in the view of their hydrogen bonding sites. In **level I** hydrogen bonding assembly, the discrete coordination units can be linked into a 0D binuclear ring or 1D chain structure; and in **level II** assembly, the remaining H-bonding sites can be further applied, with or without the participation of counter anions and solvent molecules, leading to various kinds of dimension-increased hydrogen bonding aggregates. The structure of mononuclear units, competition among anions, solvent molecules and organic ligands in hydrogen bonds formation define the final supramolecular structures.

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