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# Hydrothermal syntheses, crystal structure and luminescent properties of four zinc(II) coordination polymers based on tipodal imidazole

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## 1. Introduction

The construction of metal-organic coordination polymers have been given considerable attention, not only for their promising applications as functional material but also for their intriguing aesthetic structures and topological features [1–11]. On the basis of this background, many metal-organic coordination polymers have been constructed via the interaction of metal ions with well defined bridging organic ligands. Although the rapid progress in metal-organic coordination polymers has been made, it is also a significant challenge to rationally prepare and control the structures and composition of target products in crystal engineering since many factors could affect on the construction, such as organic ligands, pH value, solvent system, anion and so on [13-16]. Among these factors, the most effective and facile method to construct new metal-organic coordination polymers is the judicious selection of the appropriate organic ligands. Imidazole-containing ligands have proven to be a class of aromatic N-donor for the construction of the novel metalorganic coordination polymers [16–23].

In this paper, we used the 1,3,5,-tris(1-imidazolyl) benzene and dicarboxylate ligands to construct different dimensional networks and topology. The tib ligand has been previously used in the construction of MOFs (metal–organic frameworks) with specific structures and topologies, all of which demonstrating that tib is a useful ligand in construction of MOCPs [24–26]. In this paper, four new coordination polymers  $[Zn(tib)_2(H_2O)_2(NO_3)_2]_n$  (1),  $[Zn(tib)_2Cl_2]_n$  (2),  $[Zn(tib)(L_1)]_n$  (3) and  $[Zn(tib)(L_2)(H_2O)]_n$  (4) were successfully

#### ABSTRACT

Four new Zn(II) coordination polymers  $[Zn(tib)_2(H_2O)_2(NO_3)_2]_n$  (1),  $[Zn(tib)_2Cl_2]_n$  (2),  $[Zn(tib)(L_1)]_n$  (3) and  $[Zn(tib) (L_2) (H_2O)]_n$  (4) [tib = 1,3,5,-tris(1-imidazolyl) benzene,  $H_2L_1$  = 5-hydroxyisophthalic acid and  $H_2L_2$  = 5-methylisophthalic acid] have been prepared and characterized by elemental, IR, XRPD and X-ray diffraction analyses. The structure determination reveals that complex **1** is a 2D layer network and exhibits a typical (4,4)-topological net, which further assembles into a 3D (3,6)-connected framework with (4<sup>4</sup>.6<sup>10</sup>.8) topological symbol via hydrogen bonding interactions. Complex **2** shows a 2D (3,6)-connected topological net the Schläfli symbol of (4<sup>3</sup>)<sub>2</sub>(4<sup>6</sup>.6<sup>6</sup>.8<sup>3</sup>). Complex **3** features a (4,4)-topological net and further assembles into a 3D pcu framework by hydrogen bonding interactions. Complex **4** is a 1D ladder-like chains structure. Furthermore, the photoluminescence properties are investigated. © 2013 Elsevier B.V. All rights reserved.

synthesized under hydrothermal conditions. The luminescent properties of the complexes were also investigated.

Inorganica <u>Chimica</u> Acta

#### 2. Experimental

#### 2.1. Materials and physical measurements

All reagents and solvents employed were commercially available and were used as received without further purification. Elemental analysis was carried out on a Carlo Erba 1106 full-automatic trace organic elemental analyzer. FT-IR spectra were recorded with a Bruker Equinox 55 FT-IR spectrometer as a dry KBr pellet in the 400– 4000 cm<sup>-1</sup> range. Solid-state fluorescence spectra were recorded on a F-4600 equipped with a xenon lamp and a quartz carrier at room temperature(slit width = 5 nm and scan rate = 15 nm s<sup>-1</sup>). The powder X-ray powder diffraction (XRPD) measurements were performed on a Bruker D8 diffractometer operated at 40 kV and 40 mA using Cu K $\alpha$  radiation ( $\lambda$  = 0.15418 nm).

# 2.2. Syntheses

# 2.2.1. Preparation of $[Zn(tib)_2(H_2O)_2(NO_3)_2]_n$ (1)

A mixture of  $Zn(NO_3)_2 \cdot 6H_2O$  (0.149 g, 0.5 mmol), tib (0.138 g, 0.5 mmol) and deionized water (15 mL) was sealed in a 25 mL Teflon-lined stainless steel vessel and heated at 160 °C for 96 h, and cooled to room temperature. The colorless block crystals were obtained and washed with alcohol for several times (Yield: 41% based on Zn). *Anal.* Calc. for C<sub>30</sub>H<sub>28</sub>ZnN<sub>14</sub>O<sub>8</sub>: C, 46.31; H, 3.63; N, 25.20. Found: C, 46.28; H, 3.65; N, 25.21%. IR: 3417 br, 1624 s, 1519 s, 1454 m, 1345 m, 1250 m, 1114 m, 1010 m, 940 m, 856 m, 728 m.



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#### 2.2.2. Preparation of $[Zn(tib)_2Cl_2]_n$ (2)

A mixture of ZnCl<sub>2</sub> (0.068, 0.5 mmol), tib(0.138 g, 0.5 mmol) and deionized water (15 mL) was sealed in a 25 mL Teflon-lined stainless steel vessel and heated at 160 °C for 96 h, and cooled to room temperature. The colorless block crystals were obtained and washed with alcohol for several times (Yield: 46% based on Zn). *Anal.* Calc. for  $C_{30}H_{24}ZnN_{12}Cl_2$ : C, 43.38; H, 2.91; N, 20.23%. Found: C, 43.43; H, 2.89; N, 20.27. IR: 3445 br, 1611s, 1510 m, 1371 m, 1238 m, 1157 m, 1076 m, 9734 m, 849 m, 754 m.

#### 2.2.3. Preparation of $[Zn(tib)(L_1)]_n$ (3)

A mixture of Zn(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O (0.149 g, 0.5 mmol),tib (0.138 g, 0.5 mmol), H<sub>2</sub>L<sub>1</sub> (0.5 mmol, 0.091 g) and NaOH (0.04 g, 1 mmol) and deionized water (18 mL) was sealed in a 25 mL Teflon-lined stainless steel vessel and heated at 160 °C for 96 h, and cooled to room temperature. The colorless block crystals were obtained and washed with alcohol for several times (Yield: 45% based on Zn). *Anal.* Calc. for C<sub>23</sub>H<sub>16</sub>ZnN<sub>6</sub>O<sub>5</sub>: C, 52.94; H, 3.09; N, 16.11. Found: C, 53.01; H, 3.12; N, 16.06%. IR: 1625 s, 1567 s, 1401 m, 1353 m, 1242 m, 1111 m, 1069 m, 975 m, 853 m, 788 m.

#### 2.2.4. Preparation of $[Zn(tib) (L_2) (H_2O)]_n$ (4)

The compound **4** was obtained by the similar method as described for **3** by using of  $H_2L_2$  (0.5 mmol, 0.09 g) in place of  $H_2L_1$ . The colorless crystals were obtained in pure phase, washed with water and ethanol, and dried at room temperature (Yield: 55% based on Zn). *Anal.* Calc. for  $C_{24}H_{20}ZnN_6O_5$ : C, 53.59; H, 3.75; N, 15.62. Found: C, 53.67; H, 3.72; N, 15.61%. IR: 3434 br, 1620 s, 1514 m, 1408 s, 1358 m, 1255 m, 1112 m, 1070 m, 941 m, 854 m, 763 m.

#### 2.3. X-ray crstallography

Diffraction intensity data of the single crystals of four compounds were collected on a Bruker SMART APEXII CCD diffractometer equipped with a graphite monochromated Mo K $\alpha$  radiation ( $\lambda$  = 0.71073 Å) by using a  $\omega$ -scan mode. Empirical

absorption correction was applied using the sADABS programs [27]. All the structures were solved by direct methods and refined by full-matrix least-squares methods on  $F^2$  using the program SHEXL 97 [28]. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms were located by geometrically calculations, and their positions and thermal parameters were fixed during the structure refinement. The C atoms (C29A, C29B, C30A, C30B, C35A, C35B, C36A and C36b) in complex **4** are disordered two positions. The crystallographic data are summarized in Table 1. The date of relevant bond lengths and angles are listed in Table S1.

## 3. Results and discussions

#### 3.1. Description of crystal structures

#### 3.1.1. $[Zn(tib)_2(H_2O)_2(NO_3)_2]_n$ (1)

Single crystal X-ray diffraction analysis revealed that compound **1** crystallizes in the monoclinic space group  $P2_1/n$ , and each asymmetric unit consists of Zn(II) ion, one tib ligands and one coordination water molecule. As illustrated in Fig. 1a, the Zn(II) ion is located at an inversion center with an octahedral geometry surrounded by four nitrogen atoms from four different tib ligands occupying the basal positions [Zn(1)-N(1) = 2.153(2) Å] and Zn(1)-N(4) = 2.140(2) Å] and two water molecule forming the apical positions [Zn(1)-O(1w) = 2.197(2) Å].

The tib ligands link Zn(II) ions to form a two-dimensional layer. The nitrate ions distribute in the middle of layers. The nitrate ions and coordinated water molecules interact by the intermolecular hydrogen bonds which lead to form a three-dimensional framework (Fig. 2b). The hydrogen-bonded right- and left-helical chains are constructed by oxygen atoms of nitrate ions and oxygen atoms of coordinated water molecules through the hydrogen bonds  $[01w\cdots 01\ 3.036(4)$ Å and  $01w\cdots 03\ 2.818(3)$ Å]. The pitch of the hydrogen-bonded helical chain is 11.513 Å. In the view of topology, the 3D supramolecular framework can be simplified the 3,6-connected net with  $(4^4.6^{10}.8)$  topological symbol. The tib ligands link

#### Table 1

Crystallographic data and	structure refinement	summary for compl	exes 1-4.
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Complex	1	2	3	4
Empirical formula	C <sub>30</sub> H <sub>28</sub> ZnN <sub>14</sub> O <sub>8</sub>	$C_{30}H_{24}Cl_2N_{12}Zn$	$C_{23}H_{16}ZnN_{6}O_{5}$	$C_{24}H_{20}ZnN_6O_5$
Formula weight	778.05	688.88	521.79	537.85
Crystal system	monoclinic	trigonal	monoclinic	triclinic
Space group	$P2_1/n$	P31c	P21/c	ΡĪ
Unit cell dimensions				
a (Å)	10.628(7)	11.472(3)	9.699(5)	8.020(1)
b (Å)	11.513(7)	11.472(3)	13.677(5)	17.175(3)
<i>c</i> (Å)	13.554(9)	15.217(4)	16.584(5)	17.577(3)
α (°)	90.000	90.000	90.000	97.742(2)
β (°)	109.744(8)	90.000	93.644 (5)	96.306(2)
γ (°)	90.000	120.000	90.000	96.306(2)
$V(Å^3)$	1561.0(12)	1734.4(12)	2195.5(15)	2358.3(7)
$ZD_{calc} (mg/m^3)$	21.655	21.319	41.579	21.515
Independent reflections $(I > 2\sigma(I))$	3039	1057	5004	6309
F(000)	800	704	1064	1104
$\theta$ (°)	2.38-27.52	2.05-27.58	1.93-27.51	2.27-22.32
Limiting indices	$-7 \leqslant h \leqslant 13$	$-14\leqslant h\leqslant 14$	$-12\leqslant h\leqslant 12$	$-8\leqslant h\leqslant 10$
	$-14\leqslant k\leqslant 14$	$-10\leqslant k\leqslant 14$	$-17 \leqslant k \leqslant 16$	$-22\leqslant k\leqslant 21$
	$-17 \leqslant l \leqslant 16$	$-19\leqslant l\leqslant 19$	$-21 \leqslant l \leqslant 20$	$-22\leqslant l\leqslant 19$
Goodness-of-fit (GOF) on F <sup>2</sup>	1.053	1.016	1.040	1.002
$R_1^{a}, wR_2^{b} [I > 2\sigma(I)]$	$R_1 = 0.0335$	$R_1 = 0.0581$	$R_1 = 0.0381$	$R_1 = 0.0508$
	$wR_2 = 0.0851$	$wR_2 = 0.0.1511$	$wR_2 = 0.0974$	$wR_2 = 0.1074$
$R_1^a$ , $wR_2^b$ (all data)	$R_1 = 0.0406$	$R_1 = 0.0684$	$R_1 = 0.0524$	$R_1 = 0.0988$
	$wR_2 = 0.0888$	$wR_2 = 0.1552$	$wR_2 = 0.1041$	$wR_2 = 0.1266$
Largest difference in peak and hole $(e/Å^3)$	0.493 and -0.308	0.464and -0.335	0.633 and -0.301	0.478 and -0.350

<sup>a</sup>  $R = \Sigma(||F_0| - |F_C||)/\Sigma|F_0|.$ 

<sup>b</sup>  $wR = [\Sigma w(|F_0|^2 - |F_C|^2)^2 / \Sigma w(F_0^2)]^{1/2}.$ 



Fig. 1. (a) Local coordination geometry of the central Zn(II) atom of compound (all hydrogen atoms are omitted for clarity; (b) Viewing of the left- and right helical chains. The hydrogen bonds are indicated by dotted lines; (c) The 3,6-connected net formed *via* the interaction of sql and hcb layers interwove.



**Fig. 2.** (a) Local coordination geometry of the central Zn(II) atom of compound (all hydrogen atoms are omitted for clarity); (b) the (3,6)-connected framework with the Schläfli symbol of  $(4^3)_2(4^6,6^6,8^3)$ .

the Zn(II) ions to form the (4,4)-connected sql layer, and the coordinated water molecules and nitrate ions link Zn(II) ions to form the 3-connected hcb topology (Figs. S1 and S2). The sql layer and the hcb layer are interwove to form the (3,6)-connected net (Fig. 2c).

# 3.1.2. $[Zn(tib)_2Cl_2]_n$ (2)

The single-crystal X-ray diffraction analysis revealed that there is one crystallographically independent zinc(II) atom in compound **2**. As illustrated in Fig. 2a, Zn(II) ion is coordinated by six nitrogen atoms [Zn-N = 2.195(3) Å] from different tib ligands, forming an octahedral geometry. The dihedral angle of phenyl ring and imidazole rings is 43.94° (Fig. S3).

The Zn(II) ion can be viewed as a node which is six-connector since it is linked by six tib ligands and each tib ligand can be regarded as a three-connector. Such connectivity repeats to give the 2D framework with the Schläfli symbol of  $(4^3)_2(4^6.6^6.8^3)$  (Fig. 2b).

#### 3.1.3. $[Zn(tib)(L_1)]_n$ (3)

X-ray crystallography reveals that compound **3** crystallizes in monoclinic space group  $P2_1/c$ . As shown in Fig. 3a, each Zn(II) ion is surrounded by two oxygen atoms [Zn(1)-O(1) = 1.931(2) Å and Zn(1)-O(3) = 1.946(2) Å] from two individual L<sub>1</sub> ligands and two nitrogen atoms [Zn(1)-N(1) = 2.025(2) Å and Zn(1)-N(6) = 2.027(2) Å] from tib ligands, showing a tetrahedral geometry. In tib ligand, one of imidazole group does not participate in the



**Fig. 3.** (a) Local coordination geometry of the central Zn(II) atom of compound (all hydrogen atoms are omitted for clarity; (b) simplification of the structure to a (4,4) network; (c) 3D supramolecular structure via hydrogen bonding interactions; (d) the pcu topology for the 3D supramolecular structure.



**Fig. 4.** (a) Local coordination geometry of the central Zn(II) atom of compound (all hydrogen atoms are omitted for clarity; (b) 32-membered tetrametal ring formed by  $L_2$  ligands; (c) the 1D chains formed by tib ligands; (d) the 1D ladder-like chain formed by  $L_2$  and tib ligands.

coordination, and thus each tib ligand connects two zinc(II) ions. The Zn(II) ions are linked by carboxylate groups to form a chain and further connected by tib ligands to form a two-dimensional layer (Fig. 3b). The 2D layers are further assembled by the intermolecular hydrogen bonding interaction with a  $H(5) \cdots O(2)$  distance of 1.95 Å and the angle of  $168^{\circ}$  [O(5)–H(5)  $\cdots$ O(2)], which leading to the formation of a 3D framework (Fig. 3c).

Considered the hydrogen bonding interaction, The Zn(II) can be regarded as six-connected nodes and the 3D supramolecular structure can be rationalized as a 6-connected pcu net with a Schäfli symbol of  $(4^{12}.6^3)$  (Fig. 3d).

#### 3.1.4. $[Zn(tib)(L_2)(H_2O)]_n$ (**4**)

The single crystal X-ray diffraction study reveals that compound **4** crystallizes in triclinic space group  $P\bar{1}$ . As depicted in Fig. 4a, the structure of the compound **4** contains two crystallographically unique (but the coordination number and geometry identical) zinc atoms. The Zn1 is coordinated by three oxygen atoms [Zn(1)–O(1) = 1.967(2) Å, Zn(1)–O(5) = 2.410(3) Å and Zn(1)–O(7) = 2.068(3) Å] and two nitrogen atoms [Zn(1)–N(1) = 2.043(3) Å and Zn(1)–N(7) = 2.027(3) Å], exhibiting a square–pyramidal geometry. The tib ligands link zinc(II) atoms to form a 1D zigzag chain along *c*-axis. The L<sub>2</sub> ligands connect zinc atoms to construct 32-membered



Fig. 5. Fluorescent emission spectra of compounds 1-4 in the solid state at room temperature.

tetrametal ring. The 32-membered tetrametal rings and 1D zigzags are combined to form a 1D ladder-like chain.

#### 3.2. Structure diversity of the complexes 1-4

When the metal salts are changed from  $Zn(NO_3)_2$ · $6H_2O$  to  $ZnCl_2$ , the topological structures of the complexes **1** and **2** are changed from the 3D supramocular structure to 2D layer structure. In **3** and **4**, the 5-position substituted groups are changed which lead to the structure from 3D supramolecular to 1D ladder-like chain.

# 3.3. XRPD of the complexes

In order to check the phase purity of compounds **1–4**, the X-ray powder diffraction (XRPD) patterns were measured at room temperature. As shown in Fig. S4, the peak positions of the simulated and experimental XRPD patterns are in agreement with each other, demonstrating the good phase purity of the compounds.

#### 3.4. Luminescent properties of the complexes

Metal–organic coordination polymers, especially those with Zn(II) ion, have been investigated for their photoluminescent properties and potential applications as fluorescence emitting materials [29–32]. As depicted in Fig. 5, the solid-state fluorescent properties of compounds **1–4** have been investigated at room temperature. Intense luminescence emission bands are observed at 416 nm ( $\lambda_{ex}$  = 352 nm) for compound **1**, 405 nm ( $\lambda_{ex}$  = 348 nm) for compound **2**, 364 nm ( $\lambda_{ex}$  = 325 nm) for compound **3** and 389 nm ( $\lambda_{ex}$  = 328 nm) for compound **4**, respectively. Intense emission bands have been observed at 405 nm ( $\lambda_{ex}$  = 280 nm), 380 nm ( $\lambda_{ex}$  = 280 nm) for free tib, H<sub>2</sub>L<sub>1</sub> and H<sub>2</sub>L<sub>2</sub> ligands [33–35]. The emissions of complexes **1–4** are tentatively assigned to the intraligand transition due to their similarity to ligands, since Zn(II) is difficult to oxidize or to reduce due to its d<sup>10</sup> configuration[36,37].

#### 4. Conclusions

In this paper, four Zn(II) metal coordination polymers have been successfully prepared under hydrothermal conditions. When we charged the metal salts from  $Zn(NO_3)_2 \cdot 6H_2O$  to  $ZnCl_2$ , the complexes **1** and **2** show the structural differences which indicate that the type of inorganic salts can affect the framework of the

complexes. Structural comparisons indicate that the coexistent groups of organic ligands on the dicarboxylate ligand play an important role in governing the structures of complexes **3** and **4**. Furthermore, the complexes **1–4** exhibit emission in the solid state at room temperature.

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#### Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.ica.2013.01.003.

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