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Hydrothermal synthesis and characteristics of 3-D hydrated bismuth oxalate coordination polymers with open-channel structure

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Abstract

Two new 3-D porous bismuth coordination polymers, $(C_5NH_6)_2[Bi_2(H_2O)_2(C_2O_4)_4] \cdot 2H_2O$ **1** and $(NH_4)[Bi(C_2O_4)_2] \cdot 3H_2O$ **2**, have been hydrothermally synthesized and characterized by single-crystal X-ray diffraction. Compound **1** crystallizes in the monoclinic symmetry, $P2_1/c$ space group with $a = 10.378(2)$ Å, $b = 17.285(3)$ Å, $c = 16.563(5)$ Å, $\alpha = 90°$, $\beta = 119.66(2)°$, $\gamma = 90°$, $V = 2581.8(10)$ Å^3, $Z = 4$, $R_1 = 0.0355$ and w$R_2 = 0.0658$ for unique 4713 reflections $I > 2\sigma(I)$. Compound **2** crystallizes in the tetragonal symmetry, $I4_1/amd$ space group with $a = 11.7026(17)$ Å, $b = 11.7026(17)$ Å, $c = 9.2233(18)$ Å, $\alpha = 90°$, $\beta = 90°$, $\gamma = 90°$, $V = 1263.1(4)$ Å^3, $Z = 32$, $R_1 = 0.0208$ and w$R_2 = 0.0518$ for unique 359 reflections $I > 2\sigma(I)$. Compounds **1** and **2** are 3-D open-framework structures with a 6^6 uniform net, which consist of honeycomb-like layers connected to each other by oxalate units. While different guest molecules fill in their cavities of honeycomb-like layers, study of ultrasonic treatment on **2** indicates the replacement of NH_4^+ by K^+ on potassium ion exchange. Thermogravimetric analysis indicates that the open-channel frameworks are thermally stable up to 200 °C, and other characterizations are also described by elemental analysis, IR and ultraviolet–visible diffuse reflectionintegral spectrum (UV–Vis DRIS).
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1. Introduction

Currently, porous materials are widely regarded as promising materials for their potential applicability in various fields such as catalysis, separation, gas storage, sensing, magnetism, and ion exchange, etc. [1–7]. Therefore, there has been intense research effort on the synthesis of such materials and several metal-organic frameworks possessing zeolitic structures have been reported [8–11]. With the features of versatile bridging modes, carboxylates are often employed as building block to construct porous coordination polymers [12–16]. Oxalate is one of the simplest carboxylates; up to now a large number of metal

oxalates synthesized in the presence of structure-directing amines have been restricted to those of d-block transition metal and rare-earth-metal [17–25], and most of these oxalates with 3-D structure generally possess honeycomb layers cross-linked by oxalate units. For example, the yttrium oxalates $(C_6N_2H_{16})_{0.5}[Y(H_2O)(C_2O_4)_2] \cdot 2H_2O$ and $(C_5N_2H_{12})[Y(C_2O_4)_2]$ have 3-D structures. These oxalates possess honeycomb-like apertures layers, which are pillared by another oxalate unit to form 3-D structures with channels [26,27]. As compared to the reports of d-block transition metal and rare-earth-metal oxalates, bismuth-based oxalates are scarce to the best of our knowledge [8,28–31], resulting from the hydrolytic behavior of bismuth compounds even in strong acid solutions. However, the larger bismuth atoms can exhibit higher coordination and highly variable coordination numbers from 3 to 10, which provides unique opportunities for the discovery of new network topologies and unique physical properties [32], and the presence of lone

pairs on bismuth centers not only influences the coordination geometry, but also directs the structure to some extent [8,28]. In recent years, bismuth compounds have been widely applied in medical treatment, catalysts, luminescence, etc. [33–36], therefore bringing great interests in the investigation of bismuth oxalates.

In our previous studies, we reported three bismuth(III) coordination polymers, $(enH_2)_{1.5}[Bi_3(C_2O_4)_6(CO_2CONH CH_2CH_2NH_3)] \cdot 6.5H_2O$, $[NH(C_2H_5)_3][Bi_3(C_2O_4)_5]$ and $[NH(C_2H_5)_3][Bi_3(C_2O_4)_5]$, in which organic amine were located in the channels [28,29]. We attempted to use some larger amine ligands, such as pyridine or 1,10-phenanthroline to synthesize bismuth(III) coordination polymers with larger open-channel structure. As unexpected, we synthesized two new 3-D open-framework structures, $(C_5NH_6)_2 [Bi_2(H_2O)_2(C_2O_4)_4] \cdot 2H_2O$ **1** and $(NH_4)[Bi(C_2O_4)_2] \cdot 3H_2O$ **2**, with different guest molecules accommodated in their open-channel structures, and compound 2 has potential applicability in ion exchange.

2. Experimental

2.1. Reactants and methods

All reagents were used as purchased from commercial sources without further purification. The elemental analyses

of C, H, and N were performed with an Elementar Vario EL III elemental analyzer. The IR spectra were carried out on a Perkin-Elmer Spectrum 2000 spectrometer in the 4000–400 cm^{-1} region with a pressed KBr pellet. The UV–Vis DRIS spectrum were measured on a Perkin-Elmer Lambda 900 UV–Vis spectrometer. The TGA were performed on a Delta Series TGA7 instrument in N_2 atmosphere with heating rate of 10 °C/min from 35 to 400 °C.

2.2. Synthesis

2.2.1. Synthesis of $(C_5NH_6)_2[Bi_2(H_2O)_2(C_2O_4)_4] \cdot 2H_2O$ **1**

A mixture of $Bi(NO_3)_3 \cdot 5H_2O$ (0.25 g, 0.5 mmol), $H_2C_2O_4 \cdot 2H_2O$ (0.80 g, 5.00 mmol), pyridine (0.20 mL),

Table 1
Crystal data

Compound	1	2
Formula	$Bi_2C_{18}N_2O_{20}H_{20}$	$Bi_{0.125}C_{0.50}H_{1.25}N_{0.125}O_{1.375}$
Molecular mass	1002.32	57.14
Crystal system	Monoclinic	Tetragonal
Space group	$P2_1/c$	$I4_1/amd$
a (Å)	10.378(2)	11.7026(17)
b (Å)	17.285(3)	11.7026(17)
c (Å)	16.563(5)	9.2233(18)
α (°)	90	90
β (°)	119.66(2)	90
γ (°)	90	90
V (Å^3)	2581.8(10)	1263.1(4)
Z	4	32
$\rho_{calcd.}$ (g/cm^3)	2.579	2.388
μ (mm^{-1})	13.712	14.006
Crystal size (mm)	$0.25 \times 0.12 \times 0.10$	$0.24 \times 0.13 \times 0.13$
λ(MoKα) (Å)	0.71073	0.71073
Collected reflections	23889	5004
Independent reflections	5832 ($R_{int} = 0.0641$)	399 ($R_{int} = 0.0451$)
Observed reflections ($>2\sigma I$)	4713	359
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0355$ $wR_2 = 0.0658$	$R_1 = 0.0208$ $wR_2 = 0.0518$
R indices (all data)	$R_1 = 0.0503$ $wR_2 = 0.0701$	$R_1 = 0.0253$, $wR_2 = 0.0533$
Largest diff. map peak and hole (e Å^{-3})	1.316 and −1.173	0.742 and −0.462

$R_I = \sum||F_o|-|F_c||/\sum|F_o|$; $wR_2 = \{\sum[w(F_o^2-F_c^2)^2]/\sum[w(F_o^2)^2]\}$.
$w = 1/[\sigma^2(F_o^2)+(ap)^2+bp]$, where $P = (F_o^2+2F_c^2)/3$, where $a = 0.0219$ and $b = 5.9705$ for **1** and $a = 0.0270$ and $b = 5.3877$ for **2**.

Table 2
Selected bond lengths (Å) and angles (°) for compounds **1** and **2**

*Compound **1***[a]

Bi(1)–O(5)#1	2.440(4)	Bi(1)–O(9)#2	2.461(4)
Bi(1)–O(15)	2.481(5)	Bi(1)–O(3)#2	2.495(4)
Bi(1)–O(18)	2.495(5)	Bi(1)–O(8)#1	2.509(4)
Bi(1)–O(6)	2.510(6)	Bi(1)–O(12)	2.512(4)
Bi(1)–O(11)	2.569(5)	Bi(2)–O(10)	2.414(4)
Bi(2)–O(7)	2.454(4)	Bi(2)–O(14)	2.494(5)
Bi(2)–O(13)	2.495(6)	Bi(2)–O(2)#3	2.499(4)
Bi(2)–O(1)	2.499(4)	Bi(2)–O(4)	2.517(4)
Bi(2)–O(17)	2.518(5)	Bi(2)–O(16)	2.521(5)
O(5)#1–Bi(1)–O(8)#1	66.78(14)	O(18)–Bi(1)–O(12)	65.12(16)
O(9)#2–Bi(1)–O(3)#2	66.44(14)	O(15)–Bi(1)–O(11)	63.87(15)
O(15)–Bi(1)–O(12)	143.60(18)	O(15)–Bi(1)–O(11)	63.87(15)
O(9)#2–Bi(1)–O(11)	140.08(18)	O(9)#2–Bi(1)–O(8)#1	133.68(14)
O(18)–Bi(1)–O(8)#1	141.66(17)	O(5)#1–Bi(1)–O(6)	74.72(16)
O(3)#2–Bi(1)–O(12)	131.57(17)	O(8)#1–Bi(1)–O(11)	67.35(17)
O(5)#1–Bi(1)–O(9)#2	144.51(16)	O(5)#1–Bi(1)–O(15)	130.56(16)
O(9)#2–Bi(1)–O(15)	84.78(17)	O(5)#1–Bi(1)–O(3)#2	124.20(16)
O(9)#2–Bi(1)–O(3)#2	66.44(14)	O(15)–Bi(1)–O(3)#2	68.05(18)

*Compound **2***[b]

Bi(1)–O(2)#1	2.418(6)	Bi(1)–O(2)#2	2.418(6)
Bi(1)–O(2)#3	2.418(6)	Bi(1)–O(2)#4	2.418(6)
Bi(1)–O(1)#5	2.445(5)	Bi(1)–O(1)	2.445(5)
Bi(1)–O(1)#6	2.445(5)	Bi(1)–O(1)#7	2.445(5)
O(2)#1–Bi(1)–O(2)#2	92.41(5)	O(2)#1–Bi(1)–O(2)#3	156.3(3)
O(2)#2–Bi(1)–O(2)#3	92.41(5)	O(2)#1–Bi(1)—O(2)#4	92.41(5)
O(2)#2–Bi(1)–O(2)#4	156.3(3)	O(2)#3–Bi(1)–O(2)#4	92.41(5)
O(2)#1–Bi(1)–O(1)#5	66.74(18)	O(2)#2–Bi(1)–O(1)#5	80.34(11)
O(2)#3–Bi(1)–O(1)#5	136.93(18)	O(2)#4–Bi(1)–O(1)#5	80.34(11)
O(2)#1–Bi(1)–O(1)	80.34(11)	O(2)#2–Bi(1)–O(1)	66.74(18)
O(2)#3–Bi(1)–O(1)	80.34(11)	O(2)#4–Bi(1)–O(1)	136.93(18)
O(1)#5–Bi(1)–O(1)	132.02(15)	O(2)#1–Bi(1)–O(1)#6	136.93(18)
O(2)#2–Bi(1)–O(1)#6	80.34(11)	O(2)#3–Bi(1)–O(1)#6	66.74(18)
O(2)#4–Bi(1)–O(1)#6	80.34(11)	O(1)#5–Bi(1)–O(1)#6	70.2(2)
O(1)–Bi(1)–O(1)#6	132.02(15)	O(2)#1–Bi(1)–O(1)#7	80.34(11)
O(2)#2–Bi(1)–O(1)#7	136.93(18)	O(2)#3–Bi(1)–O(1)#7	80.34(11)
O(2)#4–Bi(1)–O(1)#7	66.74(18)	O(1)#5–Bi(1)–O(1)#	132.02(15)
O(1)–Bi(1)–O(1)#7	70.2(2)	O(1)#6–Bi(1)–O(1)#7	132.02(15)

[a]Symmetry transformations used to generate equivalent atoms:#1: $-x$, $y-1/2$, $-z+1/2$; #2: $+1$, y, z.

[b]Symmetry transformations used to generate equivalent atoms: #1: $-x+3/4$, $y+3/4$, $z+1/4$; #2: $-x+1$,$-y$,$-z+1$; #3: $x-1/4$, $-y+1/4$, $z+1/4$; #4: x, $y+1/2$, $-z+1$; #5: $x-1/4$, $-y+3/4$, $-z+5/4$; #6: $-x+3/4$, $y+1/4$, $-z+5/4$ #7: $-x+1$, $-y+1/2$, z.

Ni(CH$_3$COO)$_2$·4H$_2$O (0.2 g, 0.8 mmol) and distilled water (3.50 mL) was neutralized to pH 2.5 by adding drops of LiOH (1.0 M solution in water) under continuous stirring, and was heated at 110 °C for 60 h in a sealed 15 mL Teflon-lined stainless vessels. After slowly cooling to room temperature, the block colorless crystals of compound **1** were obtained, suitable for single-crystal X-ray diffraction, and filtered and washed with deionized water. The crystalline product is stable in air and insoluble in water. Yield: 18% based on Bi. Anal. Calc. for C$_{18}$H$_{20}$Bi$_2$N$_2$O$_{20}$: C, 21.57; H, 2.01; N, 2.79. Found: C, 20.98; H, 2.10 ; N, 2.82%.

2.2.2. Synthesis of (NH$_4$)[Bi(C$_2$O$_4$)$_2$]·3H$_2$O 2

The compound **2** was prepared similarly to **1** with the exception that pyridine (0.20 mL) was replaced by o-phenanthroline (0.02 g, 0.01 mmol) and the pH value was adjusted to 2.8 by adding drops of NH$_3$·H$_2$O. The block colorless crystals of compound **2** were filtered off and washed with deionized water. The crystalline product is a stable crystalline solid in air and can be kept in a dry atmosphere for extended periods of time. Yield: 25% based on Bi. Anal. Calc. for C$_4$H$_{10}$BiNO$_{11}$: C, 10.51; H, 2.20; N, 3.06. Found: C, 10.49; H, 2.24; N, 3.02%.

2.2.3. Ultrasonic treatment and ion exchange

The ion exchange of NH$_4^+$ with K$^+$ in synthetic (NH$_4$)[Bi(C$_2$O$_4$)$_2$]·3H$_2$O **2** single crystals was investigated under ultrasonic treatment. The ultrasonic treatment was conducted using saturated KCl solutions at room temperature for 72 h. After ion exchange, samples were filtered, washed, and air-dried at room temperature.

2.3. Crystallographic measurements and structure determination

The reflection intensities were collected on a Rigaku Weissenberg IP diffractometer with graphite-monochromated MoKα radiation ($\lambda = 0.71073$ Å) at 298 ± 2 K. Lp correction and a ψ empirical absorption correction were made for the intensity data. The structures of compounds **1** and **2** were solved by direct methods using SHELXS-97 [37] and refined by the full-matrix least-squares method on F^2 with anisotropic thermal parameters for all non-hydrogen atoms using SHELXL-97 program [38]. The remaining hydrogen atoms were generated geometrically and not refined. In compound **1**, due to the statistical disorder of C and N atoms positions of pyridine, it was difficult to detect and ascertain the positions of the hydrogen atoms. The hydrogen atoms were generated for satisfying the charge requirement of the framework. The crystal data are given in Table 1. Selected bond lengths and angles are shown in Table 2.

3. Results and discussion

3.1. Crystal structure of compounds 1 and 2

X-ray crystallographic analysis shows that compound 1 exhibits a novel 3-D microporous network, which is made up of [Bi$_2$(H$_2$O)$_2$(C$_2$O$_4$)$_4$]$^{2-}$, two protonated pyridine anions and two lattice water molecules. As shown in Fig. 1, there are two crystallographically unique Bi centers in the crystal structure, Bi(1) and Bi(2). Both Bi centers possess a distorted monocapped square antiprism geometry, which is completed by one oxygen atom from a water molecule with Bi–O bond lengths of 2.495(6)–2.510(6) Å,
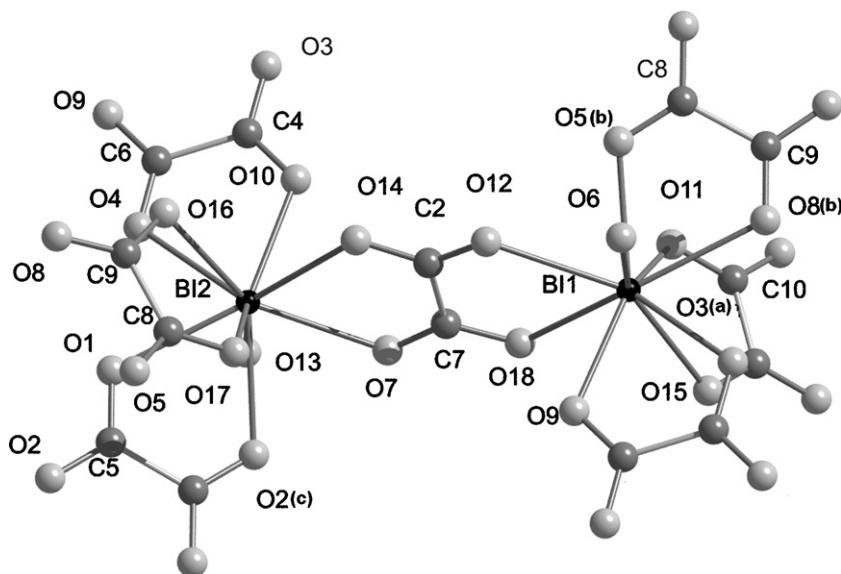

Fig. 1. Perspective view of the coordination environment of the Bi(III) ion in compound **1**. Symmetry codes for the generated atoms: (a) $1+x$, y, z; (b) $-x$, $-y$,$-z$; and (c) $-x$, $-0.5+y$, $0.5-z$. Thermal ellipsoids are drawn at the 10% probability level.
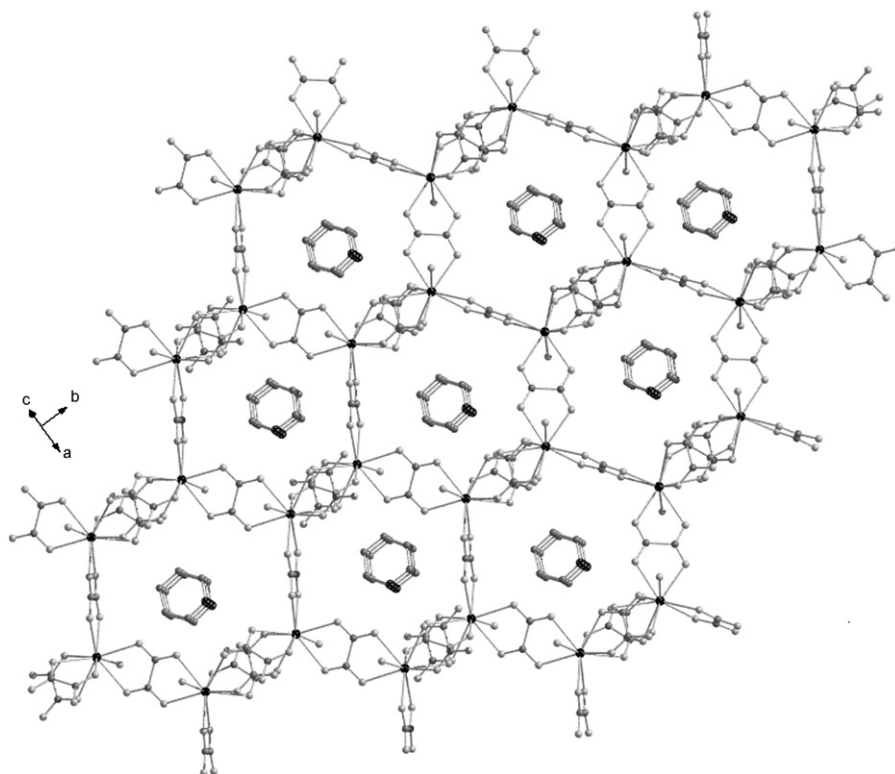
Fig. 2. A viewgraph of 2-D porous network containing cavities with dimensions of $6.6 \times 8.0 \,\text{Å}^2$.

and eight oxygen atoms from four oxalate ligands with Bi–O distances of 2.414(4)–2.569(5) Å. The O–Bi–O angles range from 64.23(15)° to 146.73(18)°. The two Bi centers are interconnected by the bridging oxalate ligands to form a 2-D honeycomb-like layer, as shown in Fig. 2, The layers, in turn, are connected by oxalate groups to give the 3-D structure with 1-D elliptical channels. The extraframework water molecules and protonated pyridine anion occupy the channels. The size of the pore is estimated to be $6.6 \times 8.0 \,\text{Å}^2$. In addition, the N–H⋯O and OW–H⋯O hydrogen bond (N⋯O distances and OW⋯O distances are in the range of 2.819–3.098 and 2.781–3.454 Å, respectively) effect enhances the stability of the compound **1** (Table 3).

Compared to **1**, a different coordination environment is found for the bismuth center in compound **2**, although the same ligands are used. As shown in Fig. 3, the Bi centers have a distorted dodecahedral environment of eight oxygen atoms arising from four oxalate anions. Bond distances around the Bi center are typical of other Bi-based coordination polymers containing oxalate ligands: Bi–O bonds distances in the range of 2.418(5)–2.443(5) Å, consistent with corresponding bond lengths found in K[Bi(C$_2$O$_4$)$_2$]·5H$_2$O [19] and Bi(NH$_4$)(C$_2$O$_4$)$_2$·xH$_2$O [20]. Similar to **1**, each oxalate ligand in **2** acting as bidentate bridging ligand connects two bismuth atoms to form 2-D layers, as shown in Figs. 4 and 5; then the layers are also connected through oxalate ligands, therefore resulting in the 3-D structure, as shown in Fig. 6. The channel has a

Table 3
Selected hydrogen bond lengths (Å) and angles (°) in compound **1**[a]

D–H⋯A	D(D–H)	d(H⋯A)	d(D⋯A)	<DHA
N1–H1⋯O3#1	0.880	2.013	2.819	151.69
N1–H1⋯O8	0.880	2.447	3.098	131.22
N2–H2⋯O17#2	0.880	2.100	2.961	166.03
OW1–HW1A⋯O14	0.850	2.045	2.831	153.46
OW1–HW1B⋯O13#3	0.850	2.185	2.685	117.48
OW1–HW1B⋯O4#4	0.850	2.619	3.454	167.33
OW2–HW2A⋯O16	0.850	2.127	2.912	153.23
OW2–HW2B⋯O6#5	0.850	1.967	2.781	160.03

[a]Symmetry transformations used to generate equivalent atoms: #1 –x-1, y+1/2, −z+1/2; #2 −x, y−1/2, −z+1/2; #3 x, −y−1/2, z−1/2; #4 x, −y−1/2, z−1/2; #5 x−1, y, z.

$5 \times 10 \,\text{Å}^2$ pore. The uncoordinated water molecules and protonated ammonium molecules were located in the channels. In these cavities the ammonium ions and water molecules are bound by weak hydrogen bonds (N⋯O distances are in the range of 3.1446–3.32 Å) (Table 4).

It is found that the use of hydrothermal techniques and the involvement of nickel acetate are very essential in the formation of the title compounds. In the absence of nickel acetate, only amorphous compounds are obtained. It is possible that the nickel acetate attenuate the sensitivity of bismuth-based salts to water [39]. In addition, the template effect of organic amines also plays an important role in the construction of the title compounds. In the absence of organic amines, only amorphous compounds are obtained too.

Potassium ion exchange cause discernible changes to the infrared spectra, as shown in Fig. 7. After potassium exchange, the peak of N–H at about 3161 cm^{-1} is disappeared, indicating the replacement of NH_4^+ by K^+ on potassium ion exchange. Compound 2 has potential applicability in ion exchange.

3.2. Spectroscopy

The IR spectra of **1** and **2** exhibit strong and broad bands in the range of 3610–3280 cm^{-1} assignable to O–H stretching vibrations of lattice and/or coordinated water molecules [40]. The peak at about 3063 cm^{-1} is attributed
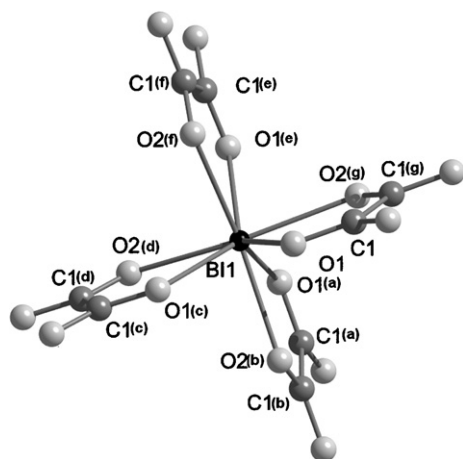
Fig. 3. Perspective view of the coordination environment of the Bi(III) ion in compound **2**. Symmetry codes for the generated atoms: (a) 0.75−x, 0.75−y, 1.25−z; (b) 0.75−x, 0.75+y, 0.25+z; (c) 1−x, 0.5−y, z; (d) 1−x, 0.5+y, 1−z; (e) −0.25+x, 0.25+y, 1.25−z; (f) −0.25+x, 0.25−y, 0.25+z; and (g) x, −y, 1−z. Thermal ellipsoids are drawn at the 10% probability level.

to N–H of pyridine molecules asymmetric stretching vibrations in the spectra of **1**. The absence of the expected absorption at 1690–1730 cm^{-1} for the protonated oxalate groups illustrates the complete deprotonation of the ligand in the reaction with bismuth. The absorptions of the coordinated oxalate group occur at 1583, 1487, 1299, and 790 cm^{-1} for compound **1**, 1600, 1402, 1289, and 790 cm^{-1} for compound **2**, respectively. It shows that the oxalate anion behaves as a bichelating oxygen-donor ligand [41]. Moreover, the absorption at 1299 cm^{-1} can also be attributed to the C–N stretching vibration. Bands in the region 713–1060 cm^{-1} can be assigned to O–C=O in plane bending vibrations. The peak at 493 cm^{-1} is ascribed to Bi–O stretching vibrations and O–C=O out of plane bending vibrations [29].

In UV–Vis spectrum of the title compounds, it shows more intense bands centered at 247 and 285 nm for **1**, 251 and 292 nm for **2**, which can be assigned to π–π* transfer transition of ligand and L–M transfer transition, respectively [42].

3.3. Thermal analysis

Thermogravimetric analyses for these two compounds have been determined. For **1**, the first weight loss is 3.7% (calc. 3.4%) in the range 98–129 °C, corresponding to the removal of lattice water molecules. The second weight loss is 54.2% (calc. 54.9%) in the temperature range of 230–297 °C, corresponding to the release of pyridine, coordinated water molecules, and oxalate molecules. For **2**, the weight loss of 12.3% (calc. 11.8%) at 61–120 °C is consistent with the decomposition of solvent water molecule. The weight loss of 41.8% (calc. 42.5%) at 250–293 °C arises from the removal of oxalate molecules and ammonium molecules.
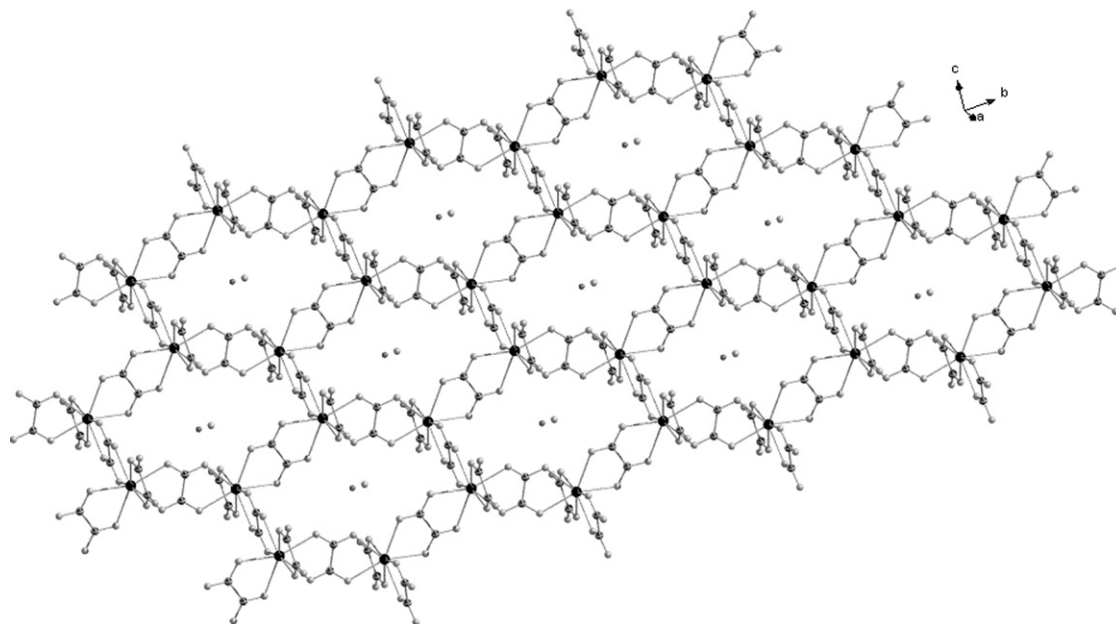

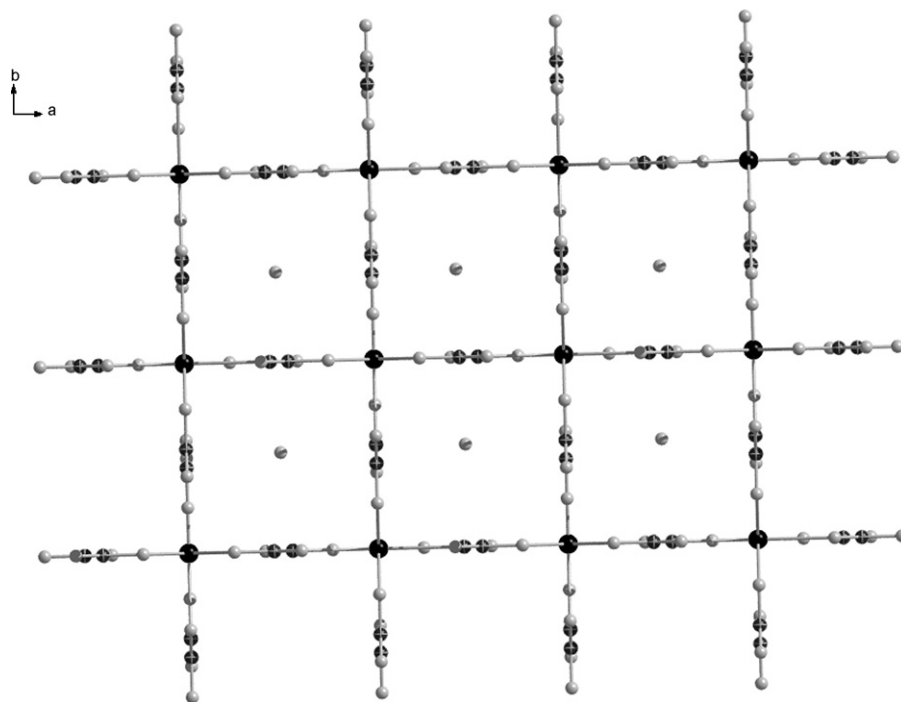Fig. 4. View of 2-D layer structure with of 6+6 membered aperture.

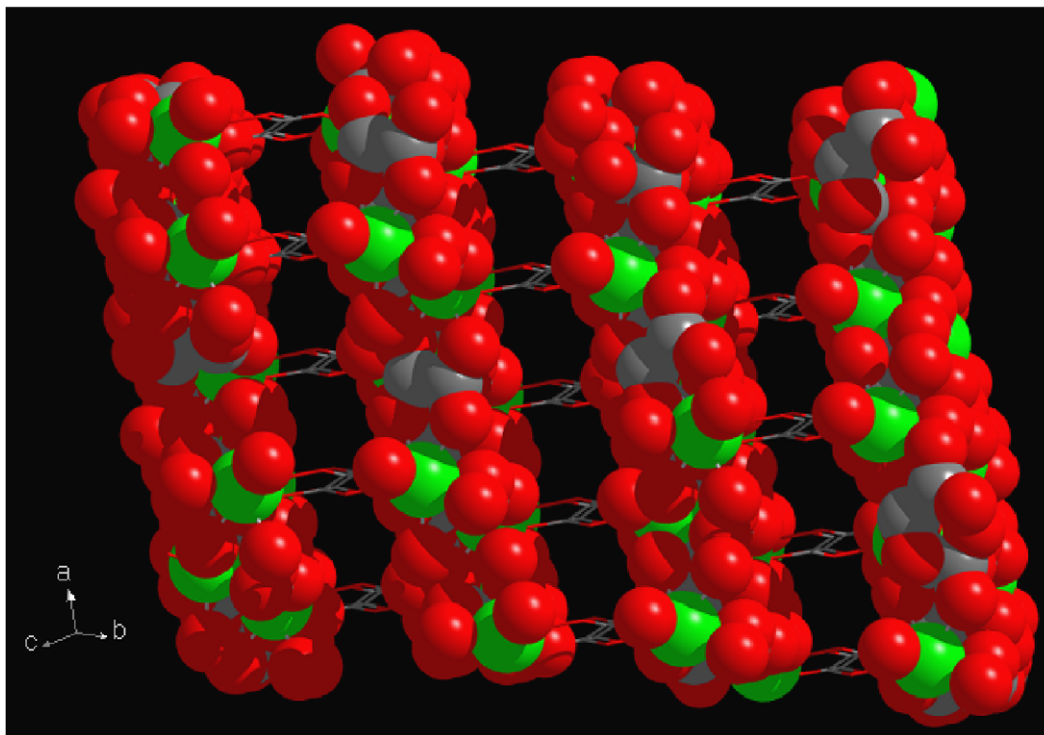Fig. 5. Side viewgraph of the 2-D porous layer.

Fig. 6. View of 3-D structure with channels (hydrogen atoms are omitted for clarity).

4. Conclusion

Two new 3-D oxalate-based bismuth coordination polymers have been prepared in the presence of organic amines and nickel acetate by hydrothermal synthesis. Compounds 1 and 2 possess honeycomb-like layers pillared by oxalate units to give a 3-D open-framework structure with channels. While different guest molecules are located

Table 4
Selected hydrogen bond lengths (Å) and angles (°) in compound **2**[a]

D–H⋯A	d(D–H)	d(H⋯A)	d(D⋯A)	<DHA
N1–H1⋯OW2_$1	0.90	2.25	3.1446	177
N1–H1⋯OW1_$4	0.90	2.80	3.32	118

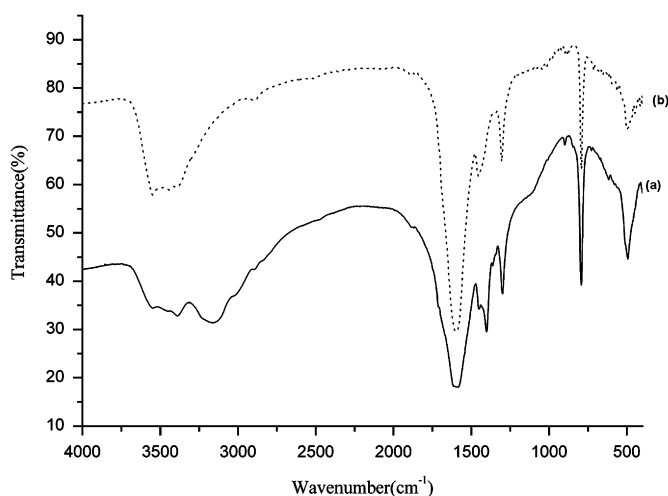[a]Symmetry transformations used to generate equivalent atoms: $1 $-x+5/4$, $y+1/4$, $z-1/4$, $4 -x+1$, $-y+1/2$, z.

Fig. 7. Infrared spectra of (a) $(NH_4)[Bi(C_2O_4)_2] \cdot 3H_2O$ (b) K^+-exchanged.

in their open-channel frameworks. To make further realization on the zeolitic properties of materials, our future work will target the synthesis of more versatile 3-D porous bismuth-based compounds introducing other carboxylate ligands such as malonate, succinate, fumarate, glutarate, adipate, etc. under similar conditions. Compound 2 has potential applicability in ion exchange.

Acknowledgments

Appendix A. Supplementary materials

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jssc.2008. 02.018.

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