

Syntheses, crystal structures and blue emission of three zinc(II) coordination polymers with a 4,4'-dicarboxybiphenyl sulfone ligand

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Three novel zinc complexes $[\text{Zn}(\text{dbsf})(\text{H}_2\text{O})_2]$ (1), $[\text{Zn}(\text{dbsf})(2,2'\text{-bpy})(\text{H}_2\text{O})]\cdot(i\text{-C}_3\text{H}_7\text{OH})$ (2) and $[\text{Zn}(\text{dbsf})(\text{DMF})]$ (3) (H_2dbsf = 4,4'-dicarboxybiphenyl sulfone, 2,2'-bpy = 2,2'-bipyridine, $i\text{-C}_3\text{H}_7\text{OH}$ = *iso*-propanol, DMF = *N,N*-dimethylformamide) were first obtained and characterized by single crystal X-ray crystallography. Although the results show that all the complexes 1–3 have one-dimensional chains formed via coordination bonds, unique three-dimensional supramolecular structures are formed due to different coordination modes and configuration of the dbsf^{2-} ligand, hydrogen bonds and π – π interactions. *iso*-propanol molecules are in open channels of 2 while larger empty channels are formed in 3. As compared with emission band of the free H_2dbsf ligand, emission peaks of the complexes 1–3 are red-shifted, and they show blue emission, which originates from enlarging conjugation upon coordination. Copyright © 2006 John Wiley & Sons, Ltd.

KEYWORDS: zinc complex; 4,4'-dicarboxybiphenyl sulfone; crystal structure; blue emission

INTRODUCTION

In recent years, metal–organic frameworks (MOFs) have attracted much interest from chemists in the field of crystal engineering and chemistry materials, by their intriguing structures and molecular topologies,^{1–3} and also their potential applications as functional materials in luminescence, catalysis, gas storage, ion exchange and molecular recognition.^{4–7} Among the most extensively studied MOFs, transition metals are widely used due to their lower coordination numbers and stable coordination geometries.^{1,2,8,9} These characteristics make them popular in the forecast, design and synthesis of MOFs. Moreover, d^{10} metal complexes are fascinating because they exhibit intriguing photoluminescent properties.^{10–12} Generally speaking, zinc(II) is usually selected in combination with organic ligands to construct coordination polymers with blue emission properties.^{13–15}

On the other hand, researches on transition metal MOFs with rigid and linear dicarboxylic ligands have been widely carried out owing to their diverse structures along

with various coordination modes.^{2,8,9,16,17} However, studies on semi-rigid V-shaped dicarboxylate ligands are relatively scarce.^{18–21} 4,4'-Dicarboxybiphenyl sulfone (H_2dbsf) is a semi-rigid V-shaped dicarboxylate ligand. To the best of our knowledge, no example of coordination compound constructed by H_2dbsf has been reported. Herein, we report the syntheses, structures and emission properties of three novel zinc(II) complexes with H_2dbsf ligand: $[\text{Zn}(\text{dbsf})(\text{H}_2\text{O})_2]$ (1), $[\text{Zn}(\text{dbsf})(2,2'\text{-bpy})(\text{H}_2\text{O})]\cdot(i\text{-C}_3\text{H}_7\text{OH})$ (2) and $[\text{Zn}(\text{dbsf})(\text{DMF})]$ (3).

EXPERIMENTAL

Materials and physical measurements

All reagents were used as received without further purification. The C, H, N microanalyses were carried out with a Vario EL elemental analyzer. The IR spectra were recorded with a Nicolet Avatar 360 FT-IR spectrometer using the KBr pellet technique. Fluorescence spectroscopy data of complexes 1–3 were recorded on a Shimadzu RT-5301PC spectrofluorophotometer.

Synthesis of $[\text{Zn}(\text{dbsf})(\text{H}_2\text{O})_2]$ (1)

A mixture of $\text{Zn}(\text{OAc})_2\cdot 2\text{H}_2\text{O}$ (0.030 g, 0.1 mmol), H_2dbsf (0.015 g, 0.05 mmol), NaOH aqueous solution (0.05 ml, 0.65

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m), HCl aqueous solution (0.05 ml, 1.20 M), distilled water (5 ml), *iso*-propanol (5 ml) and DMF (0.1 ml) was sealed in a Teflon-lined stainless vessel (25 ml) and heated at 180 °C for 72 h under autogenous pressure. The vessel was then cooled slowly to room temperature. Colorless flake crystals were obtained by filtration, washed with distilled water, and dried in air. Yield: 59.2%. Elemental analysis for C₁₄H₁₂O₈SZn (405.67): calcd, C, 41.45; H, 2.98%. Found: C, 41.52; H, 3.15%. IR (KBr, cm⁻¹): 3257s, 1646w, 1594s, 1555s, 1488w, 1402s, 1383s, 1328m, 1300m, 1162s, 1138w, 1102m, 1012w, 868m, 783w, 751s, 737m, 702w, 622m, 578m, 480w.

Complex **1** could also be obtained by the diffusion method. A 10 ml aliquot of mixed solvent of distilled water and methanol (1 : 5) was carefully layered on top of a 1 ml DMF solution of H₂dbsf (0.030 g, 0.1 mmol), to which a solution of Zn(OAc)₂·2H₂O (0.022 g, 0.1 mmol) in methanol (2 ml) was added. Colorless flake crystals were obtained after two months at room temperature.

Synthesis of [Zn(dbsf)(2,2'-bpy)(H₂O)]·(*i*-C₃H₇OH) (**2**)

A mixture of Zn(OAc)₂·2H₂O (0.022 g, 0.1 mmol), H₂dbsf (0.030 g, 0.1 mmol), 2,2'-bpy (0.016 g, 0.1 mmol), NaOH aqueous solution (0.15 ml, 0.65 M), distilled water (5 ml), *iso*-propanol (5 ml) and DMF (0.1 ml) was sealed in a Teflon-lined stainless vessel (25 ml) and heated at 180 °C for 72 h under autogenous pressure. The vessel was then cooled slowly to room temperature. Colorless block crystals were obtained by filtration, washed with distilled water and dried in air. Yield: 41.4%. Elemental analysis for C₂₇H₂₆N₂O₈SZn (603.93): calcd, C, 52.92; H, 4.44; N, 4.57%; found: C, 53.18; H, 4.63; N, 4.36%. IR (KBr, cm⁻¹): 3450s, 1597s, 1554s, 1445m, 1401s, 1296m, 1162m, 1132w, 1100m, 743s, 622m.

Synthesis of [Zn(dbsf)(DMF)] (**3**)

A mixed solvent of 3 ml methanol and 3 ml DMF was carefully layered on top of a 2 ml DMF solution of H₂dbsf (0.030 g, 0.1 mmol), to which a solution of Zn(OAc)₂·2H₂O (0.022 g, 0.1 mmol) in methanol (2 ml) was added. Colorless block crystals were obtained after 2 months at room temperature. Yield: 29.4%. Elemental analysis for C₁₇H₁₅NO₇SZn (442.73): calcd, C, 46.58; H, 4.30; N, 5.43%; found: C, 46.29; H, 4.52; N, 5.53%. IR (KBr, cm⁻¹): 3431s, 1646s, 1568m, 1406s, 1297m, 1162m, 1135w, 1101m, 1014w, 781w, 741s, 696w, 622m, 577w, 503w.

X-ray crystallography

Diffraction data for complexes **1–3** were collected at 293 K on a Bruker SMART 1000 CCD with graphite-monochromatized Mo-Kα radiation (λ = 0.71073 Å), using the ω and φ scan technique. A semi-empirical absorption correction was applied with SADABS²² program. The structures were solved by direct-methods (SHELXS 97²³) and refined by full-matrix least-squares on F² (SHELXL 97).²⁴ All non-hydrogen atoms were refined with anisotropic displacement ellipsoids and hydrogen atoms were placed in their geometrically calculated positions. Crystallographic data for **1–3** are summarized in Table 1, selected bond lengths and angles are listed in Table 2, and selected hydrogen bonding parameters are presented in Table 3.

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC nos 614 933–614 935 for complexes **1–3**, respectively. Copies of this data can be obtained free of charge from the CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK [fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk].

Table 1. Crystal data and structure refinement parameters of complexes **1–3**

	1	2	3
Empirical formula	C ₁₄ H ₁₂ O ₈ SZn	C ₂₇ H ₂₆ N ₂ O ₈ SZn	C ₁₇ H ₁₅ NO ₇ SZn
Formula weight	405.67	603.93	442.73
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	<i>P</i> 2 ₁ / <i>c</i>	<i>P</i> 2 ₁ / <i>n</i>	<i>C</i> 2/ <i>m</i>
<i>a</i> (Å)	13.317(3)	6.7630(9)	22.151(7)
<i>b</i> (Å)	5.0376(13)	16.478(2)	12.872(4)
<i>c</i> (Å)	12.120(3)	24.194(3)	9.993(3)
β (°)	116.295(4)	90.674(2)	99.584(5)
<i>V</i> (Å ³)	728.9(3)	2696.0(6)	2809.5(15)
<i>Z</i>	2	4	4
<i>F</i> (000)	412	1248	904
<i>D</i> _c (mg m ⁻³)	1.848	1.488	1.047
θ range (deg)	3.4 to 26.4	1.5 to 26.4	1.8 to 25.0
μ (mm ⁻¹)	1.871	1.041	0.974
GOF	1.05	1.00	1.26
Final <i>R</i> indices	<i>R</i> ₁ = 0.036	<i>R</i> ₁ = 0.045	<i>R</i> ₁ = 0.059
[<i>I</i> > 2σ(<i>I</i>)]	<i>wR</i> ₂ = 0.079	<i>wR</i> ₂ = 0.088	<i>wR</i> ₂ = 0.221

Table 2. Selected bond lengths (Å) and bond angles (deg) for complexes **1–3**

1	2	3
Zn1–O1 1.991(2)	Zn1–O1 2.035(2)	Zn1–O1 2.043(3)
Zn1–O4 1.981(2)	Zn1–O7 2.030(2)	Zn1–O7 1.986(5)
	Zn1–O5 ^a 1.977(2)	Zn1–O2 ^b 2.063(3)
	Zn1–N1 2.145(3)	
	Zn1–N2 2.112(3)	
O1–Zn1–O4	O1–Zn1–O7	O1–Zn1–O7
100.36(10)	93.43(10)	103.43(15)
O1–Zn1–O1 ^a	O1–Zn1–O5 ^a	O1–Zn1–O1 ^a
99.75(13)	94.36(11)	89.4(2)
O1–Zn1–O4 ^a	O1–Zn1–N1	O1–Zn1–O2 ^b
136.01(10)	158.90(11)	88.21(16)
O4 ^a –Zn1–O4	O1–Zn1–N2	O1–Zn1–O2 ^c
91.51(15)	89.68(11)	159.74(17)
	O7–Zn1–N1	O7–Zn1–O2 ^b
	105.18(10)	96.69(15)
	O7–Zn1–N2	O1 ^a –Zn1–O2 ^b
	103.43(11)	159.74(17)
	O5 ^a –Zn1–O7	O2 ^b –Zn1–O2 ^c
	103.02(11)	87.1(2)
	O5 ^a –Zn1–N1	
	91.09(11)	
	O5 ^a –Zn1–N2	
	152.94(12)	
	N1–Zn1–N2	
	76.57(11)	

Symmetry transformations used to generate equivalent atoms: for complex **1**, ^a $-x+1, y, -z+3/2$; for complex **2**, ^a $-x-1/2, y-1/2, -z+1/2$; for complex **3**, ^a $x, -y+1, z$; ^b $-x, y, -z+1$; ^c $-x, -y+1, -z+1$.

Table 3. Selected hydrogen bond lengths (Å) and bond angles (deg) for **1–3**

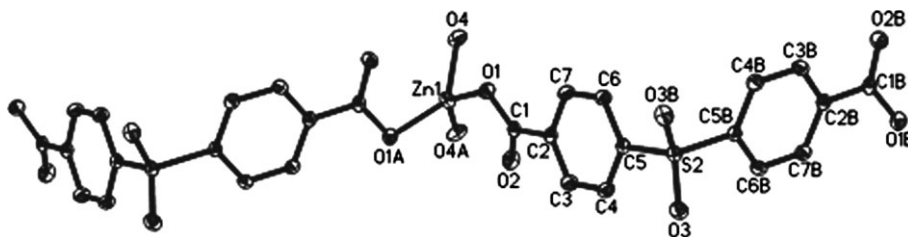
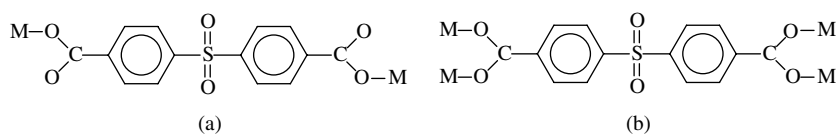
D–H···A	d(D–H)	d(H···A)	d(D···A)	∠DHA
Complex 1				
O4–H4A···O2 ^a	0.84	1.88	2.71(0)	169
O4–H4B···O1 ^b	0.85	2.01	2.79(1)	156
Complex 2				
O7–H7A···O6 ^a	0.85	1.88	2.71(0)	164
O8–H8···O2	0.82	2.19	2.92(0)	149
C13–H13···O4 ^b	0.93	2.56	3.19(1)	126
C21–H21···O6 ^c	0.93	2.36	3.26(1)	164
Complex 3				
C15–H15···O3 ^a	0.93	2.48	3.37(0)	160

Symmetry transformations used to generate equivalent atoms: for complex **1**, ^a $x, -y-1, z+1/2$; ^b $x, y-1, z$; for complex **2**, ^a $-x+1/2, y-1/2, -z+1/2$; ^b $-x, -y+2, -z+1$; ^c $x+1/2, -y+5/2, z-1/2$; for complex **3**, ^a $x-1/2, y+1/2, z$.

RESULTS AND DISCUSSION

Crystal structure of [Zn(dbsf)(H₂O)₂] (**1**)

As illustrated in Fig. 1, the Zn(II) ion is coordinated by two oxygen atoms (O1, O1A) from two different dbsf²⁻ ligands and two oxygen atoms (O4, O4A) from two water molecules. The four oxygen atoms coordinated to Zn(II) ion define a distorted tetrahedron. In complex **1**, the dbsf²⁻ ligands adopt bis(monodentate) mode (Scheme 1a), linking the Zn(II) ions to form zig-zag chains. Two types of O–H···O hydrogen bonds exist between the adjacent chains. One hydrogen-bond occurs between the oxygen atoms of the coordinated water molecules and the coordinated carboxyl oxygen atoms of the adjacent chain of the same type denoted as A, forming a wave-like array [see Fig. 2(a)]. The other occurs between the coordinated

**Figure 1.** ORTEP diagram of complex **1** showing the coordination environment of Zn(II) ion with 30% thermal ellipsoids, all hydrogen atoms are omitted for clarity. Symmetry operation: (A) $-x+1, y, -z+3/2$; (B) $-x, y, -z+1/2$.**Scheme 1.** The coordination modes of dbsf²⁻ ligand in complexes **1–3**.

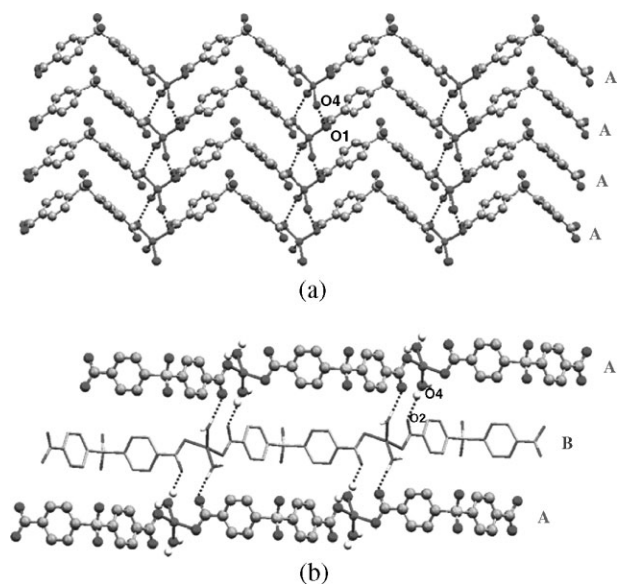


Figure 2. (a) The hydrogen bonds between the chains of same arrangement, denoted as A; (b) the hydrogen bonds between displacement chains A and B along the *b* axis (all hydrogen atoms except that involved in hydrogen bonds are omitted for clarity; hydrogen bonds are represented by dashed lines).

water oxygen atoms and the uncoordinated oxygen atoms of the dbsf^{2-} ligand, linking chains A and B, as shown in Fig. 2(b). In addition, $\pi-\pi$ stacking interactions exist between pyridyl planes of adjacent chains (the face-to-face distance is 3.83 Å). Thus, a three-dimensional supramolecular network is constructed by the interpolation of one-dimensional chains in the ...ABA... model via hydrogen bonds and $\pi-\pi$ interactions.

Crystal structure of $[\text{Zn}(\text{dbsf})(2,2'\text{-bpy})(\text{H}_2\text{O})] \cdot (i\text{-C}_3\text{H}_7\text{OH})$ (**2**)

The coordination environment of Zn(II) ion in **2** is shown in Fig. 3(a). Different from complex **1**, the Zn(II) ion

is five-coordinated. The structural index τ , defined as $[\beta - \alpha]/60$ with α and β being the two largest angles, is zero for an ideal square pyramid, and becomes unity for an ideal trigonal bipyramid.²⁵ In complex **2**, the τ value is 0.1 for Zn(II), indicating that it displays a distorted square-pyramid geometry. The geometry is completed by two nitrogen atoms from one chelating 2,2'-bpy ligand, Table 2, two oxygen atoms from two dbsf^{2-} ligands, and another oxygen atom from one coordinated water molecule. The dbsf^{2-} ligands adopt bis(monodentate) mode (Scheme 1a), the same as that in complex **1**, but they connect Zn(II) ions into completely different one-dimensional chains. Because of the introduction of 2,2'-bpy into the coordination sphere, steric congestion forces the dbsf^{2-} ligands to twist so that a one-dimensional square-like chains are constructed, as shown in Fig. 3(b). Between adjacent chains, there are two types of hydrogen bonds. (a) O-H...O hydrogen bonds are observed between the uncoordinated oxygen atoms of dbsf^{2-} and coordinated water molecules of the adjacent chains, and (b) C-H...O hydrogen bonds are found between the C-H of 2,2'-bpy molecules and the uncoordinated carboxylate oxygen atoms of the adjacent chains (Table 3). There are also $\pi-\pi$ stacking interactions between pyridyl planes of 2,2'-bpy ligands (the face-to-face distance is 3.35 Å). Hydrogen bonds and $\pi-\pi$ stacking interactions lead to the formation of a three-dimensional porous supramolecular structure, with open channels along the *a* axis [see Fig. 3(b)]. *Iso*-propanol molecules are located in the channels. They form hydrogen bonds with the uncoordinated oxygen atoms of the dbsf^{2-} ligands (Table 3).

Crystal structure of $[\text{Zn}(\text{dbsf})(\text{DMF})]$ (**3**)

In the asymmetric unit of **3**, there is one Zn(II) ion, one dbsf^{2-} ligand and one coordinated *N,N*-dimethylformamide molecule. Each Zn(II) ion is coordinated to five oxygen atoms, four oxygen atoms from four dbsf^{2-} ligands and one oxygen atom from one DMF molecule, as shown in Fig. 4(a). O1,

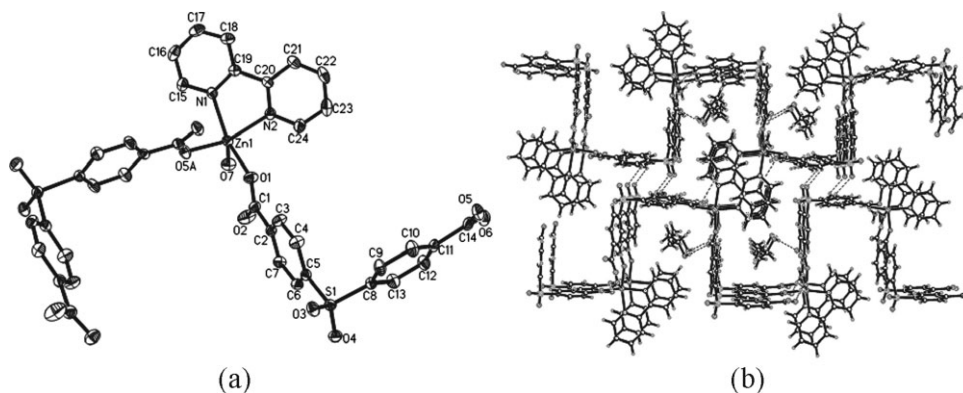


Figure 3. (a) ORTEP diagram of complex **2** showing the coordination environment of Zn(II) ion with 30% thermal ellipsoids, all hydrogen atoms are omitted for clarity; (b) view of the three-dimensional supramolecular structure of **2** along the *a* axis (hydrogen bonds are represented by dashed lines; *iso*-propanol molecules are in the channels). Symmetry operation: (A) $-x - 1/2, y - 1/2, -z + 1/2$.

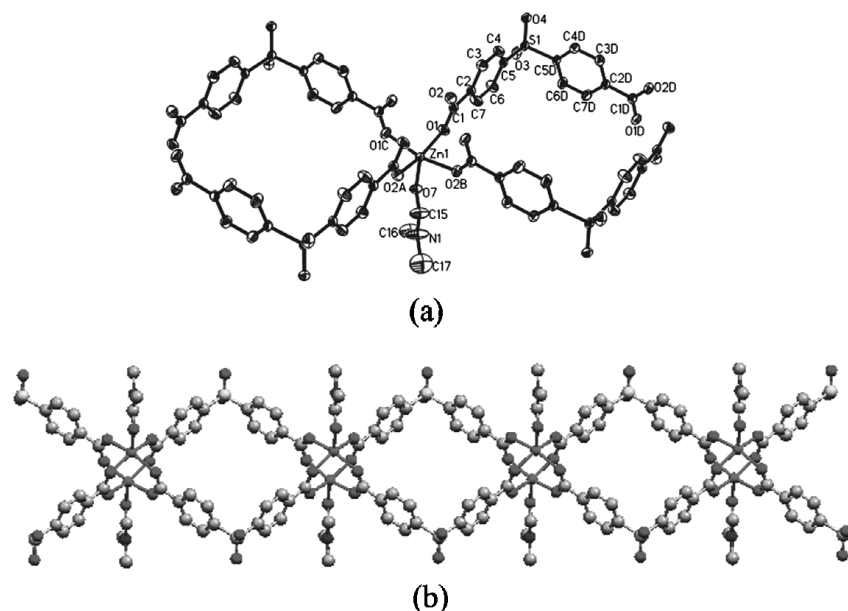


Figure 4. (a) ORTEP diagram of complex **3** showing the coordination environment of Zn(II) ion with 30% thermal ellipsoids, all hydrogen atoms are omitted for clarity; (b) the one-dimensional double-chain structure of **3**. Symmetry operations: (A) $x, -y + 1, z$; (B) $-x, y, -z + 1$; (C) $-x, -y + 1, -z + 1$; (D) $x, -y, z$.

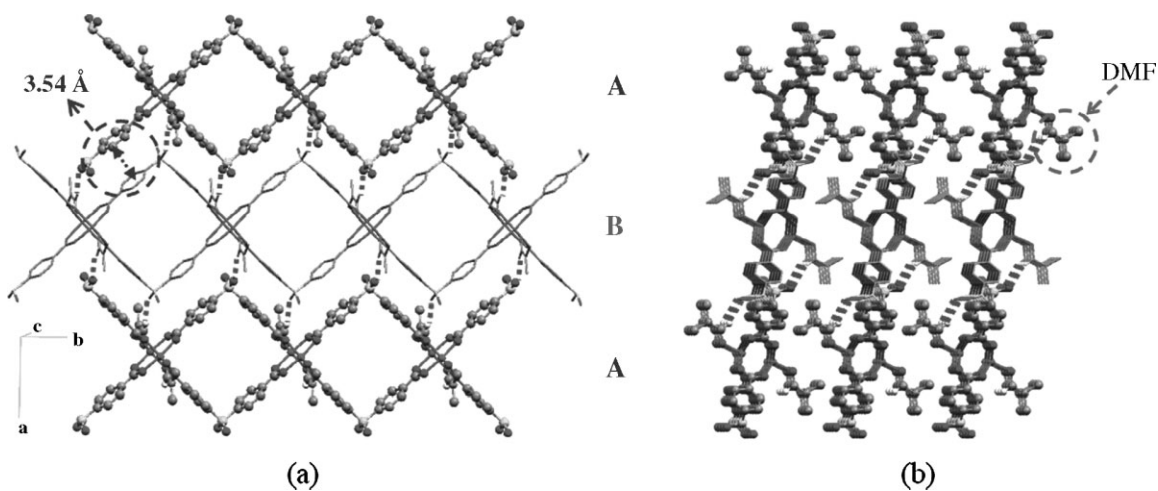


Figure 5. (a) The two-dimensional layer architecture of **3** linked by hydrogen bonds; (b) the three-dimensional supramolecular structure of **3** along the b axis (all hydrogen atoms are omitted for clarity; hydrogen bonds are represented by dashed lines).

O1C, O2A, O2B form the equatorial plane (the average Zn–O bond distance is 2.05 Å) and O7 occupies the axial position (the Zn–O bond distance is 1.98 Å), giving a square-pyramid geometry ($\tau = 0$). In **3**, dbsf^{2-} ligands adopt a bis(bridging-bidentate) mode (Scheme 1b) to coordinate to four Zn(II) ions, by contrast to the situation found for **1** and **2**. Thus, pairs of Zn(II) ions are joined by four dbsf^{2-} ligands to form a dinuclear unit, in which the Zn···Zn distance is 2.96 Å. These dinuclear units are connected into a one-dimensional double-chain, as shown in Fig. 4(b). The one-dimensional double-chain possesses voids formed by encircling the dinuclear units

and the dbsf^{2-} ligands. Along the a -axis, the double-chains pack in an ...ABA... fashion [Fig. 5(a)]. There is a C–H···O hydrogen bond between the C–H15 of the DMF molecule and the oxygen atom (O3) of dbsf^{2-} ligand (Table 3). The close π – π stacking interactions occur between phenyl rings of adjacent chains [with a face-to-face distance of 3.54 Å; see Fig. 5(a)]. In this way, a layer structure is formed via hydrogen bonds and π – π stacking interactions. Interestingly, the DMF molecules act as wings of the one-dimensional double-chains, spread out of the two-dimensional layer due to steric effect and thus locate between two-dimensional layers, as shown

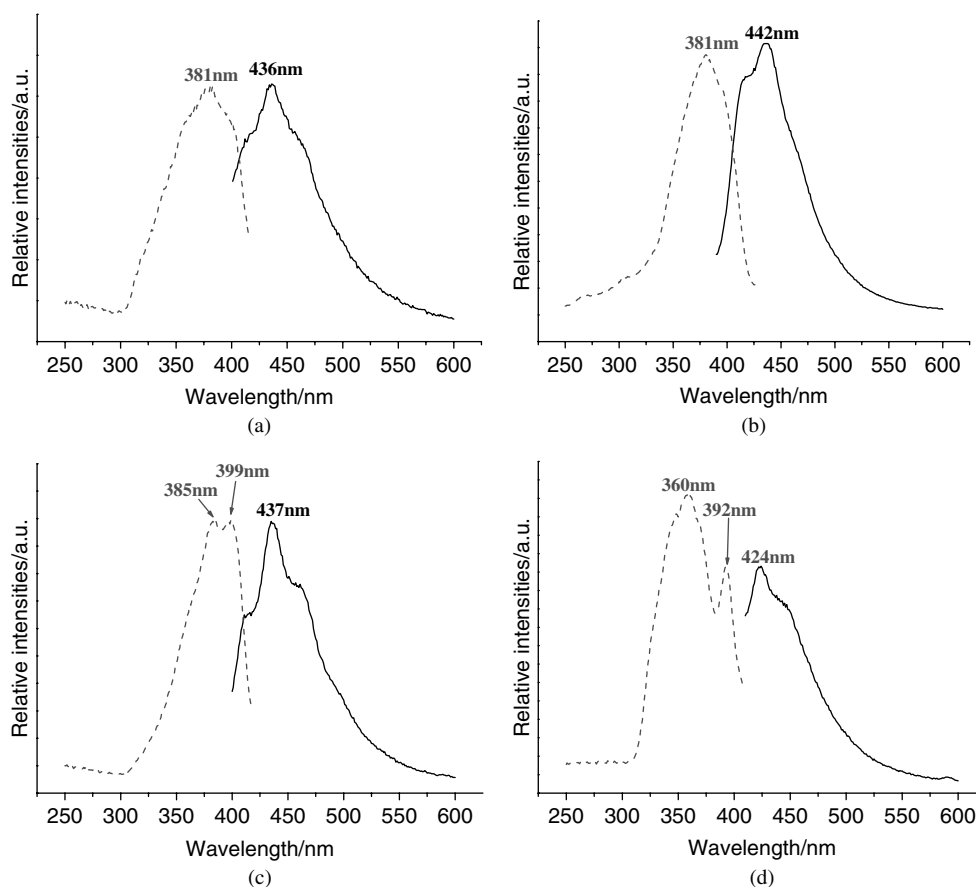


Figure 6. The excitation (dashed line) and emission spectra (solid line) of complexes **1** (a); **2** (b); **3** (c); and free H₂dbsf ligand (d).

in Fig. 5(b). All the two-dimensional layers array regularly, and are interlaced because the DMF molecules arrange in zipper-like mode [see Fig. 5(b)]. This regular arrangement makes the voids form channels but guest molecules are not accommodated. Computation of the channel void using PLATON²⁶ suggests a value of 1229.0 Å³, corresponding to 43.8% of the unit-cell volume (2809.5 Å³).

Photophysical properties

The fluorescent properties of complexes **1–3** in the solid state were measured and they exhibit strong blue fluorescence at room temperature. As shown in Fig. 6(a), the emission spectrum of complex **1** possesses a broad peak at 436 nm when excited at 381 nm. Fig. 6(b) shows the spectrum of complex **2**, with the emission peak at 442 nm at the excitation wavelength 381 nm. The emission peak position of complex **3** is similar to that of complex **1**, with a broad peak at 437 nm when excited at 385 nm [Fig. 6(c)]. We further measured the emission spectrum of the free H₂dbsf ligand, which displays one peak at 424 nm when excited at 392 nm [Fig. 6(d)]. The emission spectrum of H₂dbsf originates from the H₂dbsf $\pi^* \rightarrow \pi$ transition. From a comparison of complexes **1–3** with the free H₂dbsf ligand, we can see that their emission shapes and locations are similar, indicating

that the emission spectra of **1–3** possibly originate from a ligand-centered $\pi^* \rightarrow \pi$ transition of dbsf²⁻,²⁷ and red-shift with respect to the free H₂dbsf ligand is due to the increase in conjugation upon coordination and thus the lowering of emission state level on the ligand. On the other hand, it is noticeable that the position of emission peak of complex **2** red-shifts larger than complexes **1** and **3**, which may be related to the involvement of auxiliary 2,2'-bpy ligand.

CONCLUSION

In summary, three novel zinc(II) complexes have been successfully synthesized by reaction of zinc(II) salts with the H₂dbsf ligand. The coordination numbers of the zinc ions in these complexes are 4 (complex **1**) and 5 (complexes **2** and **3**). The dbsf²⁻ ligands adopt two coordination mode (see Scheme 1) in complexes **1–3**, and serve to link the Zn(II) ions to form three types of chains, and coupled with hydrogen bonds and $\pi-\pi$ interactions, which leads to the formation of three three-dimensional supramolecular networks. The photoluminescence properties of complexes **1–3** show that they possess blue-emitting properties.

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