Research Article

Synthesis, Structure, and Thermal Stability of Supramolecular Polymers with 1D Chain:

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Two supramolecular polymers $[\text{M}(\text{BPP})_2(\text{TBTA})(\text{H}_2\text{O})_2]_n$ [$\text{M} = \text{Mn}(\mathbf{1}), \text{Co}(\mathbf{2})$] have been constructed from 2,3,5,6-tetrabromoterephthalic acid (H_2TBTA) and 1,3-bis(4-pyridyl)propane (BPP) ligands in water-methanol dissolvant. Single-crystal X-ray and powder diffraction, IR spectroscopy, thermogravimetric analysis, and elemental analysis were employed to characterize all samples. Both of the complex compounds **1** and **2** belong to triclinic crystal system with *P*-1 space group. The crystal structures analysis indicates that the metal atom coordinated by two carboxylate groups from different TBTA ligands, two BPP molecules and two H_2O molecules formed a slightly distorted octahedral configuration. The compounds of **1** and **2** are 1D chains made with TBTA spacers, BPP molecules, and H_2O molecules.

1. Introduction

Rational design and preparation of supramolecular polymers have attracted considerable attention owing to their potential applications in optics, magnetism, adsorption, ion exchange, catalysis, and so forth [1–7]. In principle, the most effective approach for producing such metal-organic supramolecular polymers is primarily dependent on the rational choice of the organic-bridging ligands as well as the metal ions [8–13]. To date, polycarboxylate ligands, especially the rigid dicarboxylate ligands, have been commonly used in preparing the novel metal-organic frameworks [14–17]. Comparison to terephthalic acid, the ortho substituents on the related ligands can significantly affect the relative orientation of the carboxylate groups. Some scholars regard the effect of high steric hindrance groups as the special “knockon effect” of H_2TBTA [18–21]. Besides, some complexes of carboxylate acid with secondary organic ligands (such as 2,2'-bipyridine, 4,4'-bipyridine, and 1,3-bis(4-pyridyl)propane) have been widely investigated. Different from 4,4'-bipyridine and its derivatives, 1,3-bis(4-pyridyl)propane is flexible and can adopt different conformations characterized due to the aliphatic

chain between the two pyridine rings. In view of the bridging and blocking function of the 2,3,5,6-tetrabromoterephthalic acid (H_2TBTA) and 1,3-bis(4-pyridyl)propane (BPP) ligands, therefore, in this paper, we have been assembled two supramolecular polymers $[\text{M}(\text{BPP})_2(\text{TBTA})(\text{H}_2\text{O})_2]_n$ [$\text{M} = \text{Mn}(\mathbf{1}), \text{Co}(\mathbf{2})$] conformed Mn and Co metal ions with H_2TBTA and BPP under the same conditions, and the phase purity of the crystalline products was confirmed by powder X-ray diffraction (PXRD, see Figures S7 and S8). (See Supplementary Material available online at doi: 10.1155/2012/274508).

2. Experimental

2.1. Synthesis of Complexes **1** and **2**

2.1.1. $[\text{Mn}(\text{BPP})_2(\text{TBTA})(\text{H}_2\text{O})_2]_n$ (**1**). A mixture of H_2TBTA (10 mg, 0.02 mmol), BPP (10 mg, 0.05 mmol) in the distilled water (5 mL), and $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ (20 mg, 0.10 mmol) in methanol (5 mL) was placed in the straight glass tube. Then, the tube is sealed with a plastic wrap. Upon

slow evaporation of the solvents, colourless block single crystal suitable for X-ray diffraction was obtained after one week in 75% yield. Anal. Calcd (%) for $C_{34}H_{32}Br_4MnN_4O_6$: C, 42.222; H, 3.335; N, 5.793. Found (%): C, 42.19; H, 3.249; N, 5.80. IR (KBr, cm^{-1}): 3215 (s), 2938 (s), 1604 (vs), 1559 (s), 1394 (s), 1329 (vs), 1300 (m), 1214 (w), 1081 (w), 1006 (m), 811 (m), 699 (m), 556 (s), 512 (m).

2.1.2. $[Co(BPP)_2(TBTA)(H_2O)_2]_n$ (**2**). The same synthetic procedure as that for **1** was used except that the metal salt was replaced by $Co(NO_3)_2 \cdot 6H_2O$ (20 mg, 0.07 mmol), producing red block crystal after 10 days in 64% yield. Anal. Calc (%) for $C_{34}H_{32}Br_4CoN_4O_6$: C, 42.049; H, 3.321; N, 5.769. Found (%): C, 41.56; H, 3.296; N, 5.94. IR (KBr, cm^{-1}): 3393 (s), 2938 (s), 1612 (vs), 1559 (s), 1394 (s), 1319 (vs), 1305 (m), 1216 (w), 1073 (w), 1006 (w), 811 (m), 699 (w), 564 (m), 520 (w).

2.2. *Structure Determination*. Single-crystal X-ray diffraction data for the complexes were collected on a Bruker Apex II CCD diffractometer at room temperature with MoK α radiation ($\lambda = 0.71073 \text{ \AA}$). There was no evidence of crystal decay during data collection. All structures were solved by direct methods using the SHELXS program of the SHELXTL package and were refined by full-matrix least squares on F^2 using SHELX-97 [22, 23]. Nonhydrogen atoms were refined with anisotropic displacement parameters during the final cycles. H atoms were placed in geometrically idealized positions and were treated as riding on their parent atoms, distances of 0.93 $\text{\AA}$ (aromatic CH; sp^2) and 0.96 $\text{\AA}$ (CH_3), an O–H distance of 0.82 $\text{\AA}$ and with $U_{iso}(H)$ values of 1.2 $U_{eq}(C)$ and 1.5 $U_{eq}(O)$. The crystallographic data for complexes **1** and **2** are summarized in Table 1. The selected bond lengths and bond angles are listed in Table 2.

3. Results and Discussion

3.1. *Crystal Structure of Complexes 1 and 2*. The single-crystal X-ray diffraction analysis indicates that both of compounds **1** and **2** are triclinic crystal system with $P-1$ space group. The asymmetric unit of **1** and **2** consists of one metal ion, one coordinated BPP molecule, half of the $TBTA^{2-}$ ligand, and one water molecule (Figure 1). Due to the steric effect of the the $TBTA$ anionic, the two terminal carboxylate groups adopted a nearly perpendicular motif with the plane of Br_4 -substituted phenyl ring (the dihedral angle is 93.289° and 92.852° in **1** and **2**, resp.).

In the structure of compounds **1** and **2**, each metal center is coordinated by two carboxylate groups from different $TBTA$ ligands in the monodentate mode, two BPP molecules and two H_2O molecules (Figure S1). As a result, each $TBTA^{2-}$ ligand bridges two metal ions (the distances of $Mn \cdots Mn$ and $Co \cdots Co$ is 11.522 and 11.406 $\text{\AA}$), which generated a 1D chain (Figure 2). Two of the oxygen atoms from carboxylate groups of $TBTA^{2-}$ are naked and provide the hydrogen-bonding acceptors, therefore, each uncoordinated O2 of carboxylate atom tends to form an intramolecular H-bonding interaction with the coordinated H_2O molecule,

TABLE 1: Crystallographic data and structural refinement summary for complexes **1** and **2**.

Compound	1	2
Empirical formula	$C_{34}H_{32}Br_4MnN_4O_6$	$C_{34}H_{32}Br_4CoN_4O_6$
Formula weight	967.21	969.19
T (K)	296.15	296.15
Wavelength ($\text{\AA}$)	0.71073	0.71073
Crystal system	Triclinic	Triclinic
Space group	$P-1$	$P-1$
a ($\text{\AA}$)	9.4671 (11)	9.4522 (8)
b ($\text{\AA}$)	9.6578 (14)	9.6130 (9)
c ($\text{\AA}$)	11.5218 (14)	13.2728 (12)
α ($^\circ$)	77.576 (2)	105.3150 (10)
β ($^\circ$)	84.697 (2)	106.8240 (10)
γ ($^\circ$)	60.649 (2)	118.8890 (10)
V ($\text{\AA}^3$)	896.6 (2)	885.58 (14)
Z	1	1
D_{calc} (mg/mm^3)	1.733	1.817
μ (mm^{-1})	4.872	5.047
$F(000)$	446	477
Crystal size (mm)	$0.19 \times 0.13 \times 0.12$	$0.23 \times 0.17 \times 0.15$
2θ range for data collection ($^\circ$)	3.62–59.62	3.66–56.74
Index ranges	$-13 \leq h \leq 13,$	$-12 \leq h \leq 12,$
	$-13 \leq k \leq 13,$	$-12 \leq k \leq 12,$
	$-16 \leq l \leq 16$	$-17 \leq l \leq 17$
Reflections collected	13629	12212
Data/restraints/parameters	5103/0/174	4404/0/223
GOF on F^2	0.982	1.008
R_1 indices [$I > 2\sigma(I)$]	0.0402	0.0340
wR_2 indices (all data)	0.0887	0.0797
Largest diff. peak/hole ($e\text{\AA}^{-3}$)	0.51/–0.52	0.50/–0.48

which may further stabilize 1D chain coordination motif. The details of the intramolecular H-bonding interaction are listed in Table 3.

In the structure of **1**, the adjacent 1D chains linked by $C(12)–H(12B) \cdots O(2)^c$ ($c: x, y-1, z$) bonds between the BPP ligands and carboxylates afford a 2D layer (see Figure 3). These 2D patterns are overlapped and further extended by $C(15)–H(15) \cdots O(2)^b$ ($b: 1-x, 1-y, 1-z$) H-bonding between the BPP ligands and carboxylates to construct a 3D supramolecular architecture (Figure S2). However, due to the aliphatic chain between the two pyridine rings, the BPP ligand is flexible and can adopt different conformations characterized, resulting in different distances between the nitrogen atoms and different H-bonding donors and acceptors in the polymers **1** and **2**. Therefore, in the structure

TABLE 2: Selected bond lengths (Å) and bond angles (°) for compounds **1** and **2**.

1					
Bond	Dist.	Bond	Dist.	Bond	Dist.
Mn(1)–O(1)a	2.144(18)	Mn(1)–O(1)	2.1449(18)	Mn(1)–O(3)a	2.1926(8)
Mn(1)–O(3)	2.1926(8)	Mn(1)–N(1)a	2.3087	Mn(1)–N(1)	2.3087
Angle	(°)	Angle	(°)	Angle	(°)
O(1)–Mn(1)–O(1)a	180.0	O(1)–Mn(1)–O(3)	93.13(6)	O(1)a–Mn(1)–O(3)	86.87(6)
O(1)–Mn(1)–O(3)a	86.87(6)	O(1)a–Mn(1)–O(3)a	93.13(6)	O(1)–Mn(1)–N(1)	88.04(6)
O(1)a–Mn(1)–N(1)	91.96(6)	O(1)a–Mn(1)–N(1)a	88.04(7)	O(1)–Mn(1)–N(1)a	91.96(7)
O(3)a–Mn(1)–O(3)	180.0	O(3)–Mn(1)–N(1)	90.78(3)	O(3)a–Mn(1)–N(1)	89.22(3)
O(3)–Mn(1)–N(1)a	89.22(3)	O(3)a–Mn(1)–N(1)a	90.78(3)	N(1)a–Mn(1)–N(1)	180.00(6)
2					
Bond	Dist.	Bond	Dist.	Bond	Dist.
Co(1)–O(3)b	2.120(3)	Co(1)–O(3)	2.120(3)	Co(1)–O(1)b	2.078(2)
Co(1)–O(1)	2.078(2)	Co(1)–N(1)	2.186(4)	Co(1)–N(1)b	2.186(4)
Angle	(°)	Angle	(°)	Angle	(°)
O(3)b–Co(1)–O(3)	180.00(16)	O(3)–Co(1)–N(1)b	90.68(13)	O(3)–Co(1)–N(1)	89.32(13)
O(3)b–Co(1)–N(1)	90.68(13)	O(3)b–Co(1)–N(1)b	89.32(13)	O(2)b–Co(1)–O(3)b	89.24(11)
O(1)–Co(1)–O(3)b	90.76(11)	O(1)b–Co(1)–O(3)b	90.76(11)	O(1)–Co(1)–O(3)b	89.24(11)
O(1)b–Co(1)–O(1)	180.00(18)	O(1)–Co(1)–N(1)	88.26(12)	O(1)b–Co(1)–N(1)b	88.26(12)
O(1)–Co(1)–N(1)b	91.74(12)	O(1)b–Co(1)–N(1)	91.74(12)	N(1)b–Co(1)–N(1)	180.000(1)

Symmetry transformation: a: $-x, z - y, -z$; b: $1 - x, -y, -z$. FIGURE 1: The asymmetric unit of **1** and **2** [M = Mn(**1**), Co(**2**)].

FIGURE 2: A view of the 1D polymeric chain motif (the BPP ligands are omitted for clarity and the H-bonding interactions are represented as red-dashed lines).

TABLE 3: Hydrogen-bond lengths (Å) and bond angles (°) for compounds **1** and **2**.

D–H···A	d(D–H)	d(H···A)	d(D···A)	∠DHA
1				
O(3)–H(3A)···O(2) ^a	0.852	2.669	2.678	81.38
C(15)–H(15)···O(2) ^b	0.930	2.604	3.337	136.10
C(12)–H(12B)···O(2) ^c	0.970	2.839	3.752	157.02
2				
O(3)–H(3A)···O(2)	0.850	3.023	2.675	56.95
O(3)–H(3B)···N(2) ^d	0.850	2.028	2.774	145.90
C(12)–H(12A)···O(2) ^e	0.970	2.805	3.729	159.52

Symmetry codes: (a) $-x, 2-y, -z$; (b) $1-x, 1-y, 1-z$; (c) $x, y-1, z$; (d) $x, y, z-1$; (e) $x-1, y-1, z$.

of **2**, the supramolecular architecture is linked by O(3)–H(3B)···N(2)^d (d: $x, y, z-1$) and C(12)–H(12A)···O(2)^e (e: $x-1, y-1, z$) intermolecular H-bonding interaction (Figure 4). The details of the intermolecular H-bonding interaction are listed in Table 3.

3.2. Infrared Spectra. In the IR spectra of **1** and **2** (Figure S3, Figure S4), the broad peaks that centered at ca. 3300 cm^{-1} indicate the O–H characteristic stretching vibrations of water. The IR spectra of the complexes also exhibit two characteristic strong bands of carboxylate: ν_{vs} (COO^-) (ca. 1604 cm^{-1} for **1** and ca. 1612 cm^{-1} for **2**) and ν_{s} (COO^-) (ca. 1394 cm^{-1} for **1** and **2**) according to the literature [24]. The coordination modes of carboxylate groups are reflected in the separation distance between ν_{vs} (COO^-) and ν_{s} (COO^-) [25]. That is, bidentate carboxylate groups show a $\Delta\nu(\nu_{\text{vs}}(\text{COO}^-) - \nu_{\text{s}}(\text{COO}^-)) < 200\text{ cm}^{-1}$, whereas unidentate carboxylate groups show a $\Delta\nu(\nu_{\text{vs}}(\text{COO}^-) - \nu_{\text{s}}(\text{COO}^-)) > 200\text{ cm}^{-1}$. Herein, the IR spectra of **1** and **2** indicate a unidentate bonding mode for carboxylate groups of the H_2TBTA ligand (the separation distance is 210 and 218 cm^{-1}), which is in agreement with their crystal structures. The absence of characteristic bands at $1730\text{--}1690\text{ cm}^{-1}$ for carboxyl suggests the complete deprotonation of the H_2TBTA ligand in all complexes. In addition, IR spectrum of BPP ligand presents very characteristic bands at ca. 1560 cm^{-1} corresponding to $-\text{N}=\text{C}-$ stretching vibration mode.

FIGURE 3: 2D layer of **1** constructed by intermolecular hydrogen bonds (red-dashed lines), in which the water molecules are omitted for clarity.

3.3. Thermogravimetric Analyses. The thermal stabilities of the compounds **1** and **2** are investigated by thermogravimetric analysis (TGA) technique, and their TGA curves are in Figures S5 and S6. The TG curve of **1** shows the initial weight loss of 3.70% at around 146°C , followed by a second weight loss of 41.37% at 239°C (total weight loss of 45.01%). These correspond to loss of two coordinated water molecules (calc.: 3.73%) and two BPP molecules (calc.: 40.99%), for a total theoretical weight loss of 44.72% . The final product which is a manganese oxide, MnO (observed 7.2% , calc. 7.33%) obtains at 662°C . Anal. Calc (%) for MnO : O, 22.55 . Found (%): O, 22.62 . Compared with the calculated pattern (Figure S7(a)), the PXRD pattern of the dehydrated complexes **1** (Figure S7(c)) has a serious broadening of the peaks occurring. PXRD showed that the original structure was collapsed upon removal of the solvent molecules.

FIGURE 4: The intermolecular H-bonding interaction of **2** (red dashed lines).

For complex **2**, the first weight loss of 3.16% from 139 to 163°C due to the removal of coordinated water molecules (calc.: 3.71%). With temperature raised, complexes **2** starts to decompose with a sharp weight loss. Upon further heating to 585°C, no weight loss is observed and the resulting residue is CoO (observed 8.09%, calc. 7.73%). Anal. Calc (%) for CoO: O, 21.35. Found (%): O, 21.52. The PXRD pattern of the dehydrated complexes **2** (Figure S8(c)) matched the calculated pattern (Figure S8(a)), with only a slight broadening of the peaks occurring. PXRD confirmed that the structure is still maintained upon removal of the solvent molecules. The PXRD pattern of the sample (Figure S8(d)), prepared at 139°C for 20 min and then soaked in the solvent for 12 h, revealed that the structure did not readsorb the solvent molecules.

4. Conclusion

In summary, two novel 1D chain supramolecular polymers constructed with Mn^{2+} and Co^{2+} metal ions, 2,3,5,6-tetrabromoterephthalic acid (H_2TBTA) and 1,3-bis(4-pyridyl) propane (BPP) ligands have been synthesized and characterized. Both the TBTA^{2-} adopts monodentate coordination mode and the BPP as blocking ligand play important roles in the formation of the 1D chain frameworks. The supramolecular architectures of **1** and **2** are assembled via the intermolecular interactions like $\text{O}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{N}$ hydrogen bonds.

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