Assembly of three coordination polymers from glycolate ligands: syntheses, crystal structures, and thermal properties

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Received: 17 January 2013/Accepted: 18 March 2013/Published online: 29 March 2013 © Springer Science+Business Media Dordrecht 2013

Abstract Three transition metal-organic polymers, $[Cu(\eta^2-O_2CCH_2OH)(\mu_2-O_2CCH_2OH)(H_2O)]_n$ (1), $[Cu(\mu_2-O_2CCH_2OH)_2]_n$ (2), and $[Cd(\mu_2-O_2CCH_2OH)_2]_n$ (3) constructed from the rigid glycolate (HO_2CCH_2OH) ligand have been isolated under hydro(solvo)thermal conditions and structurally characterized by single-crystal X-ray diffraction. Polymer 1 presents a one-dimensional zigzag chain, while polymer 2 has a two-dimensional sheet structure. Polymer 3 exhibits a similar sheet structure as 2; however, the coordination modes of the ligands in complexes 1 and 2 are different. The three-dimensional solidstate supramolecular structures of the three polymers involve intermolecular hydrogen bonds. The thermal properties of the polymers have also been investigated.

Introduction

In the last two decades, the design and construction of various coordination polymers have attracted much attention due to their intriguing architectures and fascinating topologies [1–4], as well as multiple applications in gas separation and storage [5–8], chemical and biological sensing [9], ion exchange [10, 11], chirality, magnetism,

catalysis [12–15], and fluorescent materials [16, 17]. For the preparation of coordination polymers, two kinds of well-established methods (hydrothermal and solvothermal methods) have been adopted in order to carry out these reactions under high temperatures which favor formation of crystalline products suitable for X-ray studies. Although a great number of coordination polymers have been prepared by hydro(solvo)thermal synthesis methods [18–21], the factors governing the reactions and formation of the products are complicated. We still cannot approach a level of design control whereby a particular type of structure can be predictably obtained. Therefore, many more coordination polymers need to be prepared under hydro(solvo) thermal conditions.

It is well known that a coordination polymer is composed of two major components: a metal center or cluster of metal centers and an organic ligand called a linker. The choice of both metal and linker has significant effects on the structure and properties of the coordination polymer. The metal's coordination preference influences the size and shape of voids by dictating how many ligands can bind to the metal and in which orientations. Depending on the size of the ligands, inorganic connecting points, and network connectivity, the structures of such coordination polymers can be readily tuned to afford open channels and pores with dimensions of several angstroms to several nanometers.

In this paper, we investigated the reactions of a simple organic glycolate ligand with transition metals, namely Cu(II) and Cd(II), under hydro(solvo)thermal conditions. In this way, we obtained three interesting coordination polymers, $[Cu(\eta^2-O_2CCH_2OH)(\mu_2-COOCH_2OH)(H_2O)]_n$ (1), $[Cu(\mu_2-O_2CCH_2OH)_2]_n$ (2), and $[Cd(\mu_2-O_2CCH_2OH)_2]_n$ (3). In these polymers, the glycolate ligand shows various coordination modes and strong coordination ability. The thermal properties of the complexes have been investigated.

Electronic supplementary material The online version of this article (doi:10.1007/s11243-013-9717-5) contains supplementary material, which is available to authorized users.

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Experimental

Materials and methods

All chemicals were of reagent grade as obtained from commercial sources and used without further purification. IR Spectra were recorded on a Nicolet NEXUS 470-FTIR spectrophotometer as KBr pellets in the 400–4,000 cm⁻¹ region. Elemental analyses (C, H, and N) were obtained out on a FLASH EA1112 Elemental Analyzer. TGA measurements were made by heating the crystalline samples from 20 to 850 °C at a rate of 10 °C·min⁻¹ in air on a Netzsch STA 409PC differential thermal analyzer.

Synthesis of complex 1

A mixture of ethanol/water (7/1 mL) containing sodium glycolate (9.8 mg, 0.1 mmol) and Cu(NO₃)₂·3H₂O (24.2 mg, 0.1 mmol) was placed in a 25 mL Teflon-lined autoclave. The pH was adjusted by addition of one drop of Et₃N to keep the mixture weakly basic. The mixture was heated for 72 h at 160 °C under autogenous pressure, then allowed to cool to room temperature at a rate of 10 °C/h. Blue stick-shaped crystals of **1** were collected in 47 % yield (based on Cu), washed with deionized water, and dried in air. Crystals of **1** are stable in air. Anal. Calcd for C₄H₈O₇Cu: C, 20.7; H, 3.5 %. Found: C, 21.0; H, 3.8 %. IR (cm⁻¹, KBr): 3334 (s), 3265 (s), 3161 (m), 2924 (w),

Table 1 Crystallographic data for polymers 1-3

1605 (w), 1388 (s), 1316 (s), 1120 (m), 1036 (m), 920 (w), 668 (w), 557 (w).

Synthesis of complex 2

A mixture of ethanol/water (7/1 mL) containing sodium glycolate (9.8 mg, 0.1 mmol) and CuCl₂·2H₂O (17.0 mg, 0.1 mmol) was placed in a 25 mL Teflon-lined autoclave. The pH was adjusted by addition of one drop of Et₃N to keep the mixture weakly basic. The mixture was heated for 96 h at 120 °C under autogenous pressure, then allowed to cool to room temperature at a rate of 10 °C/h. Dark blue stick-shaped crystals of **2** were collected in 62 % yield (based on Cu), washed with deionized water, and dried in air. Crystals of **2** are unstable in air and turn blue after 3 days. Anal. Calcd for C₄H₆O₆Cu: C, 22.5; H, 2.8 %. Found: C, 22.7; H, 2.6 %. IR (cm⁻¹, KBr): 3,257 (s), 2944 (w), 1,615 (s), 1,390 (m), 1,336 (m), 1,145 (m), 1,059 (w), 905 (w), 743 (w), 646 (w).

Synthesis of complex 3

Complex **3** was prepared in a manner analogous to the method for complex **1** except that $Cd(CH_3COO)_2 \cdot 3H_2O$ was used instead of $Cu(NO_3)_2 \cdot 3H_2O$. Light yellow cube-shaped crystals of **3** were collected in 70 % yield (based on Cd), washed with deionized water, and dried in air. Crystals of **3** are stable in air. Anal. Calcd for $C_4H_6O_6Cd$:

Compound	1	2	3
Formula	C ₄ H ₈ O ₇ Cu	C ₄ H ₆ O ₆ Cu	C ₄ H ₆ O ₆ Cd
Formula weight	231.64	213.63	262.49
Crystal system	Orthorhombic	Monoclinic	Monoclinic
Crystal size (mm ³)	$0.20\times0.20\times0.18$	$0.21\times0.20\times0.19$	$0.20\times0.20\times0.20$
Space group	<i>P2(1)2(1)2(1)</i>	P2(1)/n	P2(1)/c
a (Å)	5.2248(5)	9.427(3)	6.8211(5)
<i>b</i> (Å)	10.8413(9)	5.1197(13)	9.3258(7)
<i>c</i> (Å)	13.5133(12)	13.911(4)	10.9037(8)
α (°)	90.00	90.00	90.00
β (°)	90.00	107.362(3)	93.0020(10)
γ (°)	90.00	90.00	90.00
$V(\text{\AA}^3)$	765.44(12)	640.8(3)	692.66(9)
$Dc (Mg m^{-3})$	2.010	2.214	2.517
Ζ	4	4	4
$\mu (\text{mm}^{-1})$	2.852	3.386	3.133
Reflections collected/unique	$6,531/1,559 \ [R(int) = 0.0475]$	$3,014/1,122 \ [R(int) = 0.0224]$	4,037/1,580 [R(int) = 0.0177]
Data/restraints/parameters	1,559/2/125	1,122/0/108	1,580/2/108
R indices (all data)	$R_1 = 0.0414, wR_2 = 0.0830$	$R_1 = 0.0361, wR_2 = 0.1035$	$R_1 = 0.0265, wR_2 = 0.0660$
GOF on F^2	0.761	1.113	1.229
$\Delta \rho_{\rm min}$ and $\Delta \rho_{\rm max}$ (e Å ⁻³)	-0.310 and 0.358	-0.552 and 0.644	-0.805 and 0.751

C, 18.3; H, 2.3 %. Found: C, 18.0; H, 2.1 %. IR (cm⁻¹, KBr): 3326 (s), 2,927 (m), 1,611 (s), 1,417 (s), 1,336 (s), 1,174. (w), 1,032 (s), 909 (m), 720 (m), 594 (m).

X-ray crystallography

Crystal data and experimental details for polymers 1–3 are given in Table 1. All measurements were made on a Bruker smart APEXII CCD diffractometer with a graphite-monochromated imaging plate area detector and Mo- $K\alpha$ radiation ($\lambda = 0.71073$ Å). Suitable single crystals were selected and mounted on a glass fiber. All data were collected at room temperature using the ω -2 θ scan technique and corrected for Lorenz-polarization effects. A correction for secondary extinction was also applied. The three structures were solved by direct methods and expanded using the Fourier

Table 2 Selected bond	l lengths (Å)) and angles	(deg) for 1-3
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1			
Cu(1)–O(5)	1.951(3)	Cu(1)–O(4)	1.958(3)
Cu(1)–O(3)	1.993(4)	Cu(1)–O(6)	2.005(4)
Cu(1)–O(1)	2.394(5)	O(5)-Cu(1)-O(4)	93.09(12)
O(5)-Cu(1)-O(3)	174.99(17)	O(4)–Cu(1)–O(3)	84.92(13)
O(5)-Cu(1)-O(6)	84.78(17)	O(4)-Cu(1)-O(6)	177.54(18)
O(3)-Cu(1)-O(6)	97.11(16)	O(5)-Cu(1)-O(1)	89.37(19)
O(4)-Cu(1)-O(1)	91.39(16)	O(3)–Cu(1)–O(1)	95.26(19)
O(6)-Cu(1)-O(1)	89.80(19)		
2			
Cu(1)–O(4)	1.943(3)	Cu(1)–O(7)	1.946(3)
Cu(1)–O(5)	1.989(3)	Cu(1)–O(8)	1.998(3)
O(4)-Cu(1)-O(7)	176.70(12)	O(4)-Cu(1)-O(5)	94.37(13)
O(7)-Cu(1)-O(5)	84.87(13)	O(4)–Cu(1)–O(8)	85.39(13)
O(7)–Cu(1)–O(8)	95.26(13)	O(5)–Cu(1)–O(8)	178.13(15)
3			
Cd(1)–O(7)	2.236(3)	Cd(1)-O(10)	2.287(2)
Cd(1)–O(4)	2.322(3)	Cd(1)–O(9)	2.372(3)
Cd(1)-O(10)#1	2.377(2)	Cd(1)–O(3)	2.383(3)
O(7)–Cd(1)– O(10)	96.05(10)	O(7)-Cd(1)-O(4)	148.31(10)
O(10)–Cd(1)– O(4)	89.22(10)	O(7)-Cd(1)-O(9)	100.41(11)
O(10)–Cd(1)– O(9)	141.10(10)	O(4)-Cd(1)-O(9)	94.64(12)
O(7)–Cd(1)– O(10)#1	126.83(9)	O(10)–Cd(1)– O(10)#1	71.93(10)
O(4)–Cd(1)– O(10)#1	84.51(10)	O(9)–Cd(1)– O(10)#1	69.96(10)
O(7)–Cd(1)–O(3)	78.78(9)	O(10)-Cd(1)-O(3)	121.59(10)
O(4)-Cd(1)-O(3)	71.95(10)	O(9)-Cd(1)-O(3)	96.20(11)
O(10)#1–Cd(1)– O(3)	151.84(9)		

Symmetry transformations used to generate equivalent atoms for **3:** #1: -x + 1, -y, -z + 2

technique. The correction for extinction was not adopted. The non-hydrogen atoms were refined with anisotropic thermal parameters. The hydrogen atoms on C were positioned geometrically and refined using a riding model. The hydrogen atoms on O were found at reasonable positions in the differential Fourier map and located there. All the hydrogen atoms were included in the final refinement. The final cycle of full-matrix least squares refinement was based on 6531 observed reflections and 125 variable parameters for 1; 3,014 observed reflections and 108 variable parameters for 2; 4,037 observed reflections and 108 variable parameters for 3. All calculations were performed using the SHELX-97 crystallographic software package [22]. Selected bond lengths and angles are listed in Table 2.

Results and discussion

Synthesis of the complexes

Organic ligands of low molecular weight are frequently utilized as ancillary ligands in the crystal engineering, especially for N-donor molecules. However, ligands containing both carboxylic and hydroxyl functional groups are superior owing to their potential chelating capacity [23-25]. In this work, we used glycolate as a single ligand to explore its ability to construct coordination compounds. Polymers 1-3 have been synthesized under hydro(solvo)thermal conditions with a solvent mixture solvent of ethanol and water (v/v 7:1). We chose this solvent composition based on the analyses of the construction of MOFs [25-29]. Solvent volume ratio of 7:1 (ethanol: water) for 1-3 has been used, and the suitable crystals for X-ray diffraction can be successfully isolated. The results indicate that formation of the frameworks of 1-3 is considerably influenced by the nature of the solvent.

In addition, two distinct structures, namely a onedimensional polymer **1** and two-dimensional polymer **2**, were obtained at 160 °C for 72 h and 120 °C for 96 h, respectively. It may be concluded that reactions at relatively low temperature with long reaction times favor this outcome.

Crystal structures of polymers 1 and 2

A single-crystal X-ray diffraction analysis reveals that polymer **1** belongs to the orthorhombic system with space group P2(1)2(1)2(1), and polymer **2** possesses a two-dimensional structure crystallizing in the monoclinic space group P2(1)/n.

As shown in Fig. 2a, the asymmetric unit contains one Cu center, two anionic $HOCH_2CO_2^-$ ligands, and one water ligand. The Cu²⁺ center exhibits a distorted octahedral CuO₆ coordination environment. The glycolate ligands



Fig. 1 Coordination modes of HOCH₂CO₂⁻ anions in polymers 1-3

Fig. 2 a View of coordination arrangement of Cu center in 1. b Ball-and-stick representation of the 1-D zigzag chain (H atoms omitted for clarity)



present two different coordination modes: $\mu_2 - \eta^1 : \eta^1 : \eta^1$ (Fig. 1a) and η^2 (Fig. 1b). The bonded oxygen atoms, O3, O4 and O5, O6 from two bidentate glycolate ligands, comprise an approximate plane through the Cu center (the dihedral angle between planes O3-Cu1-O4 and O5-Cu1-O6 is 4.7°) and the Cu–O distances range from 1.9 to 2.0 Å. The bond lengths of Cu1-O7B and Cu1-O1water are 2.7 and 2.4 Å, respectively, and the angle O1–Cu1–O7B is 171.5°. A zigzag chain results from the linkage of μ_2 -HOCH₂CO₂⁻ anions, and the uninvolved η^2 -HOCH₂CO₂⁻ anions occupy alternate sites beside the chain. As depicted in Fig. 2b, the distance between the intrachain neighboring metal centers is 5.3 Å and the Cu–Cu–Cu angle is 58.6°. Hydrogen-bonding interactions play an important role in the packing of the solid-state architecture (Fig. S1, Supplementary Data). The three-dimensional network is made up of right-handed and left-handed layers connected by intermolecular hydrogen bonds. The hydrogen bond distances are in the range of 2.7–3.5 Å and weak hydrogen-bonding interactions exist between two reverse chiral layers (O6–O1#1_{water} = 3.5 Å, symmetry code: #1 x + 1, y, z).

For polymer **2**, the Cu(II) center is coordinated by six oxygen atoms from anionic HOCH₂CO₂⁻ ligands, which all adopt one kind of coordination, μ_2 - η^1 : η^1 : η^1 (Fig. 1a). Compared with the [CuO₆] unit in polymer **1**, the octahedral unit of **2** shown in Fig. 3a is slightly distorted. The dihedral angle between planes O5–Cu1–O7 and O4–Cu1– O8 is 3.6°, and the four bond distances vary from 1.9 to 2.0 Å. The bond lengths of Cu–O along the vertical axis are 2.6 and 2.8 Å, corresponding to O10B–Cu1 and O9C– Cu1, respectively. The surrounding bond angles of O–Cu– O' (where O and O' are adjacent atoms) are in the range of 80.5–95.3°. As displayed in Fig. 3b, the chain is close to a straight line with the angle formed by Cu–Cu–Cu being



171.4°. The neighboring metal centers present two distances of 5.5 and 5.2 Å, which may account for the linkage of $HOCH_2CO_2^{-1}$ ligands that are alternatively placed on the two sides.

Distinct from the 1-D polymer 1, polymer 2 incorporates a two-dimensional sheet (Fig. 3c). The layer shown in the *ab* plane contains infinite rhombus grids (the lengths of the side being 5.5 or 5.2 Å), which results in a 3-D solid-state framework with the help of intermolecular hydrogen bonds. Three kinds of H-bonds exist in the polymer: O(5)– H(5)...O(7)#1, O(5)–H(5)...O(7)#2, O(8)–H(8)...O(10)#3 (symmetry codes: #1: x, y + 1, z; #2: -x + 1/2, y + 1/2, -z + 1/2; #3: x + 1/2, -y-1/2, z + 1/2), of which the last group, as shown in Fig. S2, bridges the neighboring R-handed and L-handed chiral layers to stabilize the crystal architecture (the donor...acceptor distance being 3.2 Å).

Polymer **2** is obtained by substituting one HOCH₂CO₂⁻ ligand for a water molecule from polymer **1**. Both the distortions may derive from the electron configuration of the Cu²⁺ cation, d⁹, due to the Jahn–Teller effect, and the octahedron is stretched [30, 31]. The asymmetrically coordinated oxygen atoms also contribute to the distortion in polymer **1**. Meanwhile, the substitution of the water ligand should favor the formation of a two-dimensional

layer, with only a coordination mode for the μ_2 -HOCH₂CO₂⁻ ligands. Both polymers are stabilized by hydrogen-bonding interactions and constructed from the R-handed and L-handed chiral layers.

Crystal structure of polymer 3

The single-crystal X-ray diffraction analysis reveals that polymer 3 crystallizes in the monoclinic system with space group P2(1)/c. Similar to polymer 2, the asymmetric unit contains two anionic ligands binding the Cd²⁺ center (Fig. 4a): two bidentate glycolate ligands and another two monodentate glycolates. Besides the mode $\mu_2 - \eta^1 : \eta^1 : \eta^1$ (Fig. 1a), one HOCH₂CO₂⁻ ligand displays another coordination mode, namely $\mu_2 - \eta^1 : \eta^2 : \eta^0$ (Fig. 1c). O4 and O9 come from two hydroxyl groups, with the bond lengths to Cd(II) being 2.3 and 2.4 Å, respectively, and the O4–Cd1– O9 angle is 94.6°. Another four carboxylato oxygen atoms complete the distorted octahedron, with the distances of Cd–O varying from 2.2 to 2.4 Å which are in the expected range [32]. As shown in Fig. 4b, c, the 2-D layer is constructed from infinite chains of $\mu_2 - \eta^1 : \eta^1 : \eta^1 - HOCH_2CO_2^{-1}$ ligands. The selected 1-D chain shown in Fig. 4b could be described as—{ $(HOCH_2CO_2^-)_{1a}$ { $[Cd_2(HOCH_2CO_2^-)_{1c}]_2$ }

Transition Met Chem (2013) 38:503-509

Fig. 4 a The coordination environment of Cd in **3**. **b** The straight chain $[Cd(HOCH_2CO_2)]_n$ (H atoms omitted for clarity). **c** 2-D layer extended in *bc* plane composed of infinite 1-D chains bridged by μ_2 -HOCH₂CO₂⁻ a

b

Fig. 5 The TG curves for polymers 1–3

the degradation process starts with a large weight loss on the TG curve in one step. The thermal decay is finished at 403 °C, leaving a solid residue of CuO (found 34.9 %, calculated 37.7 %). The polymer **3** is thermally stable up to 228 °C, then the whole framework begins to collapse upon

(HOCH₂CO₂⁻)_{1a})_{*n*}—, in which the subunits [Cd₂(HOCH₂-CO₂⁻)_{1c}]₂ arrange in horizontal and vertical axes, one after another, with the Cd...Cd distance being 3.8 Å. Figure S3 exhibits the solid-state three-dimensional architecture along the *b*-axis. The parallel layers are linked by intermolecular hydrogen bonds, comparable with those observed in polymers **1** and **2** (O(4)–H(4)...O(1)#4: 2.9 Å; O(9)–H(9)...O(1) #4: 3.2 Å symmetry code: #4 -x, -y, -z + 2).

Thermogravimetric analyses

Thermogravimetric analyses were carried out in order to investigate the thermal stabilities of polymers 1-3 within the range of 30–850 °C in air (Fig. 5). The TG curve of polymer 1 presents an initial weight loss of 8 % from 125 to 228 °C, which is ascribed to the release of one water ligand (calculated 7.8 %). Then, the decomposition of the glycolate ligands begins, and the remaining CuO residue accounts for 34.6 % (calculated 34.8 %). Polymers 2 and 3 show similar thermal behavior owing to their similar structures. Polymer 2 is thermally stable until 93 °C. Then,

further heating from 228 to 517 °C. Finally, a plateau region is observed from 517 to 700 °C. The white amorphous residue is identified as CdO (found 49.1 %, calculated 49.0 %).

Conclusions

In summary, three polymers 1-3 derived from the glycolate anion are reported here and exhibit one- or two-dimensional structures. The simple rigid glycolate ligand displays three distinct coordination modes, which confirms its versatile coordination ability. The hydrogen bonding observed in all three compounds plays an important part in the stabilization of their frameworks.

Supplementary materials

Crystallographic data for these structures reported in this paper in the form of CIF files have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication CCDC nos. 903006–903008 for complexes 1–3, respectively. Copies of these data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 IEZ, UK (Fax: +44 1223 336 033; E-mail: deposit@ccdc.cam.ac.uk).

Acknowledgments We gratefully acknowledge the financial support by the Research Program of Universities of Inner Mongolia Autonomous Region (NJ10245).

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