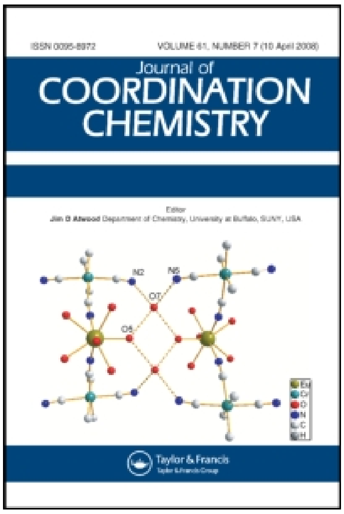

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Solvothermal syntheses, structures, and luminescence of two heterometallic metal-organic frameworks constructed from <i>m</i>-BDC (<i>m</i>-BDC = 1,3-benzene-dicarboxylate)

Guang-Bo Che[a]; Chun-Bo Liu; Liang Wang; Yun-Cheng Cui
[a] Department of Chemistry, Jilin Normal University, Siping

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Solvothermal syntheses, structures, and luminescence of two heterometallic metal-organic frameworks constructed from *m*-BDC (*m*-BDC = 1,3-benzene-dicarboxylate)

GUANG-BO CHE*, CHUN-BO LIU, LIANG WANG and YUN-CHENG CUI

Department of Chemistry, Jilin Normal University, Siping 136000

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The reactions of m-H_2BDC, NaOH with $Cd(NO_3)_2 \cdot 4H_2O$ and $Co(NO_3)_2 \cdot 4H_2O$ (m-BDC = 1,3-benzene-dicarboxylate) in DMF gave two isostructural three-dimensional (3D) heterometallic coordination polymers: $[CdNa(m\text{-}BDC)_2] \cdot [NH_2(CH_3)_2]$ (**1**) and $[CoNa(m\text{-}BDC)_2] \cdot [NH_2(CH_3)_2]$ (**2**), respectively. The compounds were characterized by single-crystal X-ray diffraction, X-ray powder diffraction, ICP, TGA, IR, and photoluminescence spectroscopy. The crystal structures were determined by X-ray diffraction and refined by full-matrix least-squares methods to $R = 0.0221$ and $wR = 0.0579$ using 1925 reflections with $I > 2\sigma(I)$ for **1**; and $R = 0.0654$ and $wR = 0.1591$ using 1881 reflections with $I > 2\sigma(I)$ for **2**. Their frameworks are constructed from M–O–C (M = Na, Cd and Co) chains composed of alternating six-coordinate Na(I) centers and four-coordinate Cd(II) or Co(II) centers. These chains are further linked by *m*-BDC to form unusual 3D open-framework structures.

Keywords: Crystal structure; Solvothermal synthesis; Photoluminescence; Coordination polymer; 1,3-Benzene-dicarboxylate

1. Introduction

Coordination frameworks constructed by linking organic ligands with metal ions attract attention due to their fascinating structural topologies and potential applications [1–4]. The rational synthesis and design of porous metal-organic frameworks (MOFs) is an active research area owing to their intriguing structural architectures as well as potential applications in gas adsorption and catalysis [5–7]. A versatile strategy for construction of porous MOFs is to assemble rigid molecular building blocks into predetermined structures by decoration and expansion of the basic net topologies [8–10]. Based on this synthetic method, many examples of MOFs have been documented [11–13]. However, the structures of the large majority of these MOFs can be described as being constructed from discrete secondary building units (SBUs) or the same metal ions. Three-dimensional periodic MOF structures based on the assembly of rod-shaped building units and heterometallic ions remain largely unexplored [14, 15].

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*Corresponding author. Email: guangbochejl@yahoo.com

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It is well known that heterometallic coordination polymers have diverse, fascinating structures and potentially interesting physicochemical properties, such as conduction, catalysis, and magnetic ordering, that result from interactions between two distinct metal centers connected by a suitable linker [16, 17]. Here, we present the syntheses and characterizations of two novel three-dimensional (3D) heterometallic frameworks $[CdNa(m\text{-}BDC)_2]\cdot[NH_2(CH_3)_2]$ (**1**) and $[CoNa(m\text{-}BDC)_2]\cdot[NH_2(CH_3)_2]$ (**2**) (*m*-BDC = 1,3-benzene-dicarboxylate), whose frameworks are constructed from infinite M–O–C (M = Na and Cd and Co) chain building units.

2. Experimental

2.1. *Preparation*

2.1.1. Synthesis of $[CdNa(m\text{-}BDC)_2]\cdot[NH_2(CH_3)_2]$ (1). Compound **1** was synthesized from the solvothermal reaction of $Cd(NO_3)_2\cdot 4H_2O$, *m*-H_2BDC, NaOH, and *N*,*N*-dimethylformamide (DMF). Typically, 0.0845 g of *m*-H_2BDC was first dissolved into 12 mL of DMF with stirring, followed by addition of 0.040 g of NaOH. Then 0.158 g of $Cd(NO_3)_2\cdot 4H_2O$ was added to the above reaction mixture. The resulting mixture was transferred into a Teflon-lined stainless steel autoclave, and heated at 140°C for 3 days. Colorless rod-shaped single crystals were washed thoroughly with DMF, and dried at room temperature. The yield was 67% based on Cd.

2.1.2. Synthesis of $[CoNa(m\text{-}BDC)_2]\cdot[NH_2(CH_3)_2]$ (2). Compound **2** was prepared in the same way as for **1**, using $Co(NO_3)_2\cdot 4H_2O$ (0.127 g), NaOH (0.040 g), and *m*-H_2BDC (0.0845 g) as the reactants. Rose-colored crystals were obtained in a 73% yield based on Co(II).

2.2. *Physical measurements*

Elemental analyses were conducted on a Perkin-Elmer 240C element analyzer. The results showed that compound **1** contains 42.67, 3.22 and 2.58 wt% of C, H and N (Calcd 42.38, 3.14 and 2.75 wt%, respectively), and compound **2** contains 47.27, 3.45 and 3.29 wt% of C, H and N (Calcd 47.34, 3.51 and 3.1 wt%, respectively). Inductively coupled plasma (ICP) analysis performed on a Perkin–Elmer Optima 3300DV spectrometer indicates that **1** contain 22.17 and 4.58 wt% of Cd and Na (Calcd Cd, 22.05; Na, 4.51 wt%), and **2** contain 12.78 and 5.01 wt% of Co and Na (Calcd Co, 12.91; Na, 5.04 wt%), respectively. These compositional analysis results are in good agreement with the empirical formulaa, $[CoNa(m\text{-}BDC)_2]\cdot[NH_2(CH_3)_2]$ (**1**) and $[CdNa(m\text{-}BDC)_2]\cdot[NH_2(CH_3)_2]$ (**2**), given by single-crystal structure analysis. The infrared spectra were recorded within the 400–4000 cm^{-1} region on a Bruker IFS 66V/S FTIR spectrometer using KBr pellets. TGA was carried out on a TA Instruments with a heating rate of 10°C min^{-1}.

2.3. *Crystal structure determination*

Crystallographic data for **1** and **2** were recorded at room temperature on a Bruker-AXS Smart CCD diffractometer equipped with a normal-focus, 2.4 kW X-ray source (graphite-monochromated Mo-Kα radiation with $\lambda = 0.71073$ Å) operating at 50 kV and 40 mA with increasing ω (width of 0.3° and exposure time 30 s per frame). The structures were solved by direct methods using SHELXS-97 [18] and refined by full-matrix least-squares techniques against F^2 using the SHELXTL-97 [19] crystallographic software package. All non-hydrogen atoms were easily found from the difference Fourier map and refined anisotropically, whereas the hydrogen atoms of the organic molecules were placed by geometrical considerations and added to the structure factor calculation. The detailed crystallographic data and structure refinement parameters for **1** and **2** are summarized in table 1 Selected bond distances and angles for **1** and **2** are listed in table 2.

3. Results and discussion

3.1. *Crystal structure*

The structure of **1** is isostructural with **2**, and therefore only the structure of **1** will be described in detail. Single-crystal structure analysis reveals that compound **1** is a new 3D heterometallic MOF with the formula $[\mathrm{CoNa}(m\text{-BDC})_2]\cdot[\mathrm{NH_2(CH_3)_2}]$. The asymmetric unit for **1** is shown in figure 1. Two carboxylate groups of one *m*-BDC connect two Cd(II) ions and three Na(I) ions. The Na(I) ion is six-coordinate to six different carboxyl groups and exhibits octahedral geometry. The Cd(II) ion is four-coordinate to four different carboxyl groups and exhibits tetrahedral geometry. Each Cd(II) atom is corner-shared with two adjacent Na(I) atoms through two

Table 1. Crystal data and structure refinements for **1** and **2**.

Compound	**1**	**2**
Formula	$C_{18}H_{16}CdNNaO_8$	$C_{18}H_{16}CoNNaO_8$
Formula weight	509.71	456.24
Crystal system	Monoclinic	Monoclinic
Space group (Å, °)	$C2/c$	$C2/c$
a	14.7366(19)	13.3751(12)
b	13.5985(17)	14.3847(12)
c	11.3369(14)	11.3178(8)
β	113.141(2)	111.405(7)
V (Å^3)	2089.1(5)	2027.3(3)
Z	4	4
R_1 ($I > 2\sigma(I)$)	0.0221	0.0654
wR_2 ($I > 2\sigma(I)$)	0.0579	0.1591
D_c (g cm^{-3})	1.621	1.495
$F(000)$	1016	932
μ (Mo-Kα, mm^{-1})	1.109	0.912
Goodness-of-fit on F^2	1.080	1.047
Reflections collected	5468	7044
Unique reflections	2049	2480
Observed reflections [$I > 2\sigma(I)$]	1925	1881

Table 2. Selected bond distances in (Å) and angles in (°) for **1** and **2**.

[CdNa(m-BDC)$_2$] · [NH$_2$(CH$_3$)$_2$] (**1**)			
Na(1)–O(3)	2.404(4)	Na(1)–O(3)$^{\#3}$	2.404(4)
Na(1)–O(1)$^{\#4}$	2.409(3)	Na(1)–O(1)$^{\#5}$	2.409(3)
Na(1)–O(2)$^{\#6}$	2.513(3)	Na(1)–O(2)$^{\#7}$	2.513(3)
Cd(1)–O(1)	2.229(3)	Cd(1)–O(1)$^{\#8}$	2.229(3)
Cd(1)–O(4)$^{\#1}$	2.285(3)	Cd(1)–O(4)$^{\#2}$	2.285(3)
O(1)$^{\#4}$–Na(1)–O(1)$^{\#6}$	180.00(8)	O(1)$^{\#4}$–Na(1)–O(2)$^{\#6}$	85.09(12)
O(1)$^{\#5}$–Na(1)–O(2)$^{\#6}$	94.91(12)	O(3)$^{\#3}$–Na(1)–O(2)$^{\#7}$	84.04(13)
O(3)–Na(1)–O(2)$^{\#7}$	95.96(13)	O(1)$^{\#4}$–Na(1)–O(2)$^{\#7}$	94.91(12)
O(1)$^{\#5}$–Na(1)–O(2)$^{\#7}$	85.09(12)	O(2)$^{\#6}$–Na(1)–O(2)$^{\#7}$	180.00(17)
O(1)–Cd(1)–O(1)$^{\#8}$	116.29(18)	O(1)–Cd(1)–O(4)$^{\#1}$	128.26(12)
O(1)$^{\#8}$–Cd(1)–O(4)$^{\#1}$	96.16(14)	O(1)–Cd(1)–O(4)$^{\#2}$	96.16(14)
O(1)$^{\#8}$–Cd(1)–O(4)$^{\#2}$	128.26(12)	O(4)$^{\#1}$–Cd(1)–O(4)$^{\#2}$	93.0(2)
[CoNa(m–BDC)$_2$] · [NH$_2$(CH$_3$)$_2$] (**2**)			
Na(2)–O(4)$^{\#2}$	2.383(6)	Na(2)–O(2)$^{\#1}$	2.418(4)
Na(2)–O(1)	2.423(4)	Na(2)–O(1)$^{\#6}$	2.423(4)
Co(1)–O(1)	2.002(3)	Co(1)–O(3)$^{\#2}$	2.081(6)
O(1)–Co(1)–O(1)$^{\#1}$	109.6(2)	O(1)–Co(1)–O(3)$^{\#2}$	131.9(3)
O(1)–Co(1)–O(3)$^{\#3}$	98.4(2)	O(3)$^{\#2}$–Co(1)–O(3)$^{\#3}$	90.3(3)
O(4)$^{\#2}$–Na(2)–O(4)$^{\#4}$	180.0	O(4)$^{\#2}$–Na(2)–O(2)$^{\#1}$	88.7(3)
O(4)$^{\#4}$–Na(2)–O(2)$^{\#1}$	91.3(3)	O(4)$^{\#2}$–Na(2)–O(1)	73.9(2)
O(4)$^{\#4}$–Na(2)–O(1)	106.1(2)	O(2)$^{\#1}$–Na(2)–O(1)	81.88(14)
O(2)$^{\#5}$–Na(2)–O(1)	98.12(14)		

Symmetry codes for **1**: $^{\#1}x+1/2,\ y+1/2,\ z$; $^{\#2}-x+3/2,\ y+1/2,\ -z+1/2$; $^{\#3}-x+1,\ -y-1,\ -z$; $^{\#4}x-1/2,\ y-1/2,\ z$; $^{\#5}-x+3/2,\ -y-1/2,\ -z$; $^{\#6}-x+3/2,\ y-1/2,\ -z+1/2$; $^{\#7}x-1/2,\ -y-1/2,\ z-1/2$; $^{\#8}-x+2,\ y,\ -z+1/2$. Symmetry codes for **2**: $^{\#1}-x+2,\ y,\ -z+0.5$; $^{\#2}x+0.5,\ y+0.5,\ z$; $^{\#3}-x+1.5,\ y+0.5,\ -z+0.5$; $^{\#4}-x+1.5,\ -y-0.5,\ -z$; $^{\#5}x,\ -y,\ z-0.5$;

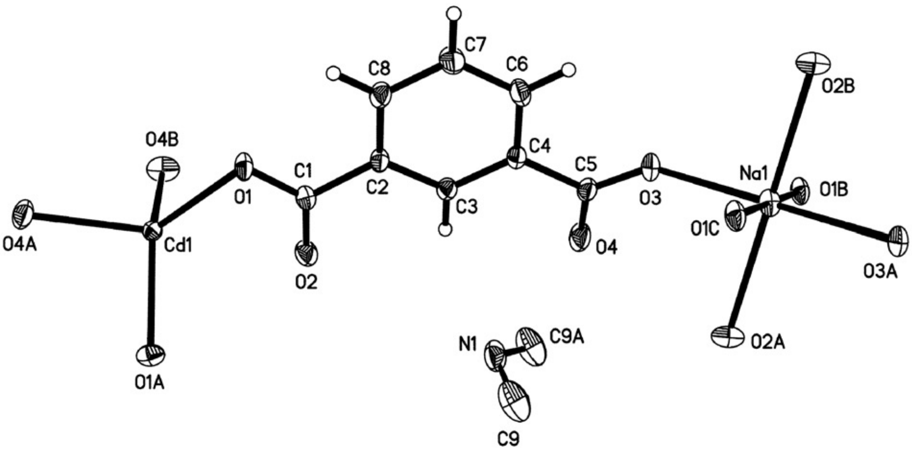

Figure 1. Thermal ellipsoid plot (30%) showing the asymmetric unit of **1**.

carboxyl groups. This connectivity pattern results in infinite M–O–C (M = Cd and Na) chains (figure 2). Each chain is further linked to four neighboring chains by the m-BDC units, leading to an interesting 3D framework structure (figures 3 and 4). Heterometallic framework complexes containing Co(II) or Cd(II) as well as Na(I) ions are relatively rare [20]. Thus, **1** represents an unusual open-framework heterometallic MOF structure formed by the infinite Cd–Na chain packing arrangement [21]. The structure of **1** has channels occupied by disordered protonated dimethylamine cations (figures 3 and 4).

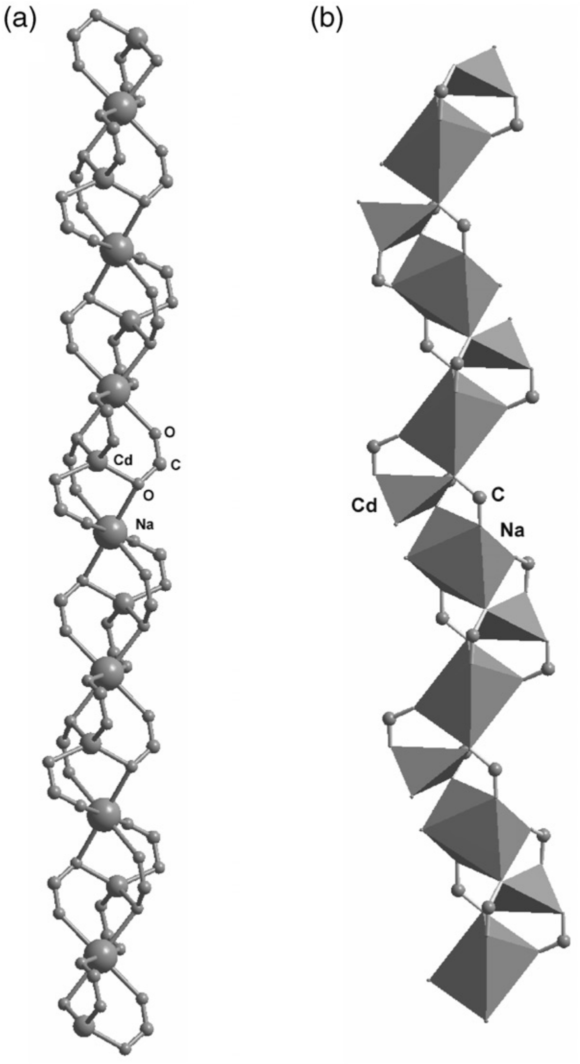

Figure 2. Infinite M–O–C chain used to assemble MOF of **1**: (a) ball-and-stick representation of M–O–C chain; (b) M–O–C chain shown as polyhedron.

In addition, there exist weak N–H ··· O hydrogen bonds between the protonated dimethylamine cations and the oxygen atoms of carboxylate groups. The observed distance from the N atom to carboxylate oxygen atom is 3.161 Å [22].

It should be pointed out that the dimethylammonium cations in the product result from decomposition of DMF under solvothermal conditions as observed in the literature [23]. When the reaction was carried out in different solvents, such as ethanol, and ethylene glycol, **1** or **2** was not obtained, demonstrating that DMF is important in the final product. When we chose dimethylamine (45 wt%) as the template and ethanol as the solvent, we could also obtain **1** or **2**. This shows that dimethylamine plays a templating role in crystallization.

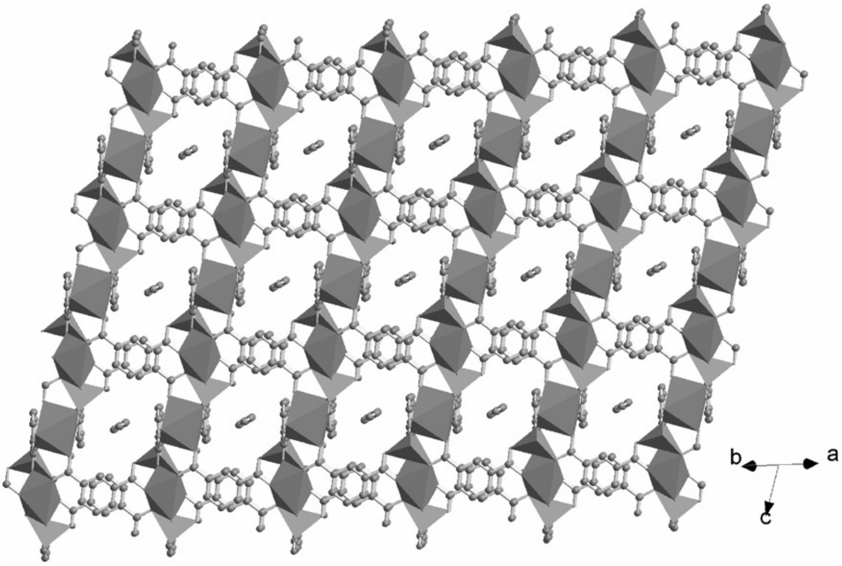

Figure 3. Framework of **1** with dimethylamine templating cations, viewed along the [111] direction. Hydrogen atoms have been omitted for clarity.

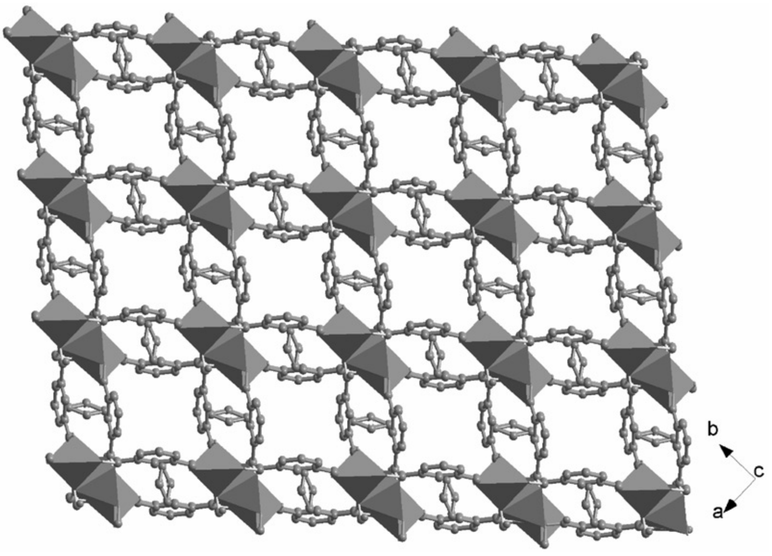

Figure 4. Framework of **1** with dimethylamine templating cations viewed along the [001] direction. Hydrogen atoms have been omitted for clarity.

3.2. *IR spectroscopy*

The IR spectra of **1** and **2** reveal the characteristic bands of the organic ligand *m*-BDC at about 1580 $\mathrm{cm^{-1}}$ for the asymmetric vibration and at about 1450 $\mathrm{cm^{-1}}$ for the symmetric vibration (figure 5). The C–H stretching mode for the phenyl ring is relatively weak and observed at about 3000 $\mathrm{cm^{-1}}$ [24, 25]. The bands at around

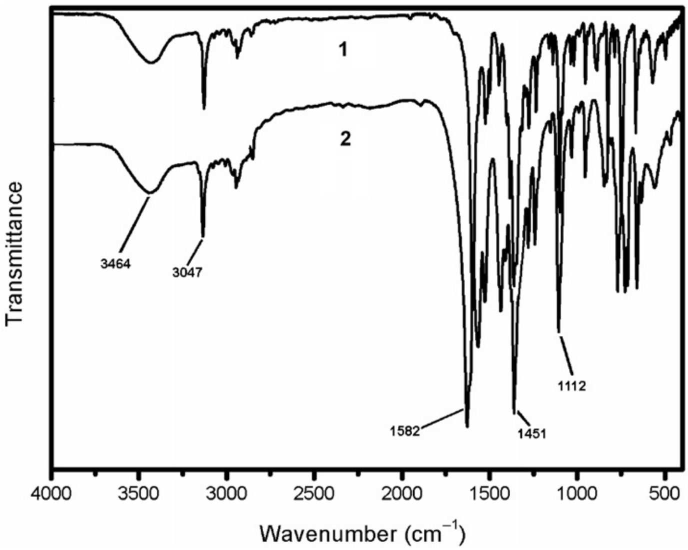

Figure 5. The IR spectra of compounds **1** and **2**.

3464 cm^{-1} are attributed to vibrations of N–H for dimethylamine molecules. The absence of the characteristic bands attributed to protonated carboxylate groups about 1750 cm^{-1} indicates complete deprotonation of the *m*-BDC ligand [26, 27].

3.3. *TG analysis*

In order to characterize more fully compounds **1** and **2**, we examined **1** and **2** using TGA. Thermogravimetric curves have been obtained in air for crystalline samples of these two complexes in the temperature range of 35 to 800°C (figure 6). As expected, the TGA curves of compounds **1** and **2** exhibit two similar weight loss stages. The first weight loss, corresponding to the removal of dimethylamine cations and the carboxylate groups of *m*-BDC ligands, is 44.32% (calculated 43.55%) for **1** in the temperature range 35–370°C, 45.56% (calculated 48.66%) for **2** in the range 35–372°C. The second weight losses, assigned to decomposition of the phenyl ring of *m*-BDC ligands, were observed from 370 to 550°C for **1** (observed 32.90%, calculated 30.61%), from 372 to 570°C for **2** (observed 33.40%, calculated 34.19%), respectively.

3.4. *Powder X-ray diffraction*

The powder X-ray diffraction (XRD) data were collected on a Siemens D5005 diffractometer with Cu-Kα radiation ($\lambda = 1.5418$ Å) and the recording speed was 0.3° min^{-1} over the 2θ range of 4–40° at room temperature. The simulated and experimental powder X-ray diffraction patterns for **1** are shown figure 7. The experimental powder X-ray diffraction pattern is in good agreement with the corresponding simulated one except for relative intensity variation because of preferred orientations of the crystals. Therefore, the phase purity of the as-synthesized products is substantiated. The powder pattern for **1** did not change after heating to 290°C.

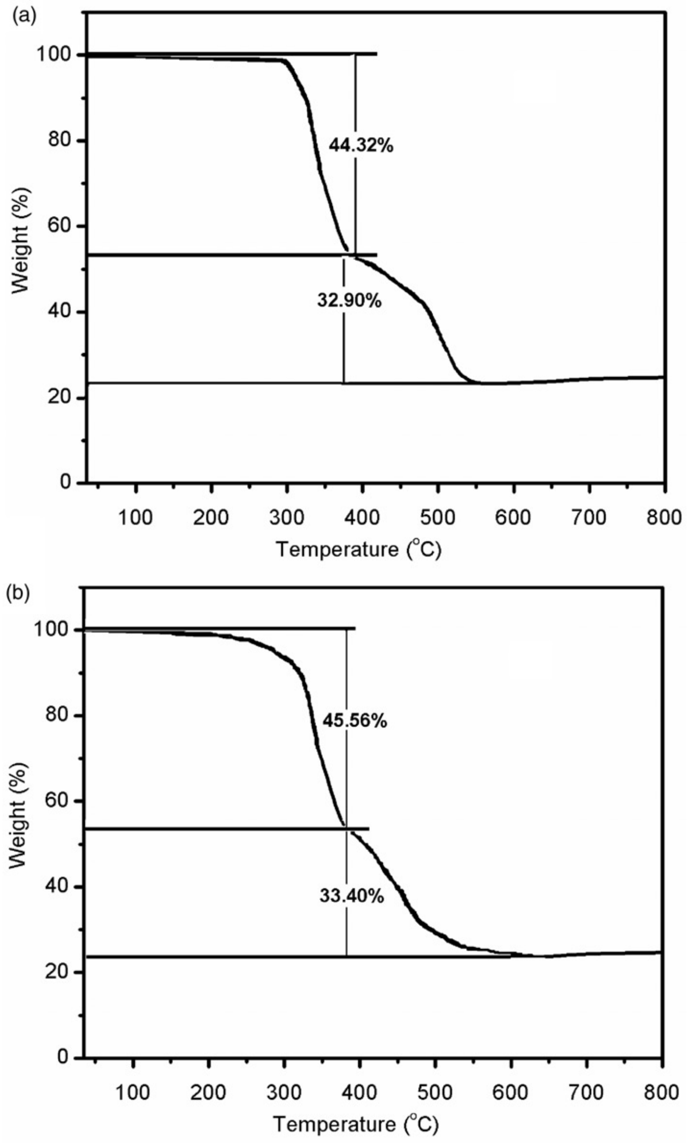

Figure 6. The TG curve for compounds **1** (a) and **2** (b).

Thus, the XRD analysis reveals that the heterometallic compound **1** has a relatively high thermal stability. However, they collapsed above 300°C with the loss of dimethylamine cations and carboxylate groups of *m*-BDC ligands.

3.5. *Photoluminescence*

The luminescent properties of compounds **1** and **2** were measured on a Perkin-Elmer LS55 spectrometer. As we and others previously reported, coordination polymers

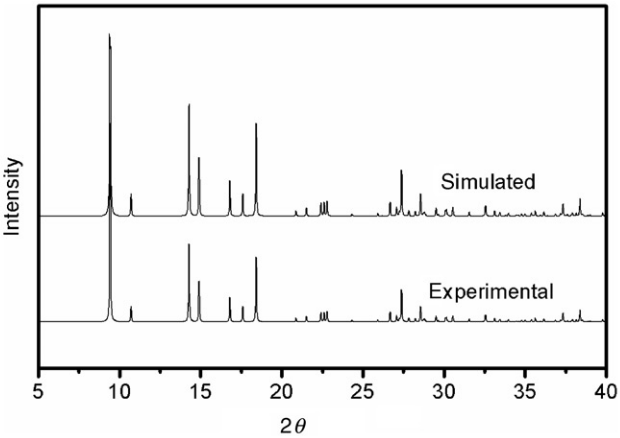

Figure 7. XRD patterns of the simulated and experimental compound **1**.

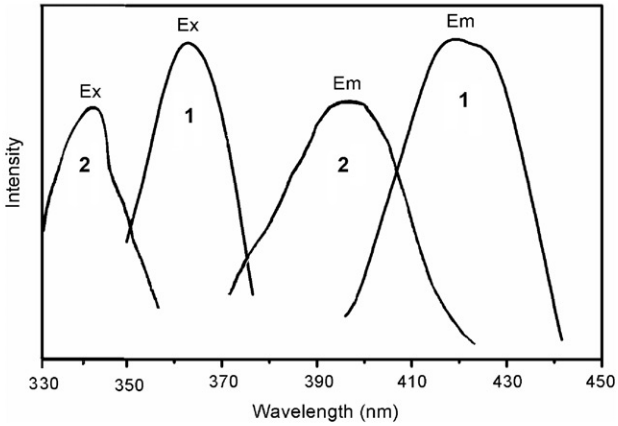

Figure 8. Photoluminescent spectra of **1** and **2** in the solid state at room temperature. Ex = excitation, Em = emission.

containing Cd(II) exhibit photoluminescence [28–30]. The solid-state photoluminescence spectra of **1** and **2** were recorded at room temperature (figure 8). The solid-state excitation–emission spectrum of the free *m*-BDC ligand shows the strongest emission peak at 380 nm with the excitation peak at 340 nm at room temperature [10]. The emission bands for this free ligand are attributable to the $\pi^* \rightarrow n$ transitions. Compared to the free ligands, the strongest excitation peak for **1** is at 355 nm, with an energy lower than that of the *m*-BDC ligand. The emission spectrum of **1** shows one strong peak at 428 nm, which may be attributed to the ligand-to-metal charge-transfer (LMCT) band [31, 32]. Compound **2** contains a non-emissive Co(II) ion and this material shows a very weak emission band at 397 nm ($\lambda_{ex} = 340$ nm), which is believed to arise from charge transfer transition between the *m*-BDC ligand and the central Co(II) ion, suggesting energy transfers from the *m*-BDC ligands to the metal centers during photoluminescence for these two compounds. Compound **1** may be a good candidate for useful photoactive material due to strong fluorescence.

4. Conclusion

Two heterometallic metal-organic frameworks have been synthesized under solvothermal condition. The frameworks are built up from infinite heterometallic chains composed of alternating NaO_6 octahedra and CdO_4 or CoO_4 tetrahedra. The chains are further connected through *m*-BDC linkers to give chain packing structures. The X-ray powder diffraction, ICP, TGA, IR, and photoluminescence spectroscopy were also studied for the compounds.

Supplementary data

The crystallographic data have been deposited at Cambridge Crystallographic Data Centre, CCDC Nos 296111 for **1** and 296112 for **2**. Copies of this information may be obtained free of charge from the director, CCDC, 12 Union Road, Cambridge, CB2 IEZ, UK (Email: deposit@ccdc.cam.ac.uk; Fax: 44-1223-336-033; http://www.ccdc.cam.ac.uk).

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