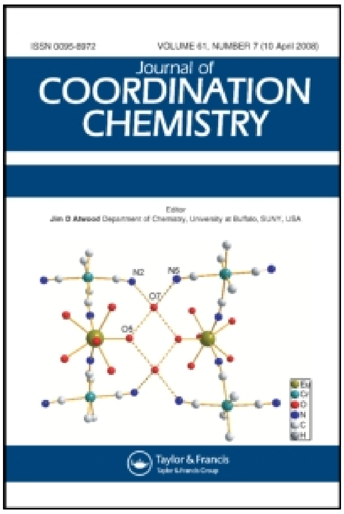

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Synthesis, structures and thermal stabilities of three 1-D coordination polymers based on flexible polycarboxylates

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Synthesis, structures and thermal stabilities of three 1-D coordination polymers based on flexible polycarboxylates

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Three new coordination polymers, $[Cu(butca)_{0.5}(bipy)(H_2O)]_n \cdot 2nH_2O$ (**1**), $[Zn(H_2butca)(phen)(H_2O)]_n \cdot nH_2O$ (**2**), and $[Cd(H_2chhca)_{0.5}(phen)(H_2O)]_n \cdot 2nH_2O$ (**3**) (H_4butca = 1,2,3,4-butanetetracarboxylic acid, H_6chhca = 1,2,3,4,5,6-cyclohexanehexacarboxylic acid), were prepared and characterized by EA, IR, TG, and X-ray crystallography. Complex **1** is a 1-D double-chain coordination polymer in which tetradentate $butca^{4-}$ coordinates to four Cu(II) ions through four monodentate carboxylates. Complex **2** is a 1-D chain with tridentate H_2butca^{2-} coordinating to two Zn(II) ions through monodentate and chelating carboxylates. Complex **3** is a 1-D double-chain coordination polymer. H_2chhca^{4-} is octadentate coordinating to four Cd(II) ions through four chelating carboxylates. Hydrogen bonds and π–π stacking interactions play important roles in the formation of supramolecular architectures. The thermal stabilities of **1**–**3** show dehydrated coordination polymers are thermally stable in the range 260–400°C.

Keywords: Coordination polymer; Crystal structure; 1,2,3,4,5,6-Cyclohexanehexacarboxylic acid; 1,2,3,4-Butanetetracarboxylic acid; Thermal analysis

1. Introduction

Coordination polymers from metal ions and multifunctional ligands have attracted considerable attention because of the structural diversity which facilitates systematic evaluation of structure property relationships [1–3]. In particular, polycarboxylates are good building blocks in the design of coordination polymers with desired topologies owing to their rich coordination modes resulting in potential applications in fields, such as porous, magnetic, luminescent, and catalytic materials [4–6]. Although rigid polycarboxylates were employed to afford coordination polymers, the use of flexible polycarboxylic acids can afford interesting polymeric networks [7, 8].

1,2,3,4-Butanetetracarboxylic acid (H_4butca) is a flexible multifunctional ligand which has four carboxyl groups and exhibits varied coordination modes. A number of

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H_4butca complexes have been synthesized and structurally characterized [9–11]. Most of them contain co-ligands, such as 1,10-phen and 2,2′-bipy. 1,2,3,4,5,6-Cyclohexanehexacarboxylic acid (H_6chhca) is another potential multifunctional ligand, which has six carboxyl groups and exhibits flexible conformations. Several H_6chhca coordination polymers have been documented, which exhibit intriguing metal–organic frameworks [12, 13]. In the course of investigating polycarboxylate coordination polymers [14–16], and to further understand coordination behaviors of H_4butca and H_6chhca, we have prepared three new polycarboxylate coordination polymers, which display 1-D single-chain or double-chain polymeric structures. We report here their synthesis, crystal structures, and thermal stabilities.

2. Experimental

2.1. *Materials and measurements*

All chemicals were reagent grade and used without purification. Elemental analyses (C, H, N) were performed on a Vario EL III analyzer. Infrared spectra were recorded with a Nicolet A370 FT-IR spectrometer using KBr pellets (400–4000 cm^{-1}). Thermogravimetric analyses (TG–DSC) were completed on a Nerzsch STA 449C thermal analyzer at a heating rate of 10°C min^{-1} in air.

2.2. *Synthesis*

2.2.1. $[Cu(butca)_{0.5}(bipy)(H_2O)]_n \cdot 2nH_2O$ (1). An ethanol solution (5 mL) of 2,2′-bipyridine (0.25 mmol) was added to a 5 mL aqueous solution containing H_4butca (0.20 mmol) and $CuCl_2 \cdot 2H_2O$ (0.30 mmol). The resulting mixture was left undisturbed at room temperature for 72 h. Blue rod crystals of **1** separated in 34% yield based on bipy. Anal. Calcd for $C_{14}H_{17}CuN_2O_7$: C, 43.24; H, 4.41; N, 7.20. Found: C, 43.35; H, 4.63; N, 7.28. IR (KBr, cm^{-1}): 3423s, 1615s, 1600s, 1566s, 1422s, 1369m, 1273m, 780m, 588m.

2.2.2. $[Zn(H_2butca)(phen)(H_2O)]_n \cdot nH_2O$ (2). A mixture of $Zn(NO_3)_2 \cdot 6H_2O$ (0.20 mmol), H_4butca (0.20 mmol), 1,10-phenanthroline (0.25 mmol), and 10 mL H_2O sealed in a 15 mL Teflon-lined stainless steel reactor, which was heated at 160°C for 72 h was used. Colorless block crystals of **2** separated in 41% yield based on phen. Anal. Calcd for $C_{20}H_{20}N_2O_{10}Zn$: C, 46.76; H, 3.92; N, 5.45. Found: C, 46.70; H, 4.02; N, 5.54. IR (KBr, cm^{-1}): 3380s, 2950w, 1730s, 1693s, 1561s, 1515m, 1426s, 1394s, 1282s, 1211m, 1148m, 851m, 727m.

2.2.3. $[Cd(H_2chhca)_{0.5}(phen)(H_2O)]_n \cdot 2nH_2O$ (3). A mixture of $Cd(NO_3)_2 \cdot 4H_2O$ (0.30 mmol), 1,10-phenanthroline (0.10 mmol), H_6chhca (0.10 mmol), and 10 mL H_2O sealed in a 15 mL Teflon-lined stainless steel reactor, which was heated at 160°C for 72 h was used. Light yellow block crystals of **3** were separated manually from a mixture. Anal. Calcd for $C_{18}H_{18}CdN_2O_9$: C, 41.67; H, 3.50; N, 5.40. Found: C, 39.30;

Table 1. Crystallographic data and structural refinement for **1**–**3**.

Complex	**1**	**2**	**3**
Empirical formula	$C_{14}H_{17}CuN_2O_7$	$C_{20}H_{20}N_2O_{10}Zn$	$C_{18}H_{18}CdN_2O_9$
Formula weight	388.84	513.75	518.74
Crystal system	Triclinic	Monoclinic	Triclinic
Space group	$P\bar{1}$	$P2_1/c$	$P1$
Unit cell dimensions (Å, °)			
a	7.1403(16)	7.7252(15)	8.6244(11)
b	10.488(2)	23.173(5)	9.1752(12)
c	10.849(2)	11.987(2)	13.3163(18)
α	87.416(3)	90	95.372(2)
β	82.795(3)	107.494(3)	103.728(2)
γ	88.555(3)	90	108.409(2)
Volume $Å^3$, Z	805.0(3), 2	2046.6(7), 4	954.9(2), 2
Calculated density ($g\,cm^{-3}$)	1.604	1.667	1.804
Absorption coefficient (mm^{-1})	1.396	1.264	1.200
$F(000)$	400	1056	520
Reflections collected	4237	10,426	4957
Unique reflections (R_{int})	2806 (0.0308)	3612 (0.0871)	3301 (0.0215)
Goodness-of-fit on F^2	1.009	1.013	1.048
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0502$, $wR_2 = 0.0864$	$R_1 = 0.0586$, $wR_2 = 0.1061$	$R_1 = 0.0358$, $wR_2 = 0.0792$
R indices (all data)	$R_1 = 0.0707$, $wR_2 = 0.0961$	$R_1 = 0.1101$, $wR_2 = 0.1239$	$R_1 = 0.0444$, $wR_2 = 0.0844$
Largest difference peak and hole ($e\,Å^{-3}$)	0.431 and −0.474	0.436 and −0.527	0.508 and −0.469

H, 2.50; N, 4.45. IR (KBr, cm^{-1}): 3466m, 3065w, 2961w, 1731m, 1687m, 1599s, 1515m, 1428m, 1406s, 847s, 727s.

2.3. *Crystal structure determination*

Well-shaped single crystals of **1**–**3** were selected for X-ray diffraction. Data collections were performed with graphite-monochromatic Mo-Kα radiation ($\lambda = 0.71073$ Å) on a Bruker Smart Apex-II CCD diffractometer at room temperature. Determinations of the crystal system, orientation matrix, and cell dimensions were performed according to established procedures. Lorentz polarization and absorption corrections were applied. The structures were solved by direct methods and refined on F^2 by full-matrix least-squares with SHELXTL [17]. All non-hydrogen atoms were refined anisotropically and hydrogens were located and included at their calculated positions. The crystal data and refinement results are summarized in table 1; selected bond distances and angles are presented in table 2.

3. Results and discussion

3.1. *Descriptions of the crystal structures*

3.1.1. $[Cu(butca)_{0.5}(bipy)(H_2O)]_n \cdot 2nH_2O$ (1). The solution reaction of $CuCl_2$ with H_4butca and 2,2′-bipyridine in a 3 : 2 : 2.5 molar ratio afforded **1**, which is a 1-D

Table 2. Selected bond distances (Å) and angles (°) for **1**–**3**.

1[a]			
Cu(1)–O(1)	1.936(3)	Cu(1)–O(5w)	2.392(3)
Cu(1)–N(2)	1.993(3)	Cu(1)–O(4)#1	1.960(3)
Cu(1)–N(1)	1.998(4)		
N(1)–Cu(1)–N(2)	81.18(15)	N(2)–Cu(1)–O(5w)	90.24(13)
O(1)–Cu(1)–N(2)	170.80(15)	O(4)#1–Cu(1)–N(1)	168.38(14)
2[b]			
Zn(1)–O(1)	2.051(4)	Zn(1)–N(1)	2.209(4)
Zn(1)–O(9w)	2.058(3)	Zn(1)–N(2)	2.070(4)
Zn(1)–O(4)#1	2.114(3)	Zn(1)–O(2)	2.520(3)
O(4)#1–Zn(1)–N(1)	169.78(16)	O(1)–Zn(1)–N(2)	140.53(15)
N(2)–Zn(1)–N(1)	77.63(17)	O(1)–Zn(1)–O(9w)	106.30(14)
O(9w)–Zn(1)–N(2)	111.08(15)	O(9w)–Zn(1)–O(4)#1	90.05(13)
3[c]			
Cd(1)–O(1w)	2.268(3)	Cd(1)–N(1)	2.393(4)
Cd(1)–O(1)	2.324(3)	Cd(1)–N(2)	2.360(4)
Cd(1)–O(2)	2.570(3)	Cd(1)–O(3)#1	2.544(3)
Cd(1)–O(4)#1	2.308(3)		
N(1)–Cd(1)–O(1)	155.69(12)	N(1)–Cd(1)–N(2)	69.93(12)
N(2)–Cd(1)–O(4)#1	141.56(12)	O(1)–Cd(1)–O(2)	52.40(11)
O(1W)–Cd(1)–O(3)#1	152.94(13)	O(3)#1–Cd(1)–O(4)#1	52.94(10)

Symmetric code: [a]#1: $x-1$, y, z; [b]#1: $x-1$, y, z; [c] #1: $-x$, $-y+1$, $-z$.

double-chain coordination polymer. As shown in figure 1(a), the asymmetric structural unit contains an independent Cu(II), half of a butca^{4-}, bipy, a coordinated water molecule, and two lattice water molecules. Cu(1) is a distorted square-pyramid, coordinated by two carboxylate oxygens, two bipy nitrogens, and water O(5w). Carboxylates O(1B) and O(4A) coordinate to Cu(1) with bond distances of 1.936(3) and 1.960(3) Å, respectively. The 2,2′-bipyridine chelates to Cu(1) with N(1)–Cu(1)–N(2) bond angle of 81.18(15)°. These nitrogen and oxygen donors form the basal plane with bond angles of N(2)–Cu(1)–O(1B) 170.80(15)° and N(1)–Cu(1)–O(4A) 168.38(14)°. Water occupies the apical position with a bond distance of Cu(1)–O(5w) 2.392(3) Å, which is obviously longer than the carboxylate Cu–O bonds.

1,2,3,4-Butanetetracarboxylic acid is fully deprotonated. There is a symmetric center at the center of butca^{4-} which is a tetradentate coordinating four Cu(II) ions through four monodentate carboxylates. This results in formation of a 1-D double-chain coordination polymer as shown in figure 1(b). The 1-D ribbon chains are parallel stacked along the *a*-axis, and the bipy planes stack in an interpenetrating mode. The ribbon chain shows an angle of 37.5° with bipy plane. The centroid-to-centroid distance between the aromatic rings of adjacent bipy ligands is 4.10 Å, indicating no obvious π–π stacking interaction. The carboxylates, coordinated water, and lattice water molecules are connected by hydrogen bonds. There exist six kinds of hydrogen bonds in the structure, which link the 1-D double-chains into a supramolecular architecture as shown in the stacking diagram (figure 1c).

3.1.2. [Zn(H$_2$butca)(phen)(H$_2$O)]$_n$ · nH$_2$O (2). The hydrothermal reaction of Zn(NO$_3$)$_2$ with H$_4$butca and 1,10-phenanthroline in a 2 : 2 : 2.5 molar ratio afforded **2**, which is a 1-D single-chain coordination polymer. As shown in figure 2(a), the asymmetric

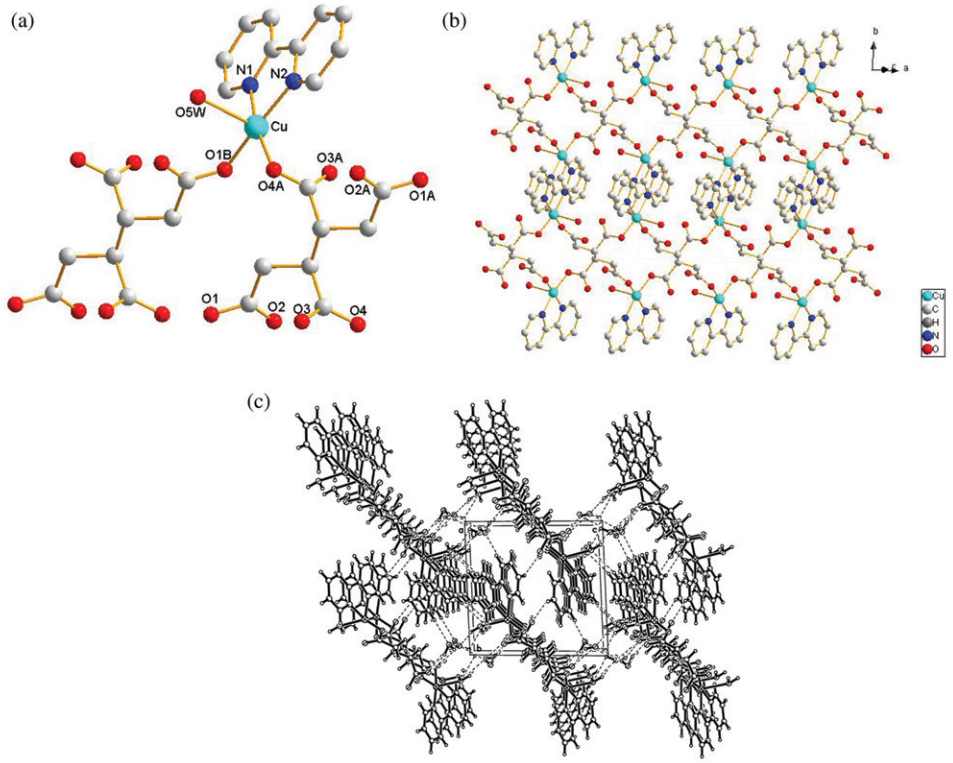

Figure 1. Views of (a) asymmetric unit, (b) 1-D polymeric double-chain, and (c) packing diagram of **1**.

structural unit contains an independent Zn(II), a H_2butca^{2-}, a phen, a coordinated water molecule and a lattice water molecule. Zn(1) adopts a distorted octahedral coordinated geometry, coordinated by four oxygens and two nitrogens. Phen chelates to Zn1 with N(1)–Zn(1)–N(2) angle of 77.63(17)°. Zn(1)–N(1) bond distance of 2.209(4) Å is longer than that of Zn(1)–N(2) 2.070(4) Å. Water O(9w) coordinates to Zn(1) with a distance of Zn(1)–O(9w) 2.058(3) Å. The carboxylates O(1) and O(4A) coordinate to Zn(1) with bond distances of Zn(1)–O(1) 2.051(4) Å and Zn(1)–O(4A) 2.114(3) Å. The carboxylate O(2) binds to Zn(1) with a weak coordinate bond. The Zn(1)–O(2) bond distance is 2.520(3) Å. Hence, the coordinate geometry around Zn(1) is between octahedral and trigonal-bipyramidal. The O(4A)–Zn(1)–N(1) bond angle is 169.78(16)°.

1,2,3,4-Butanetetracarboxylic acid is partially deprotonated to afford H_2butca^{2-}, which is different from $butca^{4-}$ in **1**. Deprotonation occurs at the 1- and 2-positions. Two carboxylates display different coordination modes. One is monodentate, whereas the other one is bidentate chelating. The H_2butca^{2-} is tridentate connecting two Zn(II) ions. This results in the formation of a 1-D single-chain coordination polymer (figure 2b). The distance between neighboring Zn(II) ions is 7.7 Å. The 1-D chains are parallel stacked along the *a*-axis, and the phen planes stack in an interpenetrating mode. The plane of phen has an angle of 55.1° with the 1-D chain. Adjacent phen aromatic rings are parallel with centroid-to-centroid distance between the aromatic rings of 3.55 Å, indicating π–π stacking interaction. The carboxyl groups, coordinated water,

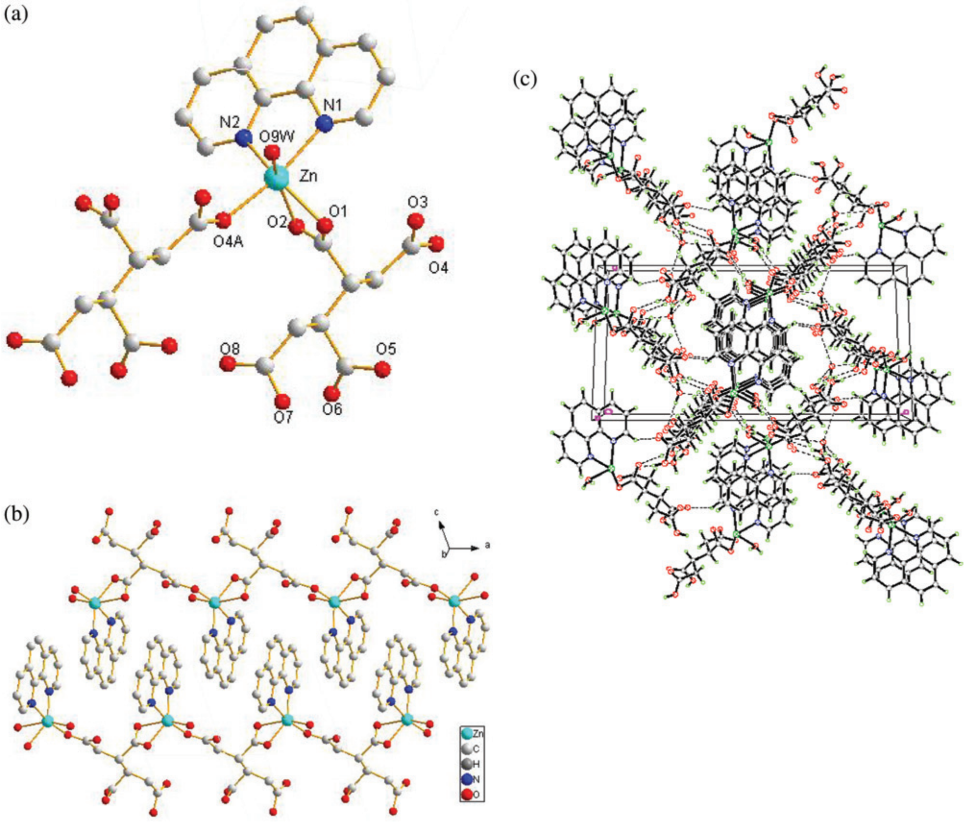

Figure 2. Views of (a) asymmetric unit, (b) 1-D polymeric single-chain, and (c) packing diagram of **2**.

and lattice water molecules are combined by hydrogen bonds. There exist five kinds of hydrogen bonds in the structure. These hydrogen bonds and π–π stacking interactions assemble the 1-D coordination polymer into a supramolecular architecture as shown in the stacking diagram (figure 2c).

3.1.3. $[Cd(H_2chhca)_{0.5}(phen)(H_2O)]_n \cdot 2nH_2O$ (3). The hydrothermal reaction of $Cd(NO_3)_2$ with 1,2,3,4,5,6-cyclohexanehexacarboxylic acid and 1,10-phenanthroline in a 3 : 1 : 1 molar ratio afforded **3**, which is a 1-D double-chain coordination polymer. As shown in figure 3(a), the asymmetric structural unit contains an independent Cd(II), half of a H_2chhca^{4-}, a phen, a coordinated water molecule and two lattice water molecules. Cd(1) is seven-coordinate by four carboxylate oxygens from two H_2chhca^{4-} ligands, two nitrogens of phen, and water O(1w). Bond angles around Cd(II) vary from 52.40(11) to 155.69(12)°. The Cd(1)–O(1w) distance is 2.268(3) Å. Average Cd–N distance is 2.371 Å. Two chelating carboxylates coordinate to Cd(1) with Cd–O bond distances varying from 2.308(3) to 2.570(3) Å. Other bond distances and angles are as expected [12].

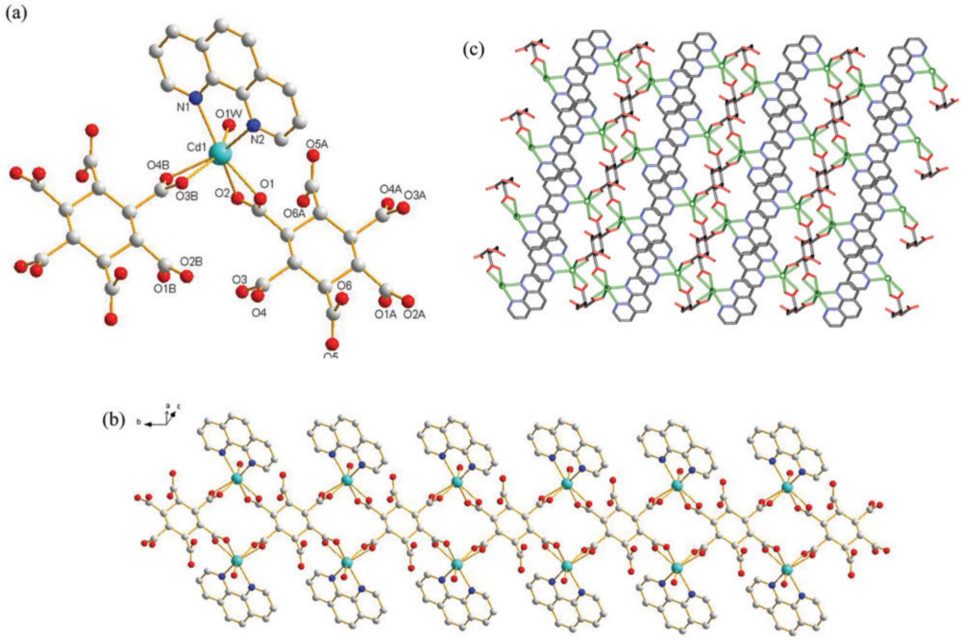

Figure 3. Views of (a) asymmetric unit, (b) 1-D polymeric double-chain, and (c) packing diagram of **3**.

Previously, 3-D cadmium coordination polymers based on 1,2,3,4,5,6-cyclohexanehexacarboxylate have been reported [12], where six carboxylates of H_6chhca are fully deprotonated. In **3**, four carboxylates of H_6chhca are deprotonated. Each H_2chhca^{4-} coordinates to four Cd(II) ions through four chelating carboxylates, leading to the formation of a 1-D double-chain coordination polymer (figure 3b). The double-chain structures of **1** and **3** are similar. The flexible H_2chhca^{4-} and butca^{4-} ligands both coordinate to four metal ions through four carboxylates, but the carboxylates of H_2chhca^{4-} are chelating whereas those of butca^{4-} are monodentate.

In **3**, the 1-D ribbon chains are parallel stacked along the *b*-axis. The parallel phen aromatic rings have a centroid-to-centroid distance of 3.56 Å, indicating π–π stacking interaction. Coordinated and lattice water molecules are hydrogen bonded to carboxylate. There exist seven kinds of hydrogen bonds in the structure. These hydrogen bonding and π–π stacking interactions play important roles in the formation of the supramolecular architecture (figure 3c).

3.2. *Thermal stability*

Thermal stability is an important parameter for metal–organic coordination polymers. The thermogravimetric analyses for **1**–**3** were performed from 20°C to 800°C (figure 4). In 40–125°C, **1** lost two lattice water molecules and one coordinated water molecule with endothermic peaks at 83.9 and 111.8°C, respectively. The total weight loss is 12.89% (Calcd: 13.90%). The dehydrated coordination polymer is stable to 260°C, and then 2,2′-bipyridine and butca^{4-} decompose with two endothermic peaks at 268.8

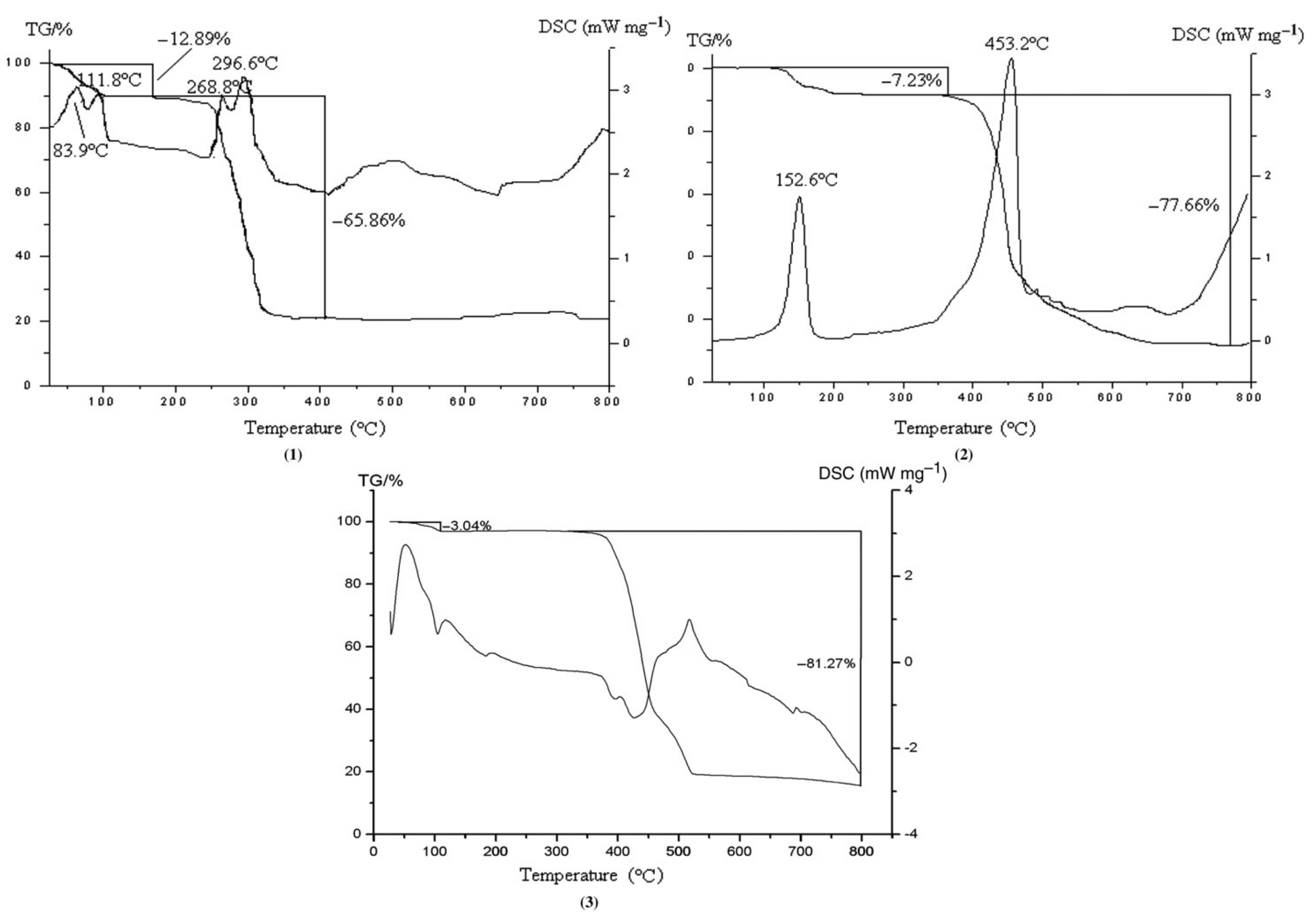

Figure 4. TG–DSC curves of **1**–**3**.

and 296.6°C. The total weight loss from 260°C to 320°C is 65.86% (Calcd: 65.65%). The final residue was CuO (found: 21.25%, Calcd: 20.46%). Complex **2** lost lattice and coordinated water molecules with an endothermic peak at 152.6°C (found: 7.23%, Calcd: 7.01%). The dehydrated coordination polymer is stable up to 370°C and then released phen and H_2butca^{2-} with an endothermic peak at 453.2°C (found: 77.66%, Calcd: 77.15%). The final residue was ZnO (found: 15.11%, Calcd: 15.84%). Complex **3** lost one coordinated water molecule at 50–125°C (found: 3.04%, Calcd: 3.47%). The dehydrated coordination polymer is stable up to 400°C and then it released phen and H_2chhca^{4-} from 400°C to 520°C, leading to 20% CdO residue which sublimed slowly at high temperature [18].

4. Conclusion

Three new coordination polymers containing flexible 1,2,3,4-butanetetracarboxylic acid and 1,2,3,4,5,6-cyclohexanehexacarboxylic acid were synthesized and structurally characterized. Both polycarboxylate ligands exhibit varied coordination modes and flexible conformation. The metal–organic networks are thermally stable. This study demonstrates that both flexible polycarboxylic acids are good building blocks in constructing coordination polymers and supramolecular frameworks.

Supplementary material

CCDC 743401–743403 contain the supplementary crystallographic data for **1**–**3**. These data can be obtained free of charge *via* http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; E-mail: deposit@ccdc.cam.ac.uk.

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