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Two Zn coordination polymers with meso-helical chains based on mononuclear or dinuclear cluster units

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ABSTRACT

Two zinc coordination polymers $\{[Zn_2(TPPBDA)(oba)_2]\cdot DMF\cdot 1.5H_2O\}_n$ (**1**), $\{[Zn(TPPBDA)_{1/2}(tpdc)]\cdot DMF\}_n$ (2) have been synthesized by zinc metal salt, nanosized tetradentate pyridine ligand with flexible or rigid V-shaped carboxylate co-ligands. These complexes were characterized by elemental analyses and X-ray single-crystal diffraction analyses. Compound **1** is a 2-fold interpenetrated 3D framework with $[Zn_2(CO_2)_4]$ clusters. Compound **2** can be defined as a five folded interpenetrating **bbf** topology with mononuclear Zn^{2+}. These mononuclear or dinuclear cluster units are linked by mix-ligands, resulting in various degrees of interpenetration. In addition, the photoluminescent properties for TPPBDA ligand under different state and coordination polymers have been investigated in detail.

1. Introduction

In the past two decades, the design and synthesis of coordination polymers (CPs) by self-assembly of metal ions as connecting nodes and organic ligands as linkers have attracted scientific attention [1–5]. Coordination polymer is an ideal example of inorganic crystal engineering with various noncovalent interactions to obtain useful function materials with different dimensionalities and topologies and provide an important interface between synthetic and material chemistry. However, predictive coordination chemistry is still a challenge and deserves our careful study, because there are many facts that can affect the structure, such as, the counter anions of metal salts, geometric requirements of metal atoms, pH, solvent, template and the structure of the organic ligand [6–11]. As we know, the use of long linkers for the design of frameworks often affords interpenetrated MOFs with smaller pores [12–14]. Although interpenetration usually makes MOFs robust, it negatively reduces the size of open pores. Highly interpenetrated nets typically have low porosity and surface area, and high density, negatively affecting the potential applications since high surface area and porosity are generally most desired in porous materials. It is noteworthy that using rod-shaped or other secondary building units (SBUs) can reduce the interpenetration, which provides MOFs with rigid architectures and permanent porosity [15–17]. Herein we have synthesized two new zinc coordination polymers under solvothermal condition, that is, $\{[Zn_2(TPPBDA)(oba)_2]\cdot DMF\cdot 1.5H_2O\}_n$ (**1**), $\{[Zn(TPPBDA)_{1/2}(tpdc)]\cdot DMF\}_n$ (2) based on Zn (II) cation, nanosized neutral tetradentate ligand TPPBDA (N,N,N′,N′-tetrakis (4-(4-pyridine)-phenyl) biphenyl-4,4′-diamine) [18–20] and carboxylate co-ligands (H_2oba =4,4′-oxybis(benzoate) or H_2tpdc=4,4″-dicarboxyl-(1,1′,3′,1″)-terphenyl, DMF = N,N-dimethylformamide). In compounds **1**-**2**, Zn^{2+}cations act as the mononuclear or dinuclear SBUs, which are interconnected by TPPBDA and carboxylate co-ligands, resulting in structural diversity and various degrees of interpenetration.

2. Experimental section

2.1. Reagents and physical measurements

All chemicals and solvents used in the syntheses were of reagent grade and used without further purification. The ligand H_2tpdc was prepared by the literature methods [21]. The ligand TPPBDA was prepared as follows on the basis of palladium-

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catalyzed cross-coupling reaction.

Preparation of N, N, N′, N′-tetrakis(4-bromophenyl)biphenyl-4,4′-diamine: An oven-dried 500 mL Schlenk flask was charged with tris(4-bromophenyl)amine (24.1 g, 50 mmol), then ether (350 mL) and N,N,N′,N′-tetramethylethylenediamine (8.2 mL) were added through a rubber septum. The reaction mixture was cooled to −78 °C. Upon slow addition of a *n*-BuLi solution (34 mL, 1.6 M in hexanes, 55 mmol), and the mixture was stirred for 1 h at −78 °C. Solid $CuCl_2$ (10.8 g, 80 mmol) was added, and the reaction mixture was then allowed to slowly warm to room temperature and stirred for an additional 10 h. The reaction mixture was exposed to air, stirred for 30 min and concentrated in vacuo. Approximately chloroform (100 mL) and water (100 mL) were added, and the layers were separated. The aqueous layer was subsequently washed with chloroform (50 mL) three times. The organic extracts were combined, dried with $MgSO_4$, and filtered. The volatile chloroform was removed under vacuum to leave a green solid. Purification by column chromatography (petroleum ether) yielded the pure product as a white solid (10.1 g, 50%). ^{1}HNMR (DMSO-d, 500 MHz, 25 °C): 7.61 (d, 4H), 7.5 (d, 8H), 7.09 (d, 4H), 7.0 (d, 8H).

Preparation of N,N,N′,N′-tetrakis (4-(4-pyridine)-phenyl) biphenyl-4,4′-diamine (TPPBDA): A 500 mL Schlenk flask was evacuated and backfilled with nitrogen and charged with N, N, N′, N ′-tetrakis(4-bromophenyl)biphenyl-4,4′-diamine (8 g, 10 mmol), the pyridine-4-boronic acid (8.41 g, 70 mmol), and K_2CO_3 (9.66 mg, 70 mmol). The flask was evacuated and backfilled with nitrogen, then $Pd(PPh_3)_4$ (2.2 mg, 0.05 mmol, 5.0 mol%) and 1,4-dioxane / H_2O (V:V=3:2) were added through a rubber septum. The reaction mixture was heated to 95 °C with stirring until the starting N, N, N′, N′-tetrakis(4-bromophenyl) biphenyl-4,4′-diamine had been completely consumed as judged by TLC. The reaction mixture was then cooled to room temperature and the 1,4-dioxane was removed under vacuum. The aqueous mixture was extracted with chloroform (50 mL). The combined organic layers were dried over anhydrous $MgSO_4$, filtered, and concentrated in vacuo. The crude material was purified by chromatography on silica gel (ethyl acetate / methanol =10:1) yielded the pure product as a yellow solid (4.2 g, 53%). ^{1}HNMR (DMSO-d, 500 MHz, 25 °C): 8.60 (s, 8H), 7.80 (d, 8H), 7.68 (s, 12H), 7.19 (d, 12H). ^{13}CNMR (DMSO-d): δ, [ppm]: 119.42, 120.41, 121.04, 124.33, 125.63, 128.15, 128.53, 131.85, 135.50, 145.91, 146.65, 148.12, 150.67. MS (ESI-Tof): Calcd for $C_{56}H_{40}N_6$, 796; found, 797.

2.2. *Syntheses of compounds **1–2***

General procedure for the preparation of $\{[Zn_2(TPPBDA)(oba)_2]\cdot DMF\cdot 1.5H_2O\}_n$ (**1**): A mixture of $H_2O/DMF/CH_3CN$ containing the TPPBDA (79.6 mg, 0.1 mmol), 4,4′-H_2oba (25.8 mg, 0.1 mmol) and $Zn(NO_3)_2\cdot 6H_2O$ (29.7 mg, 0.1 mmol) was mixed in a Teflon vessel within the autoclave. The vessel was heated at 85 °C for 72 h and then cooled to room temperature. Transparent pale yellow block crystals were collected (yield based on TPPBDA ligand: ~20% for **1**). Elemental analysis calcd. for $C_{84}H_{56}N_6O_{10}Zn_2(C_3H_7NOH_3O_{1.5})$(**1**): C, 67.84; H, 4.32; N, 6.37, Found: C, 67.80; H, 4.29; N, 6.35. The IR spectra of the corresponding complex was shown in the Supporting Information (Fig. S1).

Synthesis of $\{[Zn(TPPBDA)_{1/2}(tpdc)]\cdot DMF\}_n$ (2): Compound **2** was synthesized by dissolving 0.05 mmol of TPPBDA and 0.1 mmol of H_2tpdc in DMF (3 mL) in a 10 mL glass vial. A solution of 2 mL 0.1 mmol aqueous $Zn(NO_3)_2$ solution was then added and the solution heated at 85 °C for 3 days. Transparent pale yellow prismatic crystals were formed. Yield of the reaction was ca. 40% based on TPPBDA ligand. Elemental analysis calcd. for $C_{51}H_{39}N_4O_5Zn$ (**1**): C, 71.79; H, 4.61; N, 6.57, Found: C, 71.80; H, 4.68; N, 6.55. The IR spectra of the corresponding complex was shown in the Supporting Information (Fig. S2).

2.3. *Single-crystal structure determination*

The measurements were made on a Bruker Apex Smart CCD diffractometer with graphite-monochromated Mo-K_a radiation (λ=0.71073 Å). The structure was solved by direct methods, and the non-hydrogen atoms were located from the trial structure and then refined anisotropically with SHELXTL using full-matrix least-squares procedures based on F^2 values [22]. The distributions of peaks in the channels of **1** and TPPBDA were chemically featureless to refine using conventional discrete-atom models. To resolve these issues, the contribution of the electron density by the remaining water molecule was removed by the SQUEEZE routine in PLATON [23]. Hydrogen atoms positions were fixed geometrically at calculated distances and allowed to ride on the parent atoms. A semiempirical absorption correction was applied using SADABS [24]. The topological analysis and some diagrams were produced using the TOPOS 4.0 program [25]. Crystallographic data and structural refinements for the TPPBDA ligand and two compounds were summarized in Table 1 and important bond distances are listed in Table S1. The CCDC reference numbers are 1410720 and 1444480 for **1** and **2**. The data can be obtained free of charge via < http://www.ccdc.cam.ac.uk/conts/retrieving.html >, or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; fax:(+44) 1223–336–033; or e-mail: deposit@ccdc.cam.ac.uk.

3. Results and discussion

3.1. *Structure of $\{[Zn_2(TPPBDA)(oba)_2]\cdot DMF\cdot 1.5H_2O\}_n$ (**1**)*

Compound **1** crystallizes in the monoclinic crystal system C2/c space group with two Zn cations, one TPPBDA, two oba^{2-}and squeezed solvent in the asymmetric unit (Fig. 1a). The solvent of compound **1**, that is one DMF and one and half of lattice water molecules in per asymmetric unit, has been determined by elemental analysis and thermogravimetric analysis. Each Zn(II) unit

Table 1
Crystal data and structure refinements parameters of TPPBDA, complexes **1–2**.

Complex	**1**[a]	**2**	TPPBDA[a]
Formula	$C_{84}H_{56}N_6O_{10}Zn_2$	$C_{51}H_{39}N_4O_5Zn$	$C_{56}H_{40}N_6$
Mr	1440.09	853.23	796.94
Cryst syst	Monoclinic	Monoclinic	Monoclinic
Space group	*C2/c*	*P2₁/c*	*P2₁/c*
a (Å)	21.2969(18)	8.9575(8)	16.293(2)
b (Å)	37.398(3)	19.6850(18)	16.709(2)
c (Å)	28.267(2)	25.063(2)	17.586(2)
α (°)	90.00	90.00	90.00
β (°)	109.7320(10)	96.015(2)	92.956(2)
γ (°)	90.00	90.00	90.00
V (Å^3)	21192(3)	4395.0(7)	4781.2(10)
Z	8	4	4
ρ_{calcd} (g cm^{-3})	0.903	0.611	1.107
μ (mm^{-1})	0.497	0.498	0.066
rflns collected	64171	31908	35187
Uniq. rflns	11061	5316	4182
R(int)	0.0193	0.0722	0.0731
GOF(F^2)	1.002	1.066	1.030
$R_1[I > 2\sigma(I)]$[b]	0.0500	0.0741	0.0669
$wR_2[I > 2\sigma(I)]$[c]	0.1329	0.2112	0.1603
Min. and max resd dens (e Å^{-3})	−0.376, 0.406	−0.740, 0.826	−0.286, 0.269

[a] The residual electron densities were flattened by using the SQUEEZE option of PLATON.

[b] $R_1=\Sigma||F_o|-|F_c||/|\Sigma|F_o|$.

[c] $wR_2=\{\Sigma[w(F_o^2-F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$; where $w=1/[\sigma^2(F_o^2)+(aP)^2+bP]$, $P=(F_o^2+2F_c^2)/3$.

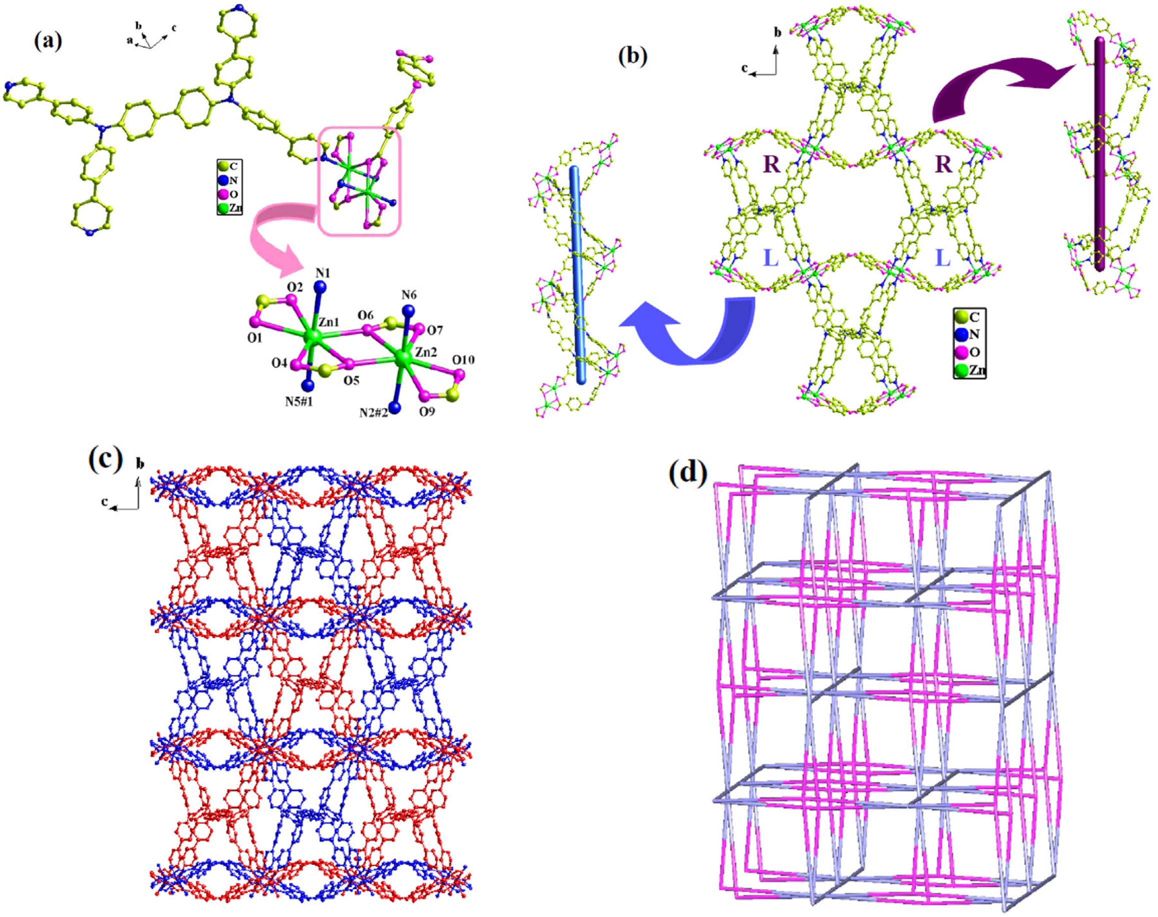

Fig. 1. (a) Coordination environments of complex **1** (30% ellipsoid probability). Most hydrogen atoms and solvent molecules are omitted for clarity. Symmetry codes for **1**: #1 = $-0.5+x$, $0.5-y$, $0.5+z$; #2 = 1.5-x, $-0.5+y$, $0.5-z$. (b) View of 3D structure with meso-helical chains (marked by L and R) in net **1**. (c) Schematic view of two interpenetrating framework. (d) Schematic view of the topology of structure **1** (oba^{2-} ligands were simplified into linkers).

has a seven coordination geometry. The carboxylate groups of oba^{2-} ligand takes bidentate-chelating and chelating-bridging tridentate coordination modes, respectively. Two Zn(II) ions are bridged by carboxylate oxygen atoms to form a four-membered $[Zn_2(CO_2)_4]$ ring. The oba^{2-} anions and TPPBDA ligands connect to the $[Zn_2(CO_2)_4]$ units to generate a 3D net.

The three-dimensional net **1** consisted of meso-helical chains (right handed helixes and left handed helixes), which are labeled as R and L chains with an approximate dimension of 9.30 Å × 16.87 Å (Fig. 1b). The oba^{2-} and TPPBDA bridge $[Zn_2(CO_2)_4]$ units into a 2_1 helix with a helical pitch of 21.30 Å. However, left-handed and right-handed helical chains coexist in the crystal structure, the whole crystal is racemic and exhibit no chirality.

The overall structure is that two equivalent nets adopt 2-fold interpenetration to minimize the large voids in the single net to form a doubly interpenetrated framework (Fig. 1c). To better understand the structure, topological approach can be taken. In this structure, TPPBDA ligands linking four $[Zn_2(CO_2)_4]$ clusters can act as 4-connected nodes; $[Zn_2(CO_2)_4]$ clusters can be seen as 6-connected nodes. On the basis of the simplification principle, the finally structure is a (4,6)-connected net with point symbol $\{4^6\}\{4^20.6^80.8^5\}$ (Fig. 1(d)).

3.2. *Structure of $\{[Zn(TPPBDA)_{1/2}(tpdc)]\cdot DMF\}_n$ (2)*

X-ray crystallography reveal the asymmetric unit of **2** contains one Zn(II) cation, half a TPPBDA ligand, one tpdc^{2-} anion, and one lattice DMF molecule (Fig. 2a). The Zn center is six coordinated by four oxygen atoms belonging to two oba ligands and two nitrogen atoms from TPPBDA ligands with the Zn-O distances ranging from 2.062(7) Å to 2.359(8) Å and the Zn-N distances ranging from 2.067(3) Å and 2.108(4) Å. The tetradentate TPPBDA ligands and tpdc^{2-} anions bridge the Zn (II) ions into a 3D network containing 1D meso-helical chains with a helical pitch of 19.69 Å along the *b*-axis, which are presented by L and R with an approximate dimension of 12.75 Å × 16.09 Å (Fig. 2b). The whole crystal is also racemic and does not exhibit chirality. Considering the Zn(II) cations and the TPPBDA ligands as 4-c nodes, **2** can be defined as a 4-connected net with **bbf** topology as shown in Fig. 2c. In order to minimize the presence of large cavities and to stabilize the framework during the assembly process, other four identical networks are filled in the cavities giving a 5-fold interpenetrating 3D architecture (class Ia) (Fig. 2d).

3.3. *The comparison of the configuration for TPPBDA ligand in different compounds*

The distortion of TPPBDA can be assessed by comparing the dihedral angles defined by the central benzene groups (A and B) and the torsion angle of Npyridyl-Ncore-Npyridyl (1 and 2). In TPPBDA, central benzene groups are almost in parallel. The angles of 1 and 2 are 120.564(42)° and 116.686(60)° (Fig. 3). In compound **1**, the ligand TPPBDA adopts a distorted conformation, and the dihedral angle between A and B is 15.262(99)°. The angles of 1 and 2 are 118.532(34)° and 124.605(34)°. In compound **2**, the dihedral angle between A and B is almost zero. The angles of 1 and 2 are both 114.978(52)°.

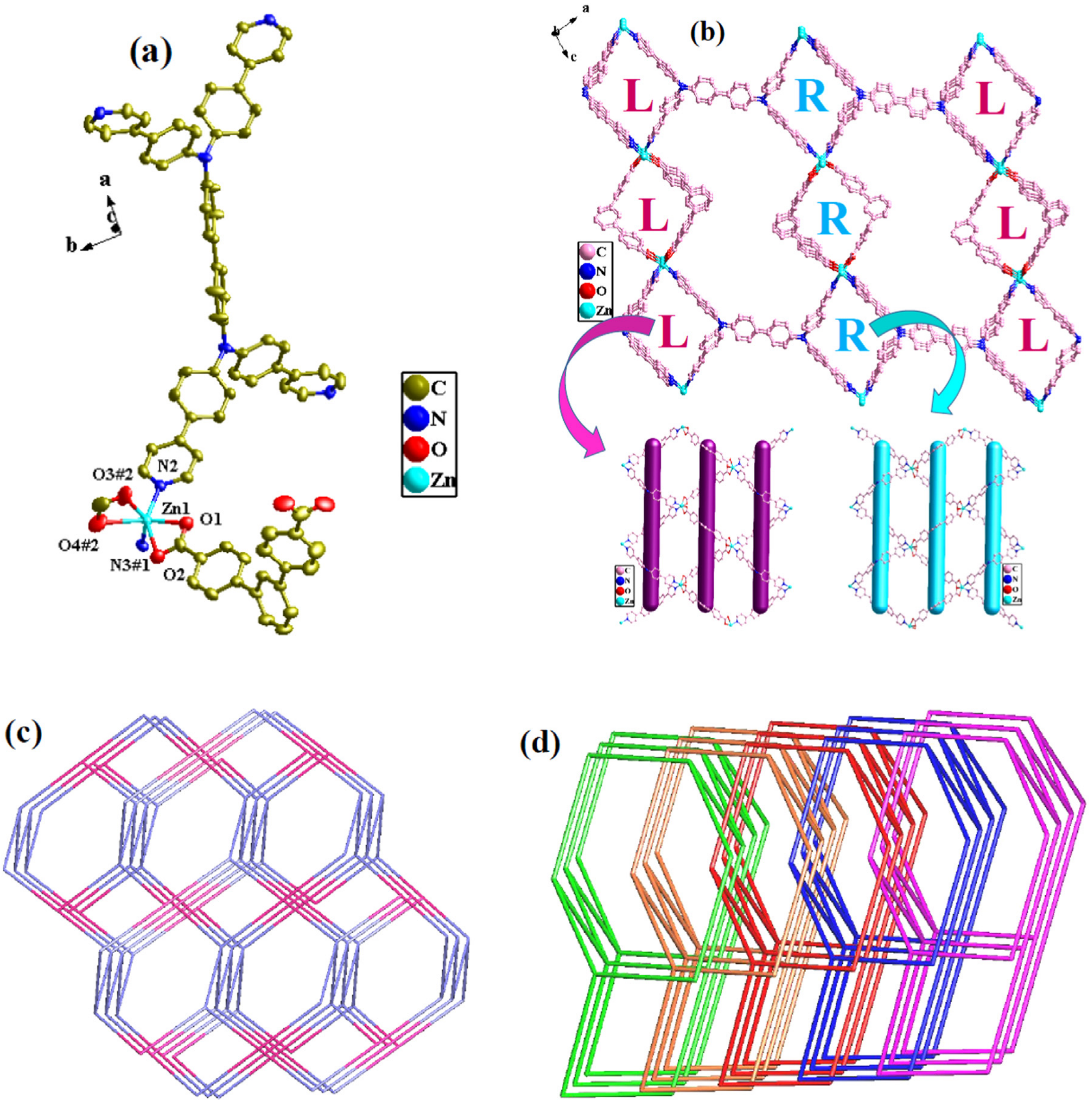

Fig. 2. (a) Coordination environments of complex **2** (30% ellipsoid probability). Most hydrogen atoms and solvent molecules are omitted for clarity. Symmetry codes for **2**: #1=1−*x*, 0.5+*y*, 1.5-*z*; #2=−*x*, 0.5+*y*, 2.5−*z*. (b) View of 3D structure with meso-helical chains (marked by L and R) in net **2**. (c) Schematic view of **bbf** topology of structure **2** (tpdc^{2-}ligands were simplified into linkers). (d) Schematic view of five interpenetrating **bbf** framework.

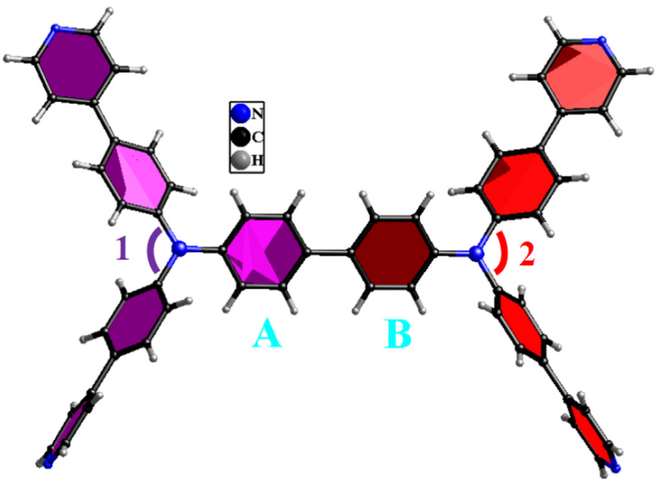

Fig. 3. The structure of TPPBDA ligand (the solvent molecules are squeezed).

3.4. Optical properties

The photoluminescent properties of d_{10} metal coordination compounds have been attracting more interest for their potential applications as fluorescence-emitting materials. Therefore, in this work, the solid-state emission spectra for complexes **1** and **2** were studied in the solid state at room temperature (Fig. 4 and S3). The emission spectra have peaks for **1** and **2** with maxima at 541 nm, 575 nm, respectively. In order to understand the nature of above emission bands, the luminescence property of the TPPBDA ligand in the solid state has also been measured (λ_{em}=555 nm). Compared to these emission peaks of TPPBDA ligand, the emissions of **1** and **2** may be attributed to intraligand transition. The differences in the band positions of these two coordination polymers might be related to the differences in the coordination environments or the degree of interpenetration in these structures [26]. In addition, the emission spectra of TPPBDA in crystalline state and in organic solvent are investigated (Figs. 5 and S4,S5). The results show that the emission

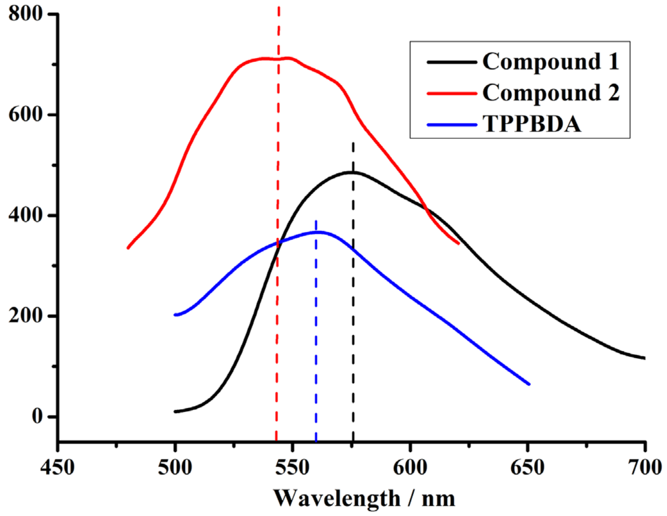

Fig. 4. Solid-state photoluminescent spectra of compounds **1**, **2** and TPPBDA ligand at room temperature.

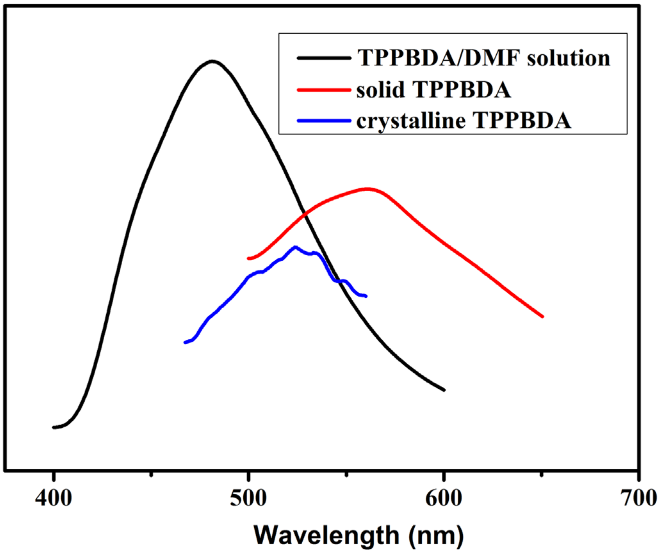

Fig. 5. The photoluminescent spectra of TPPBDA ligand in different state.

position for TPPBDA ligand is related with the status. The crystalline-state emission spectrum for the TPPBDA exhibits a messy broad peak at 522 nm, while the solution-state emission spectrum for the TPPBDA exhibits a sharp peak at 481 nm, which is probably due to the chromophores in **1** have interactions with solvents.

3.5. Thermogravimetric analyses and X-ray powder diffraction analyses

To examine the thermal stabilities of these compounds, TG analyses were carried out (Fig. S6). For compound **1**, a rapid weight loss was observed from 20 to 122 °C which was ascribed to the loss of one DMF and one and half of lattice water molecules, with a weight loss of 6.00% (calcd 6.49%), then the decomposition of the complex **1** started. The weight loss of 8.75% for compound **2** from 20 to 270 °C corresponded to the loss of one lattice DMF molecule (calcd 8.56%) then the organic ligands began to decompose. The purities of polymers **1**–**2** were confirmed by X-ray power diffraction analyses, in which the experimental spectra of polymers **1**–**2** were almost consistent with their simulated spectra (Figs. S7,S8).

4. Conclusions

We have synthesized two new zinc coordination polymers $\{[Zn_2(TPPBDA)(oba)_2]\cdot DMF\cdot 1.5H_2O\}_n$ (**1**), $\{[Zn(TPPBDA)_{1/2}(tpdc)]\cdot DMF\}_n$ (2) based on a nanosized neutral ligand and two carboxylate co-ligands. They are three-dimensional nets consisted of meso-helical chains, that is, right handed helixes and left handed helixes. Compound **1** is a 2-fold interpenetrated 3D framework with dinuclear clusters. Compound **2** can be defined as a five fold-interpenetrating **bbf** topology with mononuclear cation. These mononuclear or dinuclear cluster units are interconnected by TPPBDA and carboxylate co-ligands, resulting in interesting structural diversity and various degrees of interpenetration.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.jssc.2016.04.021.

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